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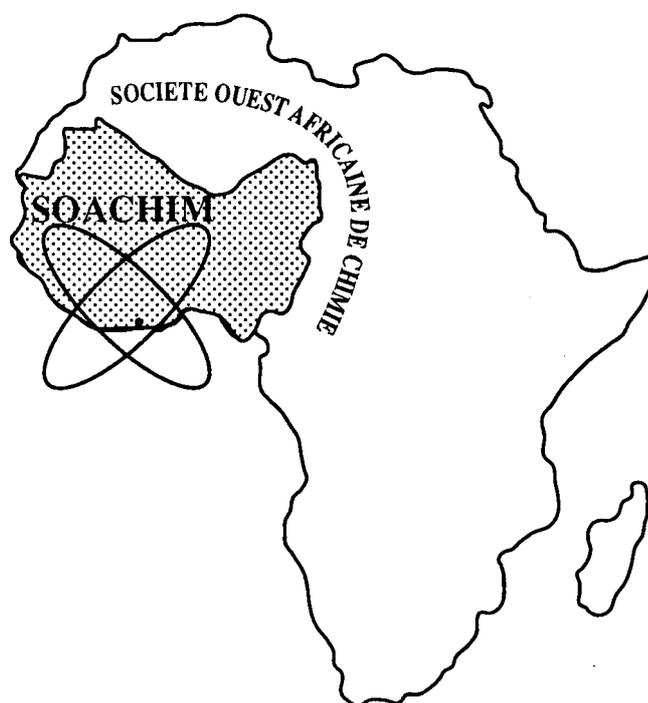
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Synthesis and crystal structure of an iron complex derived from hydrolytically unstable cyclic ligand

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Abstract: Amide ligands are susceptible to hydrolysis reaction in aqueous or in hydrated solvents in the presence of transition metal. The ligand **L** ($C_6H_{10}N_2O_2$)_n in presence of iron ion is hydrolyzed and gives fragments which react with metal to yield coordination complex. The structure of the complex reveal unexpected compound derived from oxalate and diprotonated N,N'-dimethyl-1,2-aminoethane (diMen). The compounds were characterized by elemental analysis, NMR (ligand only), mass spectra, molar conductance measurements, UV-vis, IR, TG-DTA study and XRD structure determination. The complex is a 2:1 electrolyte in nature. On the basis of spectral studies, an octahedral geometry has been assigned for the complex.

Key words: Amide, hydrolysis, complex, iron, crystal, TG-DTA.

Synthèse et structure cristalline d'un complexe de fer dérivé d'un ligand cyclique sensible à l'hydrolyse

Résumé : Les amides sont des ligands sensibles à la réaction d'hydrolyse dans une solution aqueuse ou dans des solvants hydratés, en présence d'un métal de transition. Le ligand **L** ($C_6H_{10}N_2O_2$)_n en présence des ions de fer est hydrolysé et donne des fragments qui réagissent avec le métal pour obtenir un complexe de coordination. La structure du complexe révèle un composé inattendu dérivé de l'oxalate et du N, N'-diméthyl-1,2-amino-éthane (DIMEN) diprotoné. Les composés ont été caractérisés par analyse élémentaire, par RMN (ligand seulement), par spectroscopie de masse, par mesure de conductances molaires, par UV-VIS, par IR et étude de TG-DTA. La structure est déterminée par diffraction des rayons X. Le complexe est de nature non-électrolyte. Sur la base des études spectrales, une géométrie octaédrique a été attribuée pour le complexe.

Mots clés: Amide, hydrolyse, complexe, fer, cristal, TG-ATD.

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1.1. Introduction

The use of oxalate and its oxamide derivatives in the synthesis of new complex systems is constantly growing. The reactions of these organic moieties and inorganic cations produce different structure as mononuclear^[1], polynuclear^[2] and polymeric one- (1D) [3], two- (2D)^[4] or three-dimensional (3D)^[5] assemblies. The oxalate dianion (C₂O₄²⁻) is known to be versatile upon coordination with metal ions since it can act as an unidentate, chelating or a bridging ligand^[6]. It was used widely as a building block to construct frameworks with considerable number of transition affording interesting properties. Oxamide derivatives are also intensively used due to their possibilities. Like the oxalate dianion, the oxamide dianion can act as a chelating or a bis-chelating ligand. They were known to be very efficient when using as bridge in transmitting exchange interaction between magnetic ions^[7]. Sometimes these oxamide ligands are altered owing to their sensitivity to hydrolytic decomposition giving fragments which can react with metal ions to produce complex^[8].

In this work, we report the synthesis of a novel ligand and a mononuclear precursor complex {[Fe(oxalate)₃](diMen)₂·2(H₂O)}. In addition to the single-crystal X-ray diffraction study, the characterization of the compound has been accomplished by means of IR and UV-vis spectroscopy as well as by the TG/DTA and electrochemistry analysis.

2. Experimental

2.1. Materials and Measurements

N,N'-dimethyl-1,2-diaminoethane, dimethyl oxalate and metal salts were commercial products of highest chemical grade (Aldrich). Solvents were purified according to standard procedures. Elemental analysis of C, H and N was performed by the analytical laboratory of the Department of Inorganic Chemistry (University of Padua, Italy). ¹H and ¹³C NMR spectra of the ligand and complex were obtained in CDCl₃ solutions with a Bruker Advance 400 MHz NMR spectrometer, chemical shifts are reported as δ (ppm). FTIR spectra were recorded with a Nicolet 55XC FT-IR spectrophotometer using KBr pellets. Molar conductance measurement was made using a WTW LF-330 conductivity meter with a WTW conductivity cell on about 1 mmol dm⁻³ dimethylformamide solution at room temperature. Room temperature magnetic susceptibility

measurements were made on solid complex using a Johnson-Mathey Gouy balance with Hg[Co(SCN)₄] as the calibrator. Thermal measurements were carried out on a Shimadzu DTG-60H analyser, with a heating rate of 10°C min⁻¹ in the stream of synthetic air. Electrochemical measurements were performed with a BAS CV 27 voltammogram and a BAS A-4 XY register using a glassy carbon (φ5 mm) working electrode, a platinum wire as auxiliary and a double junction, with porous ceramic wick, Ag/AgCl reference electrode, standardized for the redox couple ferricinium/ferrocene (E_{1/2}=0.400 V, ΔE_p=60 mV). Cyclic voltammetry studies of ligands and complex were carried out on 0.01 M solutions in dimethylformamide containing 0.1 M [NBu₄][PF₆] (tetrabutylammonium hexafluorophosphate: TBAHFP) as supporting electrolyte. The range of potential studied was between +1 and -1.5 V. The solution was purged with nitrogen steam for 5 min before measurement and the working electrode was polished before experiment with diamond paste. The procedure was performed at room temperature and a nitrogen atmosphere was maintained over the solution during the measurements.

2.2. Synthesis of the ligand L

In a 50 ml round bottom flask introduce dimethyl oxalate (2.36 g, 0.020 mol) dissolved in methanol (10 mL). N,N'-dimethyl-1,2-diaminoethane (1.77 g, 0.020 mol) in methanol (10 mL), was added to yield immediately a quantitative precipitate. The white precipitate formed, was separated by filtration, washed with methanol and ether and dried under vacuum (Yield 3.32 g, 58.5%); m.p.=240 °C. ¹H NMR in CDCl₃, δ(ppm) : 3.1, s, 12H, -CH₃; 3.5, s, 8H, -CH₂-. ¹³C NMR in CDCl₃, δ(ppm) : 34.86, N-CH₃, 46.12, N-CH₂-, 157.56, C=O. IR (cm⁻¹) 1598 (C=O), 1284 (C-N). Calc. Anal. for (C₆H₁₀N₂O₂)_n: C, 50.69; H, 7.09; N, 19.71. Found: C, 50.61; H, 7.12; N, 19.63%. Mass spectrum (m/z) 56, 71, 86, 142, 284.

2.3. Synthesis of the complex {[Fe(oxalate)₃](diMen)₂·2(H₂O)}

Into an aqueous solution (5 mL) of FeCl₂ (0.1586 g, 1.25 mmol) was added a methanolic solution (10 mL) of the ligand L (0.3554 g, 1.25 mmol). The resulting mixture is heated at reflux for one hour. The brown solution was filtered and then allowed to evaporate slowly in an open atmosphere.

After two weeks, brown crystals suitable for X-ray analysis were obtained. The crystals were separated, washed with cold methanol and dried (yield: 0.37 g, 55.2%); Calc. Anal. for $C_{14}H_{32}FeN_4O_{14}$: C, 31.36; H, 6.01; N, 10.45 %. Found: C, 31.41; H, 6.03; N, 10.59 %. Selected IR data (cm^{-1} , KBr pellet): 3335, 1635, 1601, 1580, 1193, 765. $A_m=169\text{ cm}^2\text{ }\Omega^{-1}\text{ mol}^{-1}$ in DMF solution. Electronic spectrum in DMF solution [(λ, nm) ($\epsilon, M^{-1}\text{ cm}^{-1}$): 440 (780), 850 (110). $\mu_{\text{eff}}=4.94\text{ }\mu_B$.

2.4. X-ray Diffraction

Slow evaporation of methanol solution gave X-ray quality crystals of the compound. The details of the X-ray crystal structure solution and refinement

are given in **Table I**. Measurements were made on a STOE IPDS diffractometer. The structures were solved by direct methods SIR97^[9] and expanded using Fourier techniques. Least-square refinements were carried out using SHELXL97^[10]. All of the non-hydrogen atoms were refined anisotropically. The water H-atoms were located in difference Fourier maps and refined isotropically. Hydrogen ammonium groups were located in difference Fourier maps and were constrained to have idealized geometry of N-H = 0.92 Å. All calculations were performed using the Crystal-Structure crystallographic software package^[11]. Molecular graphics were generated using ORTEP-3^[12]. Selected bond lengths and bond angles are listed in **Table II**.

Table I: Crystallographic data and structure refinement for the complex

Empirical formula	$C_{14}H_{32}FeN_4O_{14}$
Formula weight	536.29
Crystal size/mm ³	0.28 x 0.2 x 0.15
Crystal colour	brown
Crystal system	Monoclinic
Space group	$P2_1/n$
<i>a</i> / Å	12.5822(7)
<i>b</i> / Å	13.3774(9)
<i>c</i> / Å	13.9146(7)
β / °	101.158(4)
<i>V</i> / Å ³	2297.8(2)
<i>Z</i>	4
<i>D_c</i> / g cm ⁻³	1.550
Radiation (MoK α), λ / Å	0.71073
Temperature/K	180(2)
Absorption correction	Numerical
μ / mm ⁻¹	0.732
F(000)	1128
Diffractometer	STOE IPDS
Theta range for data collection (°)	2.00-27.99
Index ranges (<i>h, k, l</i>)	-16, 16; -16, 17; -18, 16
Measured reflections	17379
Observed reflections, $I > 2\sigma(I)$	5503
<i>R_{int}</i>	0.0363
Completeness to theta = 27.99°	99.1 %
<i>T_{min}, T_{max}</i>	0.7534, 0.8686
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5503/6/314
Goodness-of-fit on F^2	1.060
<i>R, wR</i> ($I > 2\sigma(I)$)	0.0320, 0.0794
<i>R, wR</i> (all data)	0.0395, 0.0824
Largest diff. peak and hole (e.Å ⁻³)	0.568, -0.417

Table II: Selected bond distances [Å] and angles [deg] for the complex.

Fe(1)-O(9)	2.061(1)	O(2)-C(2)	1.267(2)	O(9)-C(5)	1.264(2)
Fe(1)-O(6)	2.064(1)	O(3)-C(1)	1.244(2)	O(10)-C(6)	1.265(2)
Fe(1)-O(1)	2.071(1)	O(4)-C(2)	1.233(2)	O(11)-C(6)	1.236(2)
Fe(1)-O(2)	2.099(1)	O(5)-C(3)	1.257(2)	O(12)-C(5)	1.242(27)
Fe(1)-O(10)	2.105(1)	O(6)-C(4)	1.268(2)	C(1)-C(2)	1.560(2)
Fe(1)-O(5)	2.114(1)	O(7)-C(4)	1.228(2)	C(3)-C(4)	1.562(2)
O(1)-C(1)	1.253(2)	O(8)-C(3)	1.244(2)	C(5)-C(6)	1.552(2)
O(9)-Fe(1)-O(6)	95.11(4)	O(1)-Fe(1)-O(2)	79.40(4)	O(9)-Fe(1)-O(5)	98.45(4)
O(9)-Fe(1)-O(1)	170.55(4)	O(9)-Fe(1)-O(10)	79.54(4)	O(6)-Fe(1)-O(5)	79.41(4)
O(6)-Fe(1)-O(1)	91.44(4)	O(6)-Fe(1)-O(10)	93.46(4)	O(1)-Fe(1)-O(5)	89.43(5)
O(9)-Fe(1)-O(2)	94.77(4)	O(1)-Fe(1)-O(10)	93.30(4)	O(2)-Fe(1)-O(5)	94.07(4)
O(6)-Fe(1)-O(2)	168.87(4)	O(2)-Fe(1)-O(10)	93.36(4)	O(10)-Fe(1)-O(5)	172.43(4)

Table III: Hydrogen bonds for the complex [\AA and deg].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1W)-H(1WA)...O(6)#1	0.851(10)	1.920(12)	2.757(2)	168(4)
O(1W)-H(1WB)...O(4)	0.855(10)	2.023(12)	2.856(2)	164(3)
O(2W)-H(2WA)...O(7)#2	0.854(9)	1.964(14)	2.747(2)	152(2)
O(2W)-H(2WA)...O(8)#2	0.854(9)	2.41(2)	3.049(2)	132(2)
O(2W)-H(2WB)...O(11)#1	0.850(9)	1.836(10)	2.6810(2)	173(3)
N(1)-H(1A)...O(2)	0.92	1.88	2.766(2)	161.6
N(1)-H(1B)...O(11)#3	0.92	1.93	2.738(2)	144.8
N(1)-H(1B)...O(12)#3	0.92	2.38	3.092(2)	133.9
N(2)-H(2A)...O(4)#4	0.92	2.04	2.894(2)	154.1
N(2)-H(2A)...O(3)#4	0.92	2.22	2.782(2)	119.1
N(2)-H(2B)...O(10)#1	0.92	1.91	2.824(2)	169.3
N(3)-H(3A)...O(2W)#5	0.92	1.78	2.666(2)	161.2
N(3)-H(3B)...O(3)#6	0.92	1.83	2.654(2)	148.3
N(3)-H(3B)...O(1)#6	0.92	2.61	3.309(2)	133.2
N(4)-H(4A)...O(12)#7	0.92	1.89	2.791(2)	165.1
N(4)-H(4A)...O(9)#7	0.92	2.46	3.139(2)	130.8
N(4)-H(4B)...O(8)	0.92	1.79	2.709(2)	173.4

Symmetry transformations used to generate equivalent atoms: #1 $x+1/2, -y+1/2, z+1/2$; #2 $x+1, y, z$; #3 $-x+1, -y, -z$; #4 $-x+3/2, y-1/2, -z+1/2$; #5 $x-1/2, -y+1/2, z+1/2$; #6 $-x+1/2, y-1/2, -z+1/2$; #7 $-x+1/2, y+1/2, -z+1/2$.

3. Results and discussion

3.1. Spectroscopic studies and ligand decomposition

The reaction of dimethyloxalate and N, N'-dimethyl-1,2-aminoethane in methanol at room temperature yield a quantitative white precipitate. The ^1H NMR spectrum in CDCl_3 confirms the condensation reaction. Only two singlets assigned to the N-methyl group and the methylene group - CH_2 - are observed. The ^{13}C NMR spectrum shows three signals corresponding to one amide group, N-methyl carbon and methylene carbon, confirming the proposed structure. The absorption bands observed in the IR spectrum are consistent with the functional groups present in the molecule. The electron impact mass spectrum shows a series of peaks, i.e. 56 amu, 71 amu, 86 amu and 142 amu corresponding to various fragments. Their intensity gives an idea of stability of fragments. The spectrum shows also a low intensity peak corresponding to the molecular ion mass at 284 amu.

The ligand prepared by the reaction of dimethyloxalate with N,N'-dimethyl-1,2-diaminoethane is used for preparing complex. The production of the N,N'-dimethyl-1,2-diammonium ethane and oxalate on reaction with FeCl_2 in methanol indicates ligand hydrolysis at the amide sites. Pre-coordination of iron(II) ions to the ligand would lead to Lewis acid promoted nucleophilic attack by water at the amide sites (Scheme 1), thus producing oxalate, which could then simply complex the iron(II) center. The N,N'-dimethyl-1,2-diammonium ethane could acts as counter ion. Similar hydrolytic decompositions have been

reported in the literature [13, 14, 23]. This effect is original because the tentative to isolate analogues from oxalate salt and free diamine does not work. The complex characterized is consistent with the stoichiometry proposed and is insensitive to air and moisture. The reaction yields a coloured solution which give crystal suitable for X-ray analysis after several days of slow evaporation. The analytical data show subsequent modification of the reactant.

The most important bands of the ligand were assigned according to published data [14]. The absence of any bands in the $3400\text{--}3000\text{ cm}^{-1}$ region of the IR spectrum of L suggests the absence of any N-H amine. The spectrum of the complex exhibits bands in the $3400\text{--}3050\text{ cm}^{-1}$ region, which indicate neutral or total protonation of amine fragment from ligand decomposition [16]. These bands in the free amine stretching region, confirms that the amine fragment is not bonded to the metal ion. In the spectrum of the complex in which the secondary diamine fragment from the ligand is present in its protonated form, the band assigned to $\delta(\text{NH})$ appears at 3052 cm^{-1} suggesting his no bonding to the metal ion. These observations are consistent to the analytical data of complex. The spectrum exhibits also band of medium intensity at 3376 cm^{-1} originate from the O-H stretching vibration $\nu(\text{OH})$ of lattice water molecules. Bands which could be related to the bidentate oxalate moiety are observed. These bands are located at 1652 cm^{-1} ($\nu_{\text{as}}(\text{C}=\text{O})$), 1357 and 1315 cm^{-1} ($\nu_{\text{s}}(\text{C}=\text{O})$) and 747 cm^{-1} for $(\delta(\text{OCO}))$ [17]. The electronic spectrum recorded in DMF exhibits different patterns. In the UV region, an intense band centered at 440 nm has been appeared. This band can be assigned to the ligand to metal charge transfer (LMCT) and internal oxalate ligand transitions. In the visible region of the

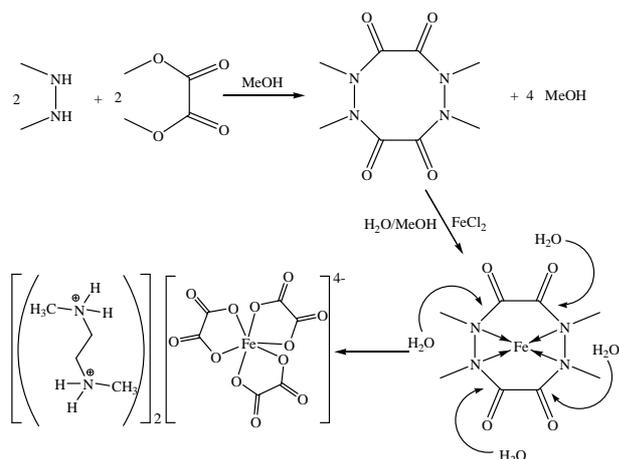


Chart 1

spectrum of the complex, a weak intensity band at 850 nm assigned to ${}^5T_{2g} \rightarrow {}^5E_g$ is observed. This is consistent with an octahedral coordination around the iron(II) ion [18, 19]. The value of the molar conductance in DMF solution ($169 \text{ cm}^2 \Omega^{-1} \text{ mol}^{-1}$) support the formulation $[\text{Fe}(\text{C}_2\text{O}_4)_3]^{4-} [(\text{MeH}_2\text{N}(\text{CH}_2)_3\text{NH}_2\text{Me})^{2+}]_2$ as a 2:1 electrolyte [20] (expected range for 2:1 electrolyte in DMF : $130\text{--}170 \text{ cm}^2 \Omega^{-1} \text{ mol}^{-1}$). Also, the magnetic moment value ($4.94 \mu_B$) for the Fe(II) complex is indicative of high-spin electronic configuration of the metal ion ($S=2$). This value is slightly lower than the expected spin-only value for high-spin d^6 complexes [21].

All these spectral suggestions have been confirmed by the X-ray structural determination.

3.2. Electrochemistry

While scanning from -0.35 V to $+0.2 \text{ V}$ with a potential rate of 0.02 V (Figure 1), the mononuclear species $[\text{Fe}(\text{C}_2\text{O}_4)_3]^{4-} [(\text{MeH}_2\text{N}(\text{CH}_2)_3\text{NH}_2\text{Me})^{2+}]_2$

displays any peaks in DMF solution. One pair of definite cathodic and anodic peaks with peaks potentials $E_{pc} = -0.148 \text{ V}$ and $E_{pa} = +0.004 \text{ V}$ and the half potential $E_{1/2}$ was calculated to be -0.76 V . The peak potential separation ΔE was 0.152 V and remains constant when increasing the scan rate. The ratio of anodic to cathodic peak currents was close to unity when the scan rate is 0.02 Vs^{-1} . The plot of i_{pc} (or i_{pa}) against the square root of potential scan rate $v^{1/2}$ is a straight line. This fact is indicative of quasi-reversible electrode process $\text{Fe}^{\text{III}}/\text{Fe}^{\text{II}}$.

3.3. Thermal study

A simultaneous TG and DTA studies of the complex is carried out in the stream of synthetic air from room temperature to $900 \text{ }^\circ\text{C}$ and show weight loss in two steps, and the corresponding curves are presented in Figure 2. The first mass loss of 7.05% (calc. 6.72%) for the compound corresponds to the removal of both water molecules and occurs over the temperature range $40\text{--}217\text{ }^\circ\text{C}$. Only one step for removal water molecules is observed. As found in crystallographic structure only lattice water molecule is present. A rapid weight loss occurring between 217 and $715\text{ }^\circ\text{C}$, corresponds to 74.09% (calc. 73.90%). This is indicative of decomposition comprising the removal of two N,N -dimethylammoniummethane together with the three oxalate moieties ($3\text{CO} + 3\text{CO}_2$). The remaining mass stays constant and corresponds to iron(II) oxide Fe_2O_3 (exp. 18.86% ; calc. 19.38%). No other amine-containing iron oxalates have been reported in the literature; however manganese, cobalt, copper and zinc oxalates containing amines have been published [22, 23]. The DTA curve gives the corresponding exothermic peaks of the two processes, one centered at $375\text{ }^\circ\text{C}$ and the other around $657\text{ }^\circ\text{C}$.

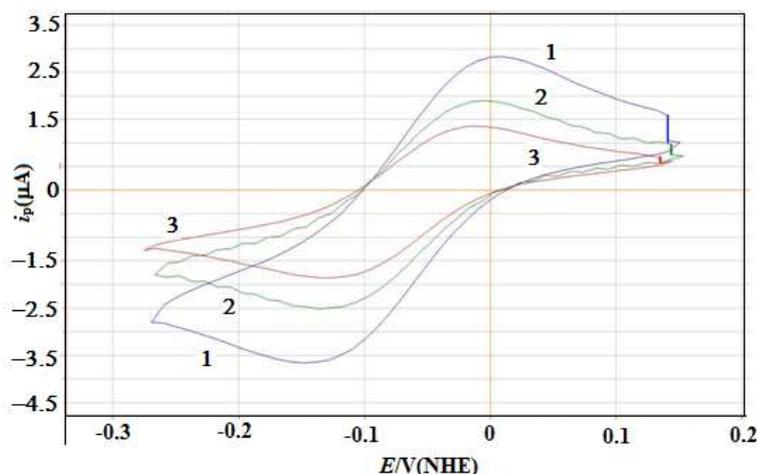


Figure 1: Cyclic voltammograms of the iron complex in DMF solution. $[\text{Complex}] = 10^{-2} \text{ mol dm}^{-3}$, $[\text{TBAHFP}] = 0.1 \text{ mol dm}^{-3}$. Scan rate = 0.1 Vs^{-1} (curve 1); 0.04 Vs^{-1} (curve 2) and 0.02 Vs^{-1} (curve 3).

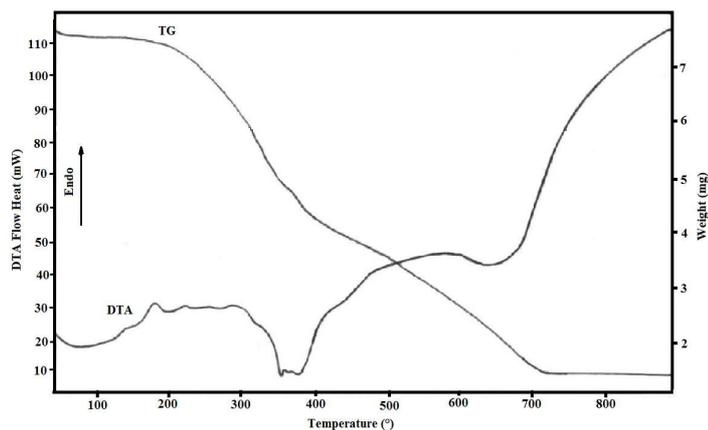


Figure 2: TG and DTA curves for $\{[\text{Fe}(\text{oxalate})_3](\text{diMen})_2 \cdot 2(\text{H}_2\text{O})\}$, showing the weight loss.

3.4. Structure Description of the complex

The hydrolytically unstable cyclic ligand $(\text{C}_6\text{H}_{10}\text{N}_2\text{O}_2)_n$, is hydrolyzed in the presence of iron ions giving oxalate dianion. These species, which are generated *in situ*, act with iron(II) ions resulting in formation of di $[\text{N}, \text{N}'\text{-dimethyldiammonium}]$ trisoxalato iron(II) dihydrate, $[(\text{C}_4\text{H}_{14}\text{N}_2)_2\text{Fe}(\text{C}_2\text{O}_4)_3 \cdot (\text{H}_2\text{O})_2]$ which was obtained as an unexpected product. The asymmetric unit of the complex is shown in Figure 3. In this title complex, prepared from the hydrolytically unstable $(\text{C}_6\text{H}_{10}\text{N}_2\text{O}_2)_n$ Schiff base ligand, the Fe^{II} atom is coordinated by three oxalate moieties through the O atom resulting in a distorted octahedral geometry^[24]. The structure exhibits an ion-pair complex comprising a $[\text{Fe}(\text{ox})_3]^{4-}$ tetraanion, two cations of dimethylethylenediammonium and crystallization water molecules linked together by means of hydrogen bonds (Figure 4c). The anionic unit contains one Fe(II) atom and three terminal oxalato ligands. The iron-terminal-oxalato bond lengths

$[2.0614(11)\text{-}2.1145(11) \text{ \AA}]$ are significantly longer than those found for the complex $[(\text{N}_2\text{C}_4\text{H}_{12})[\text{Fe}_4(\text{C}_2\text{O}_4)_3(\text{HPO}_4)_2(\text{H}_2\text{O})_2]]^{[25]}$.

The terminal oxalato are not planar owing to the torsion angles values O(1)-C(1)-C(2)-O(2), O(3)-C(1)-C(2)-O(4), O(5)-C(3)-C(4)-O(6), O(7)-C(4)-C(3)-O(8), O(10)-C(6)-C(5)-O(9) and O(11)-C(6)-C(5)-O(12) respectively $-5.0(2)$, $-5.6(2)$, $-6.8(2)$, $-7.0(2)$, $-7.1(18)$ and $-8.0(2)^\circ$. Protonated amine molecules interact with the oxalate groups through hydrogen bonds. These multiple hydrogen bonds contribute to stabilizing the three-dimensional structure of the compound. The hydrogen bond interactions in the complex between the oxalate moieties and the protonated amine groups are quite strong. The water lattice – OH group unit participate in intra- and intermolecular hydrogen O—H...O_{oxalate} bonding. Hydrogen bond interactions are listed in Table III. The packing diagram of the title salt with hydrogen bonds is shown in Figure 4c. The packing diagrams of the title compound as viewed along *a* and *b* axis are respectively shown in Figure 4a and Figure 4b.

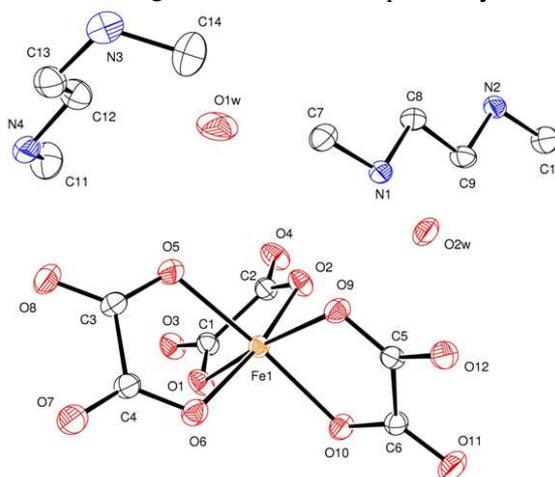


Figure 3: ORTEP plot (30% probability ellipsoids) showing the molecule structure of the complex.

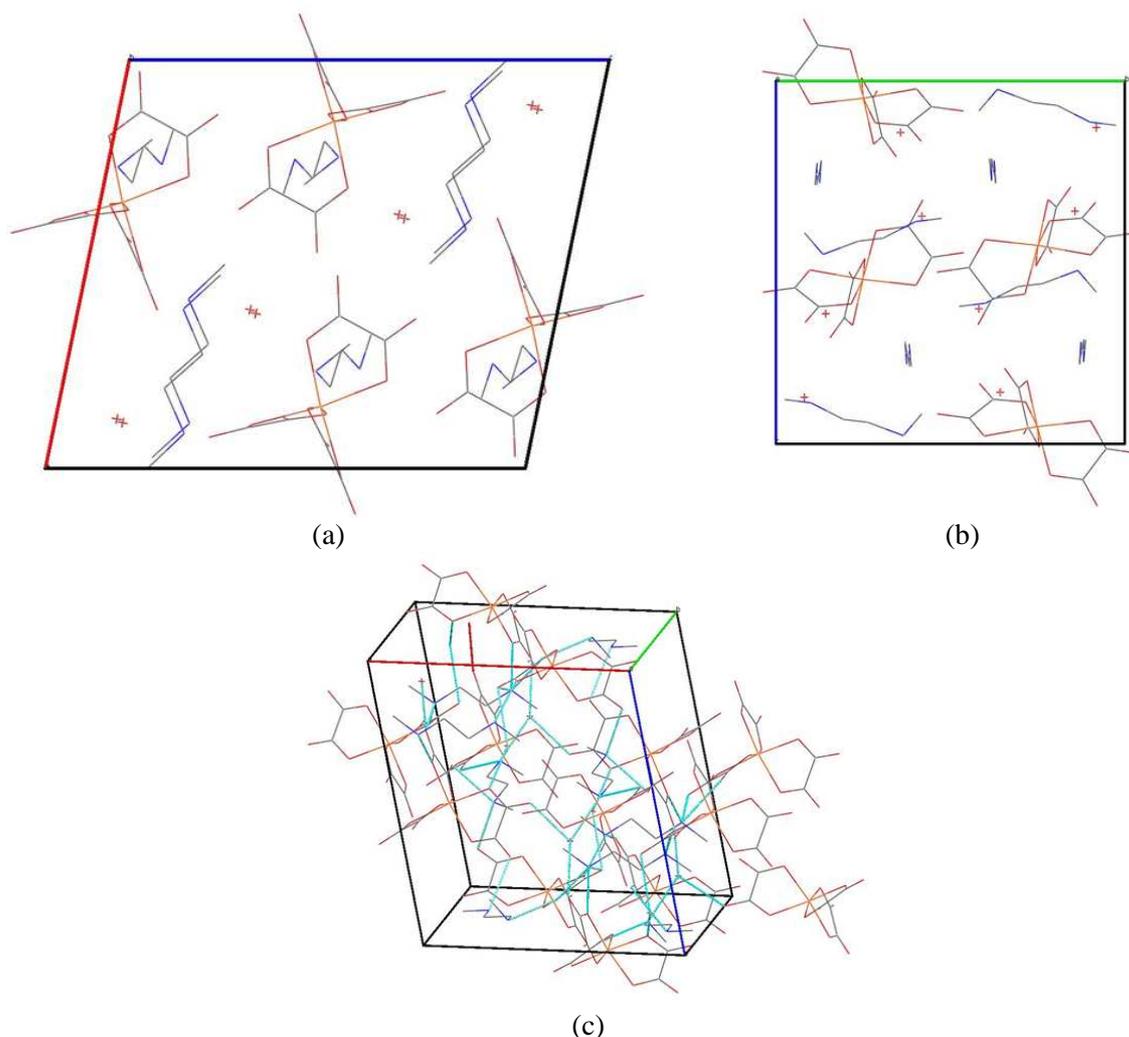


Figure 4: (a) Crystal packing of the complex as viewed along *a* axis ; (b) Crystal packing of the complex as viewed along *b* axis; (c) The 3D crystal packing with hydrogen bonds.

4. Conclusion

The cyclic Schiff base ligand derived from dimethyloxalate and *N,N'*-dimethyl-1,2-diaminoethane in presence of Fe(II) salt undergoes hydrolysis producing oxalate moieties and *N,N'*-dimethyl-1,2-diammonium. The oxalate is able to coordinate iron(II) as bidentate ligand. The six-coordinate complex possesses one $[\text{Fe}(\text{ox})_3]^{4-}$ cation. The structural analysis shows that *N,N'*-dimethyl-1,2-diaminoethane does not appear in the coordination sphere of the structure, but it is protonated as *N,N'*-dimethyl-1,2-diammonium and is present as counter ion in the compounds. Characterization of solutions of the obtained complex by spectroscopic and conductivity techniques, magnetic measurement, electrochemistry and thermal study is consistent with the solid-state X-ray crystal structure of the complex. Hydrogen bonding interactions contribute to the formation of a 3D structure.

5. Supporting Information

Supplementary data CCDC 883885 contains the supplementary crystallographic data for the complex. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

6. REFERENCES

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