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Complexes of Iron(III) and Chrom(III) Salen and Saloph Schiff Bases with Bridging 2,4,6-tris(4-nitrophenylimino-4'-formylphenoxy)-1,3,5-triazine

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Four new trinuclear Fe(III) and Cr(III) complexes involving tetradenta Schiff bases N,N'-bis(salicylidene)ethylenediamine-(salenH₂) or bis(salicylidene)-o-phenylenediamine-(saloph H₂) with 2,4,6-tris(4-nitrophenylimino-4'-formylphenoxy)-1,3,5-triazine (**TNPI-TRIPOD**) have been synthesized and characterized by means of elemental analysis carrying out ¹H-NMR., IR spectroscopy, thermal analyses and magnetic susceptibility measurements. The complexes can also be characterized as high-spin distorted octahedral Fe(III) and Cr(III) bridged by nitro. The nitro play a role as bridges for weak anti-ferromagnetic intramolecular exchange.

Keywords: Schiff base, triazine, cyanuric chloride, salen, saloph, metal complexes

1. Introduction

An important class of compounds consists of substituted s-triazine derivatives which have anticancer, antitumor, antiviral and antifungal activity. These compounds have been used in the treatment of depression and hence, gained a considerable importance. These are valuable bases for estrogen receptor modulators and also used as bridging agents to synthesize herbicides and in the production of drugs or polymers (1,2).

We have reported here that a trialdehyde and its Schiff bases have been synthesized to be a new template. The reaction of trimeric cyanuric chloride (C₃N₃Cl₃) with 3 equiv. of 4-hydroxybenzaldehyde in benzene has given the desired trialdehyde in a single step, coded to be **TRIPOD**. Trialdehyde (3,4) then reacted with 4-nitroanilin and 2,4,6-tris(4-nitrophenylimino-4'-formylphenoxy)-1,3,5-triazine (**TNPI-TRIPOD**) under reflux. It may be useful to stress at this point that the new products mentioned above are the main result of this work. These are the first in the literature and we call them "Oxy-Schiff Bases" due to the literature (1).

Schiff bases of iron(III) have been known since 1938. The magnetochemical properties of the μ -oxo-bridged complexes [$\{\text{Fe}(\text{salen})\}_2\text{O}$] [(salenH₂ = N,N'-bis(salicylidene) ethylenediamine)] and [$\{\text{Fe}(\text{saloph})\}_2\text{O}$]

[(salophH₂ = bis(salicylidene)-2-phenylenediamine)] and their X-ray studies have widely been presented in the literature (5–9). Kessel and Hendrickson (10) have studied Schiff base complexes of iron(III) incorporating p-quinone bridges. The reaction of [$\{\text{Fe}(\text{salen})\}_2\text{O}$] with carboxylic acids have been given by Wollmann and Hendrickson (11). They used trichloroacetic, trifluoroacetic, salicylic and picric acids, and characterized the prepared complexes as dimers of composition [$\text{Fe}(\text{salen})\text{X}$]₂, where X is monoanion of the appropriate acid. The complexes [$\{\text{Fe}(\text{salen})\}_2\text{L}$] and [$\{\text{Fe}(\text{saloph})\}_2\text{L}$] (where L = terephthalate, fumarate, oxalate and succinate dianion) have been prepared. The crystal and molecular structure of [$\{\text{Fe}(\text{salen})\}_2\text{ter}$] (H₂ter = terephthalic acid) have been reported (12). Other complexes of composition [$\{\text{Fe}(\text{salen})\}_2\text{L}$] (where L = glutarate, adipate, pimelate, suberate and dithiooxamidedianion) were prepared by Smekal et al. (13). Therefore, in this work, we aimed to make other 1,3,5-trinitro bridges and to present their certain influences on the magnetic behavior of the prepared complexes. We were also interested in Tripodal-Trinuclear systems formed by the 1,3,5-trinitro bridges because no satisfactory work dealing with this bridge and associated data have appeared in the literature.

2. Experimental

Elemental analyses were performed on a Carlo Erba 1106 elemental analyzer. The IR spectra were recorded using KBr discs (4000–440 cm⁻¹) on a Perkin-Elmer 1600

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series FT-IR spectrophotometer. The $^1\text{H-NMR}$ spectra in CDCl_3 , $d_6\text{-DMSO}$ was obtained using a Bruker 200 MHz spectrometer. MMM-Medcenter, Einrichtungen GmbH Vacucell 22, Vacuum Cabinets. Melting points were measured using a Buchi SMP-20 melting point apparatus. The thermal analyses were performed on Shimadzu DTA 50 and TG 50 H models using 10 mg samples. The DTA and TG curves were obtained at a heating rate of $10^\circ\text{C min}^{-1}$. In all cases, the $22\text{--}750^\circ\text{C}$ temperature range was studied under a dry nitrogen atmosphere. All other chemicals were purchased from Aldrich and used as received. The Trialdehyde (TRIPOD) [2,4,6-tris(p-formylphenoxy)-1,3,5-triazine] were prepared by established literature procedures (3). Magnetic Susceptibilities of metal samples were determined using a Sheerwood Scientific MX Gouy magnetic susceptibility apparatus and magnetic measurements were carried out using the Gouy method with $\text{Hg}[\text{Co}(\text{SCN})_4]$ as calibrant. The effective magnetic moments, μ_{eff} , per metal atom were calculated from the expression: $\mu_{\text{eff}} = 2.84\sqrt{\chi_M T B.M.}$, where χ_M is the molar susceptibility.

2.1. Preparation of ligand complexes

$[\{\text{Fe}(\text{salen})\}_2\text{O}]$, $[\{\text{Fe}(\text{saloph})\}_2\text{O}]$, $[\{\text{Cr}(\text{salen})\}_2\text{O}]$ and $[\{\text{Cr}(\text{saloph})\}_2\text{O}]$ were prepared by adding a concentrated ammonia solution to stirred hot EtOH solutions of $[\text{Fe}(\text{salen})\text{Cl}]$, $[\text{Fe}(\text{saloph})\text{Cl}]$, $[\{\text{Cr}(\text{salen})\}_2\text{Cl}]$ and $[\{\text{Cr}(\text{saloph})\}_2\text{Cl}]$, respectively, until they became alkaline (5,14).

2.2. The synthesis procedure for 2,4,6-tris(4-nitrophenylimino-4'-formylphenoxy)-1,3,5-triazine (TNPI-TRIPOD)

A suspension of TRIPOD (0.44 g, 1 mmol) in 10 mL of MeOH was added to a solution of 4-nitroaniline (0.42 g, 3 mmol) in 30 mL of MeOH, 2.5 mL of 1 M HCl/MeOH, and 3-A molecular sieves. The mixture was then boiled under reflux for 24 h. The reaction solution was left stirring overnight. After 10 min, the suspension became thicker as a yellow fluffy precipitate formed. IR indicated the disappearance of aldehyde. The mixture was centrifuged and the solvent was pipetted off. The product was washed with MeOH and hot AcOEt. and then recrystallized with benzene. The solvent was removed and the resulting solid was filtered off, dried in a vacuum cabinet and stored in a desiccator over CaCl_2 . *TNPI-TRIPOD* FT-IR(cm^{-1}) 1615 (CH=N), 1565 (C=N triazine), 1573 (NO_2), 1385 (COC). $^1\text{H-NMR}$ (CDCl_3) δ 8.50 (s, 3H), 6.96 (d, 6H, $j = 8.50$ Hz), 7.45 (d, 6H, $j = 8.50$ Hz), 6.85 (d, 6H), 8.10 (d, 6H).

2.3. $[\{\text{Fe}(\text{salen})\}_3(\text{TNPI-TRIPOD})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$ or $[\{\text{Cr}(\text{salen})\}_3(\text{TNPI-TRIPOD})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$ Complexes

$[\{\text{Fe}(\text{salen})\}_2\text{O}]$ or $[\{\text{Cr}(\text{salen})\}_2\text{O}]$ (1.5 mmol, 0.99 g or 1.5 mmol 0.98 g) was suspended in hot EtOH (25 mL) and a solution of 2,4,6-tris(4-nitrophenylimino-4'-formylphenoxy)-1,3,5-triazine (0.80 g, 1 mmol) in EtOH was added by stirring. The reaction mixture was boiled under reflux for 4 h, and the solid formed was dried under vacuum. $[\{\text{Fe}(\text{salen})\}_3(\text{TNPI-TRIPOD})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$ FT-IR (cm^{-1}): 3475 (OH), 2850 (CH_2), 1585 (CH=N), 1545 (C=N triazine), 1535 (N=O), 1380 (COC), 544 (M-N), 488 (M-O). $[\{\text{Cr}(\text{salen})\}_3(\text{TNPI-TRIPOD})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$ FT-IR (cm^{-1}): 3470 (OH), 2843 (CH_2), 1590 (CH=N), 1549 (C=N triazine), 1545 (N=O), 1369 (COC), 542 (M-N), 475 (M-O).

2.4. $[\{\text{Fe}(\text{saloph})\}_3(\text{TNPI-TRIPOD})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$ or $[\{\text{Cr}(\text{saloph})\}_3(\text{TNPI-TRIPOD})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$ Complexes

The $[\{\text{Fe}(\text{saloph})\}_2\text{O}]$ or $[\{\text{Cr}(\text{saloph})\}_2\text{O}]$ complexes were prepared in the same way as mentioned above for the $[\{\text{Fe}(\text{salen})\}_2\text{O}]$ or $[\{\text{Cr}(\text{salen})\}_2\text{O}]$ complexes using EtOH as solvent for $[\{\text{Fe}(\text{saloph})\}_3(\text{TNPI-TRIPOD})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$ or $[\{\text{Cr}(\text{saloph})\}_3(\text{TNPI-TRIPOD})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$. $[\{\text{Fe}(\text{saloph})\}_3(\text{TNPI-TRIPOD})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$ FT-IR (cm^{-1}): 3465 (OH), 1598 (CH=N), 1546 (C=N triazine), 1540 (N=O), 1378 (COC), 545 (M-N), 473 (M-O). $[\{\text{Cr}(\text{saloph})\}_3(\text{TNPI-TRIPOD})]\text{Cl}_3 \cdot 3\text{H}_2\text{O}$ FT-IR (cm^{-1}): 3480 (OH), 1580 (CH=N), 1540 (C=N triazine), 1550 (N=O), 1382 (COC), 550 (M-N), 482 (M-O).

3. Results and discussion

2,4,6-Tris(p-formylphenoxy)-1,3,5-triazine (TRIPOD) was prepared through the reaction of cyanuric chloride and 4-hydroxybenzaldehyde (Fig. 1). The structural formula of the 2,4,6-Tris(4-formylphenoxy)-1,3,5-triazine (**1**) was verified by elemental analyses, $^1\text{H-NMR}$, IR and mass spectral data (3) (Fig. 1). Condensation of 2,4,6-Tris(p-formylphenoxy)-1,3,5-triazine (**1**) with amine (**2**) was readily furnished and was a result of this reaction, "Tripodal Oxy-Schiff base" has been obtained. The Tripodal Oxy-Schiff base was obtained and characterized by their elemental analyses, thermal analyses, $^1\text{H-NMR}$ and IR, where replacement of the carbonyl by the imine group causes lowering of the energy of the C=O stretch in the IR spectrum and a shift to a higher field of the CH=N proton signal in the $^1\text{H-NMR}$ spectrum. The "Oxy-Schiff base" prepared in this way has been obtained in nearly quantitative yield and high purity (Scheme 1). The ligand is soluble in common organic solvents. The synthetic strategy for preparing Tripodal-Trinuclear uses a complex as a "ligand"

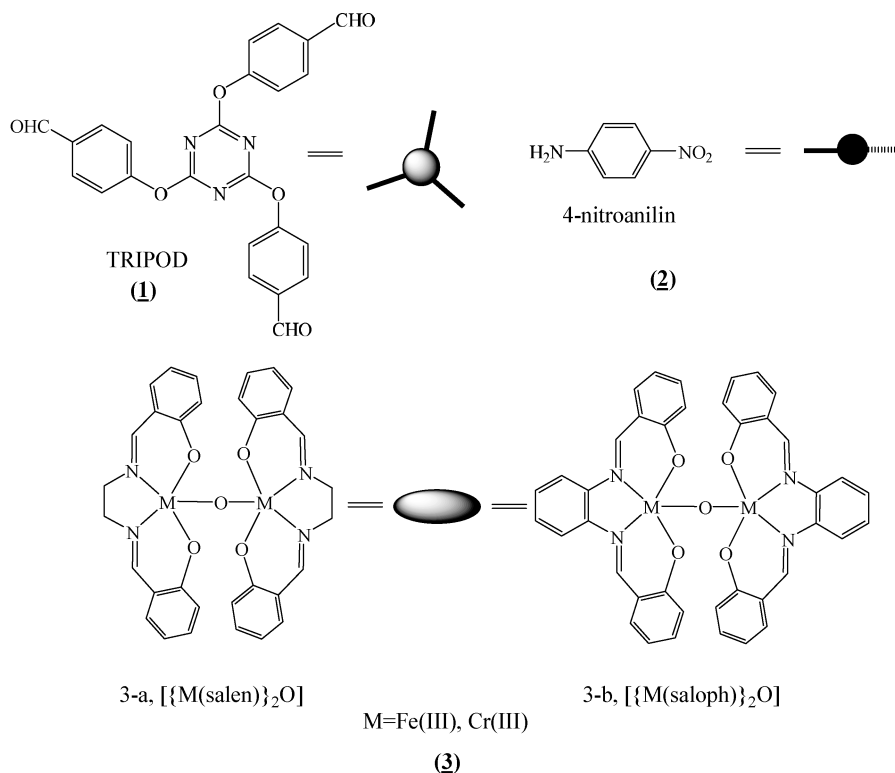


Fig. 1. Structure of TRIPOD, amine and “ligand complexes.”

that contains a potential donor group capable of coordinating to another ligand. We have chosen $[\{\text{Fe}(\text{salen})\}_2\text{O}]$, $[\{\text{Fe}(\text{saloph})\}_2\text{O}]$, $[\{\text{Cr}(\text{salen})\}_2\text{O}]$ and $[\{\text{Cr}(\text{saloph})\}_2\text{O}]$ as “ligand complex” because it can coordinate another ligand (15). These complexes are the first examples of Tripodal-Trinuclear complexes bridged by nitro anions to the iron and chromium centers (16–18) (Scheme 1 and Fig. 2). All compounds are stable at room temperature in the solid state. The results of the elemental analyses, given in Table 1, are in a good agreement with the structures suggested for the ligand and their complexes. The results show that all complexes are trinuclear (Fig. 2). All complexes are also stable at room temperature and they are only soluble in organic solvents such as DMSO, DMF and insoluble in water.

The vibrations of the azomethine C=N, triazine C=N and N=O of the free ligand have been observed at 1615, 1565 and 1573 cm^{-1} range, respectively, (19,20). In the complexes, these bands are, however, shifted to lower frequencies, indicating that the nitrogen and oxygen atoms of the Tripodal Oxy-Schiff bases ligand are coordinated to the ligand complexes. In the ligand 2,4,6-tris(4-nitrophenylimino-4'-formylphenoxy)-1,3,5-triazine (TNPI-TRIPOD), the band at 1573 cm^{-1} can be assigned to the NO_2 group vibrations (21). In the Tripodal-Trinuclear complexes, these bands disappear, suggesting chelation of oxygen to the metal. In the complexes, broad bands in the 3480–3465 cm^{-1} range are assigned to the OH stretch of hydrated

water molecules. In the Tripodal-Trinuclear complexes, the bands in the 540–550 and 470–488 cm^{-1} range can be attributed to the M–N and M–O stretching modes (22,23).

In order to identify the structures of solutions of the Tripodal Oxy-Schiff base ligand, the $^1\text{H-NMR}$ spectra were recorded in DMSO-d_6 (23,24). $^1\text{H-NMR}$ spectra also confirmed the structure of the synthesized compound. The signals in $^1\text{H-NMR}$ spectrum of 2,4,6-tris(4-nitrophenylimino-4'-formylphenoxy)-1,3,5-triazine (TNPI-TRIPOD) at δ 8.50 ppm correspond to the Oxy-Schiff base HC=N proton resonances.

The magnetic moments of the complexes given in Table 1 were measured at room temperature. On the basis of spectral evidence, the Tripodal Fe(III) and Cr(III) complexes have trinuclear structures in which the Fe(III) and Cr(III) cations have an approximately octahedral environment. The magnetic behavior of Fe(III) and Cr(III)

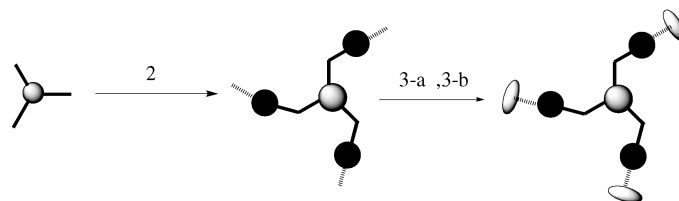
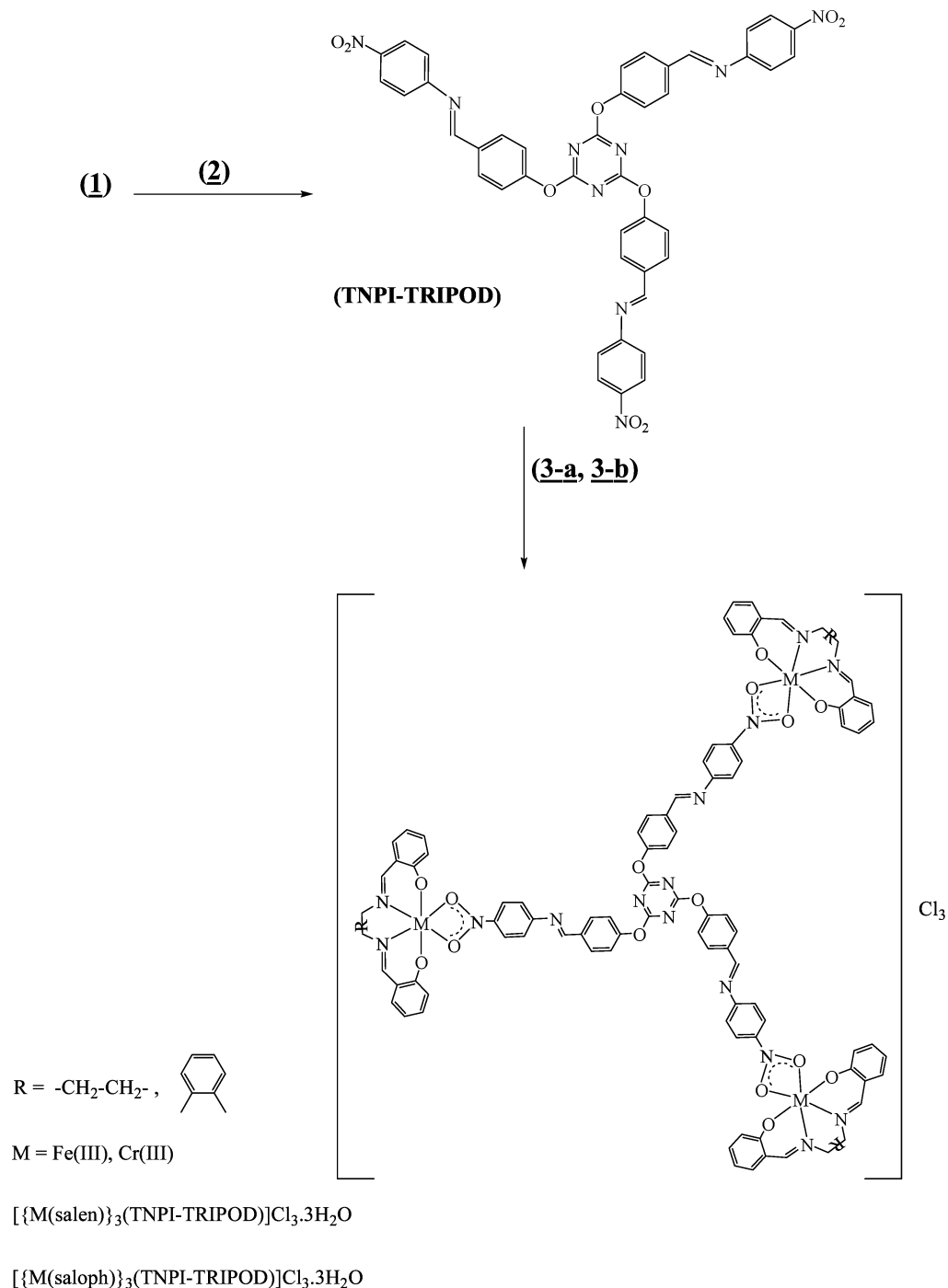


Fig. 2. Proposed structures of the Tripodal Oxy-Schiff base ligand and Tripodal-Trinuclear complexes.



Sch. 1. Synthesis of TNPI-TRIPOD and tripodal-trinuclear complexes.

complexes is in accord with proposed trinuclear structures (25). The magnetic moment per trinuclear complexes which were constructed from $[\{\text{Fe}(\text{salen})\}_2\text{O}]$, $[\{\text{Fe}(\text{saloph})\}_2\text{O}]$, $[\{\text{Cr}(\text{salen})\}_2\text{O}]$ and $[\{\text{Cr}(\text{saloph})\}_2\text{O}]$ either of 2,4,6-tris(4-nitrophenylimino-4'-formylphenoxy)-1,3,5-triazine (**TNPI-TRIPOD**) shows paramagnetic property with a magnetic susceptibility value per atom: 1.65–1.70 B.M. and 3.75–3.90 B.M., respectively. It is seen that the $[\{\text{Fe}(\text{salen})\}_2\text{O}]$, $[\{\text{Fe}(\text{saloph})\}_2\text{O}]$, $[\{\text{Cr}(\text{salen})\}_2\text{O}]$ and

$[\{\text{Cr}(\text{saloph})\}_2\text{O}]$ containing compounds are represented by the electronic structure of $t_2g^5eg^0$ and $t_2g^3eg^0$. The magnetic data for the $[\{\text{Fe}(\text{salen})\}_2\text{O}]$, $[\{\text{Fe}(\text{saloph})\}_2\text{O}]$, $[\{\text{Cr}(\text{salen})\}_2\text{O}]$ and $[\{\text{Cr}(\text{saloph})\}_2\text{O}]$ tripodal complexes show good agreement with the d^5 and d^3 metal ion in an octahedral structure. This consequence is supported by the results of the elemental analyses suggesting that these Tripodal complexes have also an octahedral structure (5).

Table 1. Colors, analytical data and magnetic properties for the compounds

Compound	Color	Yield (%)	M.p. (°C)	μ_{eff} (B.M.) 296 K	Found (Calcd.) (%)			
					C	H	N	M
TNPI-TRIPOD	Yellow	75	195	–	62.8 (62.9)	3.3 (3.4)	15.3 (15.7)	–
[{Fe(salen)} ₃ (TNPI-TRIPOD)]Cl ₃ ·3H ₂ O	Dark-brown	70	250	1.65	57.0 (57.6)	4.1 (3.7)	11.6 (11.2)	9.4 (8.9)
[{Fe(saloph)} ₃ (TNPI-TRIPOD)]Cl ₃ ·3H ₂ O	Red-brown	75	235	1.70	60.9 (60.6)	3.7 (3.4)	10.9 (10.4)	8.7 (8.4)
[{Cr(salen)} ₃ (TNPI-TRIPOD)]Cl ₃ ·3H ₂ O	Green	65	240	3.75	59.5 (58.5)	4.0 (3.7)	11.9 (11.3)	8.9 (8.4)
[{Cr(saloph)} ₃ (TNPI-TRIPOD)]Cl ₃ ·3H ₂ O	Dark-green	70	230	3.90	61.3 (61.0)	3.5 (3.4)	11.5 (10.9)	8.4 (7.9)

Table 2. Decomposition steps with the temperature range and weight loss for ligand and complexes

Compound	Temp. Range (°C)	Weight loss	
		Found (Calcd.) (%)	Fragment
TNPI-TRIPOD [Fe(salen)] ₃ (TNPI-TRIPOD)]Cl ₃ ·3H ₂ O	157–229	42.5 (42.7)	NO ₂ , N ₂ , CO
	60–120	58.4 (59.0)	H ₂ O, N ₂ , C ₂ H ₄ , H ₂ , CO, NO ₂
	230–325		
[Fe(saloph)] ₃ (TNPI-TRIPOD)]Cl ₃ ·3H ₂ O	330–450		
	60–120	50.7 (52.9)	H ₂ O, N ₂ , H ₂ , CO, NO ₂
	210–340		
[Cr(salen)] ₃ (TNPI-TRIPOD)]Cl ₃ ·3H ₂ O	345–470		
	60–122	57.3 (59.4)	H ₂ O, N ₂ , C ₂ H ₄ , H ₂ , CO, NO ₂
	200–285		
[Cr(saloph)] ₃ (TNPI-TRIPOD)]Cl ₃ ·3H ₂ O	305–430		
	60–125	56.9 (57.4)	H ₂ O, N ₂ , H ₂ , CO, NO ₂
	225–325		
	335–465		

The ligand and complexes have also been thermally investigated and their plausible degradation schemes are presented in Table 2 (26). It has been well known that there is a strong relation between the temperature range for the dehydration process and the binding mode of the water molecules of the respective metal complexes. The elimination of water takes place in a single step process attributed to the release of the hydrated water molecules (in the 60–120°C range) (27). Thermal decomposition of the anhydrous [{Fe(salen)}₂O], [{Fe(saloph)}₂O], [{Cr(salen)}₂O] and [{Cr(saloph)}₂O] complexes of the ligand (TNPI-TRIPOD) starts in the range of 200–450°C and completes in the range of 550–700°C. The final decomposition products are metal oxides. The observed weight losses for ligand and complexes are in good agreement with the calculated values.

4. Conclusions

In this work, we have synthesized and characterized, a new ligand, two ligand complexes and two new Fe(III), Cr(III) complexes of ligand. We have also investigated thermal

behaviors of Fe(III), Cr(III) complexes of ligand by DTA 50 and TG 50.

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