

# The synthesis and characterization of melamine based Schiff bases and its trinuclear [salen/salophenFe(III)] and [salen/salophenCr(III)] capped complexes

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**Abstract** Four new trinuclear Fe(III) and Cr(III) complexes involving tetradentate Schiff bases *N*, *N'*-bis(salicylidene)ethylenediamine-(salenH<sub>2</sub>) or bis(salicylidene)-*o*-phenylenediamine-(salophen H<sub>2</sub>) with 2,4,6-tris(4-carboxybenzimidino)-1,3,5-triazine have been synthesized and characterized by means of elemental analysis, <sup>1</sup>H NMR, FT-IR spectroscopy, LC-MS, thermal analyses and magnetic susceptibility measurements. The complexes have also been characterized as low-spin distorted octahedral Fe(III) and Cr(III) bridged by COO<sup>-</sup> group.

**Keywords** Melamine · Salen · Salophen · Schiff bases

## Introduction

Melamine is the common name of 2,4,6-triamino-*s*-triazine. Melamine is a trimer of cyanamide, H<sub>2</sub>NC≡N, synthesized from calcium carbide. It condenses with formaldehyde to give a thermosetting resin. Melamine resins have been used in many applications including the manufacture of plastic dishes under the trade name Melmac.

Schiff bases complexes of iron(III) have been known since 1938. The magnetochemical properties of the  $\mu$ -oxo-bridged complexes [Fe(salen)<sub>2</sub>O] [(salenH<sub>2</sub> = *N*, *N'*-bis(salicylidene) ethylenediamine)] and [Fe(salophen)<sub>2</sub>O] [(salophenH<sub>2</sub> = bis(salicylidene)-2-phenylenediamine)] and their X-ray studies have widely been presented in the literature [1–8]. Kessel and Hendrickson [9] have studied Schiff base complexes of iron(III) incorporating *p*-quinone bridges. The

reaction of [Fe(salen)<sub>2</sub>O] with carboxylic acids has been described by Wollmann and Hendrickson [10]. They used trichloroacetic, trifluoroacetic, salicylic and picric acids, and characterized the prepared complexes as dimers of composition [Fe(salen)X]<sub>2</sub>, where X is monoanion of the appropriate acid. Complexes [Fe(salen)<sub>2</sub>L] and [Fe(salophen)<sub>2</sub>L] (where L = terephthalate, fumarate, oxalate and succinate dianion) have been prepared. The crystal and molecular structure of [Fe(salen)<sub>2</sub>ter] (H<sub>2</sub>ter = terephthalic acid) has been reported [11]. Koç and Uçan [12] have reported the synthesis and characterization of 1,3,5-tricarboxylato bridges with [SalenFe(III)] and [SalophenFe(III)]. Other complexes of composition [Fe(salen)<sub>2</sub>L] (where L = glutarate, adipate, pimelate, suberate and dithiooxamidedianion) were prepared by Smekal et al. [13].

The aim of the present study is synthesis of novel tri-directional-trinuclear systems and to present their effects on magnetic behaviour of [salen/salophenFe(III)] and [salen/salophenCr(III)] capped complexes. We also report that synthesized tridirectional melamine Schiff bases present a new scaffold. The reaction of melamine (C<sub>3</sub>N<sub>6</sub>H<sub>6</sub>) with 3 equiv of 4-carboxybenzaldehyde in benzene produces the desired tris-iminocarboxylic acid in a single step under reflux, coded to be **L**. Their structures were characterized by FT-IR, <sup>1</sup>H-NMR, LC-MS, TGA and magnetic susceptibility. The metal ratios of the prepared complexes have been determined using AAS.

## Experimental

### Materials and methods

Melamine, 4-carboxybenzaldehyde and all other reagents were purchased from Merck and used without further

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purification.  $[\{\text{Fe}(\text{salen})_2\}\text{O}]$ ,  $[\{\text{Fe}(\text{salophen})_2\}\text{O}]$ ,  $[\{\text{Cr}(\text{salen})_2\}\text{O}]$  and  $[\{\text{Cr}(\text{salophen})_2\}\text{O}]$  were prepared according to previously published methods [8, 14, 15].  $^1\text{H-NMR}$  spectra were taken using a Varian-Mercury 200 NMR spectrometer. The chemical shifts for NMR spectra are ascribed in relation to the external TMS standard. IR spectra were recorded using a Perkin-Elmer 1600 FTIR spectrometer using KBr pellets. Elemental analyses were carried out using a Hewlett-Packard 185 analyzer. Metal contents in complexes were determined using Unicam 929 AAS spectrometer. Mass spectra of the compounds were obtained on Varian MAT 711 spectrometer. pH values were measured on a WTW pH, 537 pH meter. The purification of products obtained at the end of the reaction was carried out using Combi Flash Chromatography. Magnetic susceptibilities of metal complexes were determined using a Sheerwood Scientific MX Gouy magnetic susceptibility apparatus carried out using the Gouy method with  $\text{Hg}[\text{Co}(\text{SCN})_4]$  as calibrant. The effective magnetic moments,  $\mu_{\text{eff}}$ , per metal atom was calculated from the expression:  $\mu_{\text{eff}} = 2.84\sqrt{\chi_M T} B.M.$ , where  $\chi_M$  is the molar susceptibility.

#### Preparation of ligand complexes

$[\{\text{Fe}/\text{Cr}(\text{salen})\}_2\text{O}]$  and  $[\{\text{Fe}/\text{Cr}(\text{salophen})\}_2\text{O}]$  were prepared by adding concentrated ammonia solution to a stirred hot EtOH solutions of  $[\text{Fe}/\text{Cr}(\text{salen})\text{Cl}]$  or  $[\text{Fe}/\text{Cr}(\text{salophen})\text{Cl}]$ , respectively, until they were became alkaline [8, 14, 15].

The synthesis of 2,4,6-tris(4-carboxybenzimidino)-1,3,5-triazine **L (II)**

To a suspension of melamine (6.30 g, 50 mmol) in 50 mL of dry benzene, stirred at room temperature for 1 h, an

equivalent amount of 4-carboxybenzaldehyde (22.50 g, 150 mmol) was added. The mixture was stirred for 6 h. The dirty white product was collected by filtration and dried in vacuo. The obtained mixture was purified using Combi Flash Chromatography and using 1:1 acetone/chloroform mixture as eluent. The product was recrystallized from a mixture of methanol and water (1:1).  $^1\text{H-NMR}$  ( $\text{DMSO-d}_6$ )  $\delta$  13.96 (s, broad, 3H),  $\delta$  9.99 (s, 3H), 7.99–8.15 (12H, m). LC-MS data for **II**  $m/z$ :  $523 \pm 1$ . The IR data and yield for **II** is given in Table 1–2.

#### Preparation of LFeSalen (**III**), LFeSalophen (**IV**), LCrSalen (**V**) and LCrSalophen (**VI**) complexes

A solution of **L** (0.52 g, 2.0 mmol) and  $[\{\text{Fe}/\text{Cr}(\text{salen})\}_2\text{O}]$  (3.0 mmol) or  $[\{\text{Fe}/\text{Cr}(\text{salophen})\}_2\text{O}]$  (3.0 mmol) in 100 mL of absolute ethanol were refluxed for 3 h. The mixture was allowed to cool to room temperature. Then the mixture was filtered and dried in vacuum. LC-MS datas for **III**  $m/z$ :  $1486 \pm 2$ , for **IV**  $m/z$ :  $1630 \pm 2$ , for **V**  $m/z$ :  $1475 \pm 2$ , and for **VI**  $m/z$ :  $1619 \pm 2$ . The IR data and yield for **III**, **IV**, **V** and **VI** are given in Table 1–2.

## Results and discussion

The target ligands were synthesized in one-step from melamine. The conversion of melamine to the trisubstituted-melamine derivative was accomplished in 92% yield. The structural formula of the 2,4,6-tris(4-carboxybenzimidino)-1,3,5-triazine **L (II)** was verified by elemental analyses,  $^1\text{H-NMR}$ , FT-IR and mass spectral data [16] (Fig. 1, Table 1–2). The ligand is soluble in common organic solvents. Synthetic strategy for preparing trinuclear-imino-carboxylate uses a complex as a “ligand” that contains a potential donor group capable of coordinating to another

**Table 1** Some physical properties, molecular weight (g/mol) data and elemental analyses, AAS analyses of the ligands and complexes

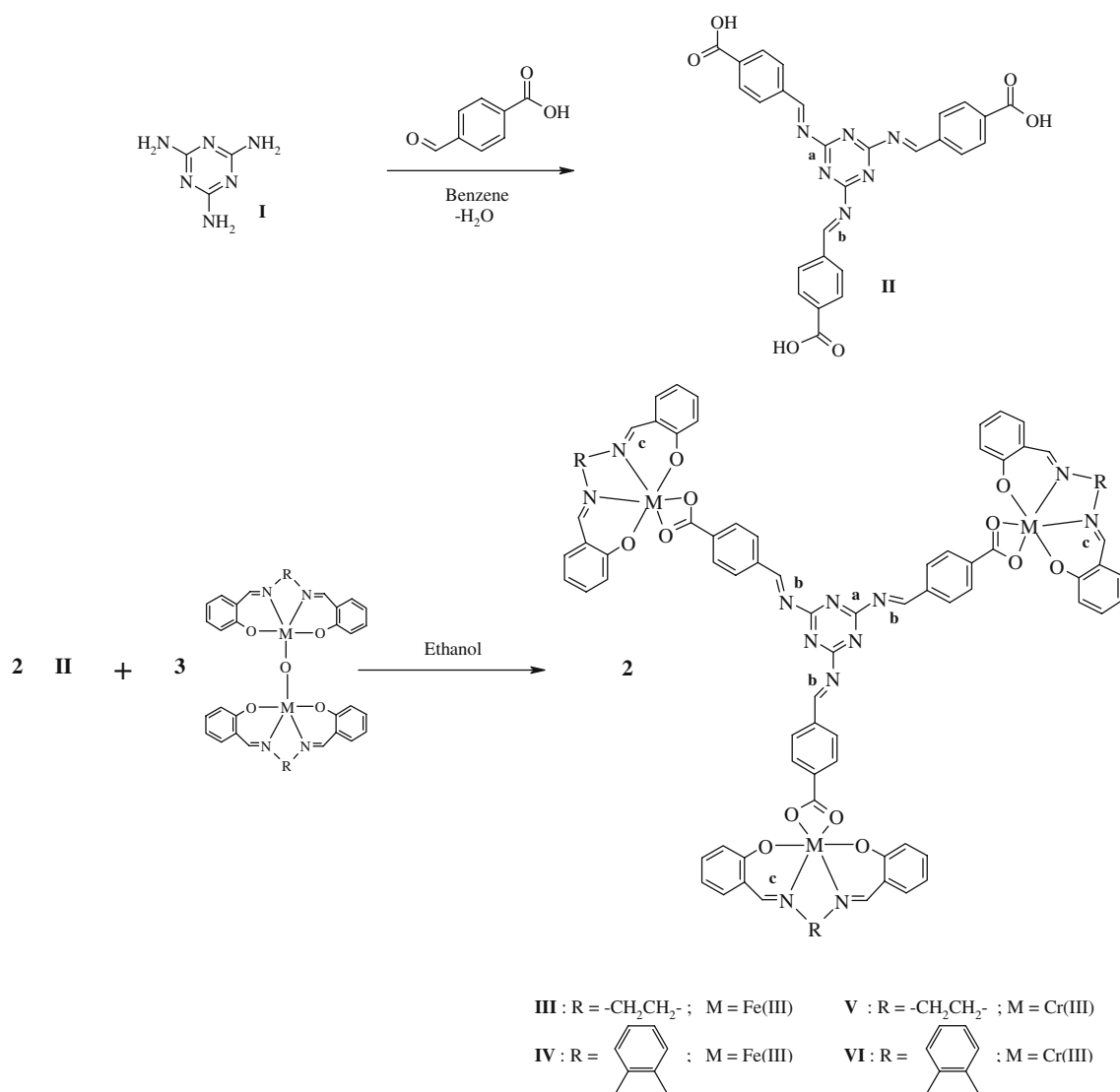
| Compounds  | $\mu_B$ | M.p. (°C)        | Yield (g) % | Color $M_w$ (g/mol)   | Found calculated % of |                       |                     |                       |                       |
|--|---------|------------------|-------------|-----------------------|-----------------------|-----------------------|---------------------|-----------------------|-----------------------|
|  |         |                  |             |                       | C                     | N                     | H                   | Fe                    | Cr                    |
| $\text{C}_{27}\text{H}_{18}\text{N}_6\text{O}_6$<br><b>L (II)</b>  | Dia.    | 345 <sup>a</sup> | (2.40) 92   | White (522.48)        | <b>61.97</b><br>62.01 | <b>16.05</b><br>16.08 | <b>3.44</b><br>3.45 | –                     | –                     |
| $\text{C}_{75}\text{H}_{57}\text{N}_{12}\text{O}_{12}\text{Fe}_3$<br><b>L(FeSalen)<sub>3</sub> (III)</b>   | 1.27    | 268 <sup>a</sup> | (2.38) 80   | Red-brown (1,485.95)  | <b>60.51</b><br>60.57 | <b>11.27</b><br>11.31 | <b>3.82</b><br>3.84 | <b>11.25</b><br>11.27 | –                     |
| $\text{C}_{87}\text{H}_{57}\text{N}_{12}\text{O}_{12}\text{Fe}_3$<br><b>L(FeSalophen)<sub>3</sub> (IV)</b> | 1.37    | 265 <sup>a</sup> | (2.54) 78   | Red (1,630.09)        | <b>64.01</b><br>64.05 | <b>10.27</b><br>10.31 | <b>3.48</b><br>3.50 | <b>10.27</b><br>10.28 | –                     |
| $\text{C}_{75}\text{H}_{57}\text{N}_{12}\text{O}_{12}\text{Cr}_3$<br><b>L(CrSalen)<sub>3</sub> (V)</b>     | 2.81    | 271 <sup>a</sup> | (1.77) 60   | Green (1,474.40)      | <b>61.00</b><br>61.04 | <b>11.34</b><br>11.39 | <b>3.85</b><br>3.87 | –                     | <b>10.55</b><br>10.58 |
| $\text{C}_{87}\text{H}_{57}\text{N}_{12}\text{O}_{12}\text{Cr}_3$<br><b>L(CrSalophen)<sub>3</sub> (VI)</b> | 2.98    | 270 <sup>a</sup> | (1.88) 58   | Dark green (1,618.53) | <b>64.45</b><br>64.50 | <b>10.35</b><br>10.38 | <b>3.74</b><br>3.75 | –                     | <b>9.61</b><br>9.64   |

<sup>a</sup> Decomposition

**Table 2** Characteristic FT-IR bands ( $\text{cm}^{-1}$ ) of complexes\*\*

| Compounds   | C=N                   | C–N   | C–C <sub>ar</sub> | C–H <sub>ar</sub> | C–H <sub>aliph</sub> | C=O   | COO   |
|---|-----------------------|-------|-------------------|-------------------|----------------------|-------|-------|
| C <sub>27</sub> H <sub>18</sub> N <sub>6</sub> O <sub>6</sub>                   | 1,594 <sup>a</sup>    | 1,128 | 1,448             | 3,155 as          | 2,863                | 1,690 | 1,405 |
| L (II)  | 1,659 <sup>b</sup>    |       | 1,552             | 1,500 w           |                      |       |       |
| C <sub>75</sub> H <sub>57</sub> N <sub>12</sub> O <sub>12</sub> Fe <sub>3</sub> | 1,595 <sup>a</sup>    | 1,126 | 1,445             | 3,147 as          | 2,898                | 1,688 | 1,385 |
| L(FeSalen) <sub>3</sub> (III)   | 1,625 <sup>b</sup>    |       | 1,466             | 1,147 w           |                      |       |       |
|   | 1,540 <sup>c</sup>    |       |                   |                   |                      |       |       |
| C <sub>87</sub> H <sub>57</sub> N <sub>12</sub> O <sub>12</sub> Fe <sub>3</sub> | 1,580 <sup>a</sup>    | 1,124 | 1,445             | 3,145 as          | 3,053                | 1,686 | 1,379 |
| L(FeSalophen) <sub>3</sub> (IV)   | 1,607 <sup>b</sup>    |       | 1,462             | 1,150 w           |                      |       |       |
|   | 1,535 <sup>c</sup>    |       |                   |                   |                      |       |       |
| C <sub>75</sub> H <sub>57</sub> N <sub>12</sub> O <sub>12</sub> Cr <sub>3</sub> | 1,621 <sup>a, b</sup> | 1,129 | 1,468             | 3,148 as          | 2,914                | 1,699 | 1,385 |
| L(CrSalen) <sub>3</sub> (V)   | 1,542 <sup>c</sup>    |       | 1,417             | 1,149 w           |                      |       |       |
| C <sub>87</sub> H <sub>57</sub> N <sub>12</sub> O <sub>12</sub> Cr <sub>3</sub> | 1,613 <sup>a, b</sup> | 1,124 | 1,443             | 3,143 as          | 2,916                | 1,695 | 1,385 |
| L(CrSalophen) <sub>3</sub> (VI)   | 1,540 <sup>c</sup>    |       | 1,474             | 1,152 w           |                      |       |       |

\*\*KBr pellet—w wagging vibration, as asymmetric stretching vibration

**Fig. 1** Synthetic routes for the preparation ligand II and its complexes

ligand. Therefore, we choose  $[\{\text{Fe}/\text{Cr}(\text{salen})\}_2\text{O}]$  and  $[\{\text{Fe}/\text{Cr}(\text{salophen})\}_2\text{O}]$  as “ligand complex” [17]. These complexes are some of the first examples of melamine-based trinuclear complexes bridged to the iron/chromium centers by carboxylate anions. All compounds are stable at room temperature in the solid state and they are only soluble in organic solvents such as ethylacetate, DMSO, DMF and insoluble in water. The results of the elemental analyses, given in Table 1, are in a good agreement with the structures suggested for the ligands and their complexes. The results show that all complexes are trinuclear.

FT-IR spectra of **II** show two strong bands for C=N (a) and C=N (b) at 1,594 and 1,659  $\text{cm}^{-1}$  in accordance with reported recently by Koc and Ucan (2007). A strong broad band at 1,690  $\text{cm}^{-1}$  indicates presence of carboxylic acid carbonyl C=O stretching vibration [18, 19]. The band at 1,448  $\text{cm}^{-1}$  is for phenyl group.

IR bands at 1625  $\text{cm}^{-1}$  for complex **III**, 1607  $\text{cm}^{-1}$  for complex **IV**, 1621  $\text{cm}^{-1}$  for complex **V** and 1613  $\text{cm}^{-1}$  for complex **VI** were assigned to C=N (b) stretching vibrations and band at 1540, 1535, 1542 and 1540  $\text{cm}^{-1}$  were assigned to C=N (c) stretching vibrations for complexes **III**, **IV**, **V** and **VI**, respectively. Bands at 1595, 1580, 1621  $\text{cm}^{-1}$  (broad) and 1613  $\text{cm}^{-1}$  were assigned to triazine ring (a) stretching vibrations for complexes **III**, **IV**, **V** and **IV**, respectively, whereas C=N (c) stretching vibration bands were found at 1,560–1,567  $\text{cm}^{-1}$  for  $[(\text{salen}/\text{saxophone})\text{Fe}(\text{III})/\text{Cr}(\text{III})]_2\text{O}$  complexes [12, 18–22]. Bands at 1688, 1693, 1699 and 1695  $\text{cm}^{-1}$  for complexes **III**, **IV**, **V** and **VI** were assigned to C=O groups since coordination of (salen/salophen)Fe(III)/Cr(III) to COO groups. Furthermore, bands at 1385, 1379, 1385 and 1385  $\text{cm}^{-1}$  were assigned to COO<sup>-</sup> ions of that complexes [12, 14, 21]. In the tri-directional-trinuclear complexes, the bands in the 558–532 and 466–483  $\text{cm}^{-1}$  ranges can be attributed to the M-N and M-O stretching modes [23].

In order to identify the structure of **II**, the <sup>1</sup>H-NMR spectra were recorded in DMSO-d<sub>6</sub> [12]. <sup>1</sup>H-NMR spectra also confirmed the structure of the synthesized compound. The signals in <sup>1</sup>H-NMR spectrum of 2,4,6-tris(4-carboxybenzimidino)-1,3,5-triazine (L) at  $\delta$  9.99 ppm correspond to three –N=CH– groups [24].

The magnetic moments of the complexes given in Table 1 were measured at room temperature. On the basis of spectral evidence, the 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 behaviour of Fe(III) and Cr(III) complexes is in accord with proposed trinuclear structures [25]. The magnetic moment per trinuclear complexes which were constructed from  $[\{\text{Fe}(\text{salen})\}_2\text{O}]$ ,  $[\{\text{Fe}(\text{salophen})\}_2\text{O}]$ ,  $[\{\text{Cr}(\text{salen})\}_2\text{O}]$  and  $[\{\text{Cr}(\text{salophen})\}_2\text{O}]$  either of 2,4,6-tris(4-carboxybenzimidino)-1,3,5-triazine (L) shows paramagnetic property with a magnetic susceptibility value per atom: 1.27–1.37 B.M. and

2.81–2.98 B.M., respectively. It is seen that the  $[\{\text{Fe}(\text{salen})\}_2\text{O}]$ ,  $[\{\text{Fe}(\text{salophen})\}_2\text{O}]$ ,  $[\{\text{Cr}(\text{salen})\}_2\text{O}]$  and  $[\{\text{Cr}(\text{salophen})\}_2\text{O}]$  containing compounds are represented by the electronic structure of  $t_{2g}^5e_g^0$  and  $t_{2g}^3e_g^0$ . The magnetic data for the  $[\{\text{Fe}(\text{salen})\}_2\text{O}]$ ,  $[\{\text{Fe}(\text{salophen})\}_2\text{O}]$ ,  $[\{\text{Cr}(\text{salen})\}_2\text{O}]$  and  $[\{\text{Cr}(\text{salophen})\}_2\text{O}]$  tripodal complexes show well agreement with the d<sup>5</sup> and d<sup>3</sup> metal ion in an octahedral structure. This consequence is supported by the results of the elemental analyses suggesting that these Trinuclear complexes have also an octahedral structure [8, 12, 15, 20, 21, 25].

Samples (IV and V) chosen in all the complexes were thermally investigated. It is well known that there is a strong relation between temperature range for the dehydration process and the binding mode of the water molecules to the respective metal complexes [26]. The elimination of water has taken place in a single step process attributed to the release of the hydrated water molecules (in the range of 60–120 °C) [12, 20, 21, 27]. Thermal decomposition of the anhydrous  $[\text{Fe}/\text{Cr}(\text{salen})]$  and  $[\text{Fe}/\text{Cr}(\text{salophen})]$  complexes left from the ligands L have started in the range of 265–275 °C and completed in the range of 395–670 °C. The final decomposition products were metal oxides and triazine ring. The observed weight losses for complexes are in good agreement with the calculated values.

In TGA-DTA diagram (Fig. 2) of  $[\text{Fe}(\text{salophen})]$  capped complexes of 2,4,6-tris(4-carboxybenzimidino)-1,3,5-triazine (IV), first decomposition step was started at 265 °C. Although the weight loss was theoretically calculated to be 7.89%, it was observed experimentally to be 5.55%. While CO<sub>2</sub> gases left from the medium,  $[\text{Fe}(\text{salophen})]$  group was also left from the main structure. At the second

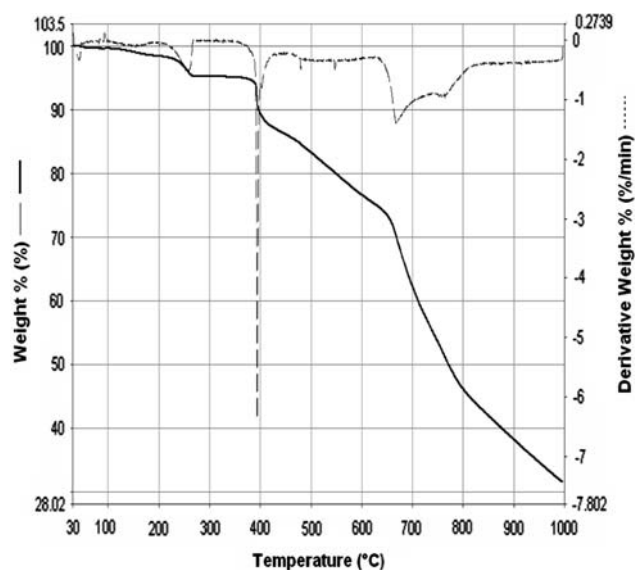
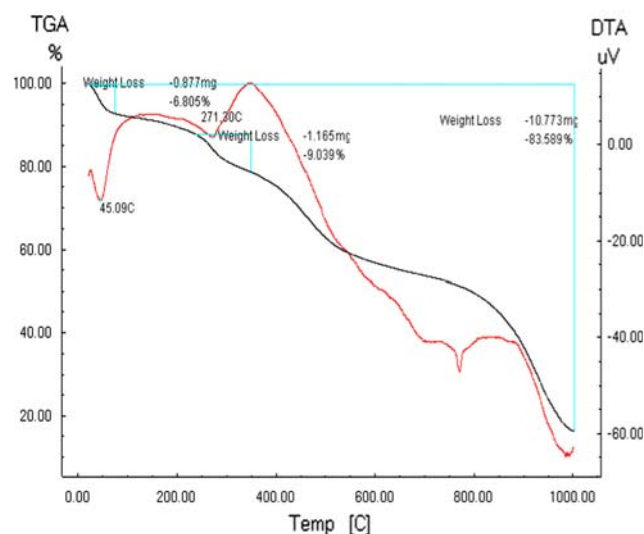


Fig. 2 The TGA-DTA diagram of  $L(\text{FeSalophen})_3$



**Fig. 3** The TGA-DTA diagram of  $L(\text{CrSalen})_3$

decomposition step,  $\text{C}_6\text{H}_6$  and  $\text{N}_2$  gasses went away from main structure at  $395^\circ\text{C}$ . Although the weight loss was theoretically calculated to be 23.85%, it was observed experimentally to be 19.45%. At the third decomposition step,  $\text{C}_6\text{H}_6$  left from the main structure at  $670^\circ\text{C}$ . Although the total weight loss was theoretically calculated to be 77.61%, it was observed experimentally to be 71.98%.

According to TGA-DTA diagram (Fig. 3) of  $[\text{Cr}(\text{salen})]$  capped complexes of 2,4,6-tris(4-carboxybenzimidino)-1,3,5-triazine (**V**), decomposition of  $[\text{Cr}(\text{salen})]$  capped complexes of **L** was determined in two steps. At the thermal decomposition steps,  $\text{CO}_2$  was firstly left from the main structure at  $271.30^\circ\text{C}$  and weight loss has theoretically been calculated to be 8.78% and measured experimentally to be 9.04%. When the sample was heated to  $450^\circ\text{C}$ , it was calculated theoretically and measured experimentally that the total weight loss was 89.46 and 83.59% respectively. And,  $\text{C}_6\text{H}_6$ ,  $\text{C}_2\text{H}_4$  ve  $\text{N}_2$  gases left from the main structure.

From the investigation of LC-MS spectra of all compounds, it has been seen that molecular weights of ligands and complexes are in good agreement with the intensity observed values in LC-MS spectra (Table 1).

## Conclusions

In this study, novel tri-directional and melamine based Schiff bases “2,4,6-tris(4-carboxybenzimidino)-1,3,5-triazine” were synthesized. Synthetic strategy for preparing Tripodal-Trinuclear uses a complex as a “ligand” that contains a potential donor group capable of coordinating to the other ligand. We have chosen  $[\{\text{Fe}(\text{salen}/\text{saloph})\}_2\text{O}]$  and  $[\{\text{Cr}(\text{salen}/\text{Saloph})\}_2\text{O}]$  as “ligand complexes” because they can coordinate to the other ligand. These complexes are the examples of

Tripodal-Trinuclear complexes bridged by carboxylate anions to the iron and chromium centers. Their structures were characterized by means of elemental analysis,  $^1\text{H NMR}$ , FT-IR spectroscopy, LC-MS, thermal analyses and magnetic susceptibility measurements. The magnetic data for the tripodal-trinuclear complexes show well agreement with the  $d^5$  and  $d^3$  metal ion in an octahedral structure.

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