# ORIGINAL ARTICLE

# The synthesis and characterization of 2,4,6-tris(3,4dihydroxybenzimino)-1,3,5-triazine and its [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-(salophenH<sub>2</sub>) with 2,4,6-tris(3,4-dihydroxybenzimino)-1,3,5-triazine have been synthesized and characterized by means of elemental analysis, <sup>1</sup>H N.M.R., FT-IR spectroscopy, thermal analyses and magnetic susceptibility measurements. The complexes have also been characterized as low-spin distorted octahedral Fe(III) and Cr(III) bridged by catechol group.

# Introduction

Melamine is the common name of 2,4,6-triamino-s-triazine. 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. 1,3,5-Triazine derivatives are widely used as herbicides [1], drugs [2] or polymers [3], like melamineformaldehyde that has excellent thermal and electrical properties [4]. Phenolic melamine is a good non-flamability owing to containing of nitrogen in the chemical structure [5].

Schiff bases complexes of iron(III) have been known since 1938. The magnetochemical properties of the  $\mu$ -oxobridged complexes [{Fe(salen)}<sub>2</sub>O] [(salenH<sub>2</sub> = N,N'-bis

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Department of Chemistry, Faculty of Science, Selcuk University, 42075 Konya, Turkey e-mail: uysal77@hotmail.com (salicylidene) ethylenediamine)] and  $[{Fe(salophen)}_2O]$  $[(salophenH_2 = bis(salicylidene)-2-phenylenediamine)]$ and their X-ray studies have widely been presented in the literature [6–13]. Kessel and Hendrickson [14] have studied Schiff base complexes of iron(III) incorporating *p*-quinone bridges. The reaction of  $[{Fe(salen)}_2O]$  with carboxylic acids has been described by Wollmann and Hendrickson [15]. They used trichloroacetic, trifloroacetic, 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 [16]. Koc and Ucan [17] 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. [18].

Chromium is a unique transition metal ion, which has been established to be biologically significant at all the levels of living organisms [19]. Out of the two stable oxidation states of chromium, -VI and -III, trivalent chromium has been shown to play positive role in controlling carbohydrate and lipid metabolism [20]. A Schiff base complex of chromium(III), [Cr(salen)(OH<sub>2</sub>)<sub>2</sub>]<sup>+</sup>, was found to enhance insulin activity and insulin derivatised with the same was found to exhibit higher activity in glucose metabolism in animal models when compared to either free insulin or other derivatives [21, 22].

Their applications have grown rapidly and a broad range of asymmetric catalytic reactions has been described including oxidations, additions and reductions such as epoxidation of olefins, epoxide ring opening, Diels-Alder reaction, imine cyanation, conjugate addition, carbon dioxide insertion into epoxides, etc. [23–27]. Various metal-Salen complexes in the homogeneous phase such as manganese(III) [28], chromium(III) [29] and nickel(II) [30] Salen have been used for the epoxidation of olefins [31].

The aim of the present study is synthesis of novel tridirectional-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 ( $C_3N_6H_6$ ) with 3 equiv of 3,4-dihydroxybenzaldehyde in benzene produces the desired tris-iminocatechol in a single step under reflux, coded to be **L**. Their structures were characterized by FT-IR, <sup>1</sup>H N.M.R., TGA and magnetic susceptibility. The metal ratios of the prepared complexes have been determined using AAS.

## Experimental

#### Materials and methods

Melamine, 3,4-dihydroxybenzaldehyde and all other reagents were purchased from Merck and used without further purification. [{Fe(salen)<sub>2</sub>}O], [{Fe(salophen)<sub>2</sub>}O],  $[{Cr(salen)_2}O]$  and  $[{Cr(salophen)_2}O]$  were prepared according to previously published methods [13, 32, 33]. <sup>1</sup>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. 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 Cromotography. Magnetic susceptibilities of metal complexes were determined using a Sheerwood Scientific MX Gouy magnetic susceptibility apparatus carried out using the Gouy method with  $Hg[Co(SCN)_4]$  as calibrant. The effective magnetic moments,  $\mu_{eff}$ , per metal atom was calculated from the expression:  $\mu_{\rm eff} = 2.84 \sqrt{\chi_{\rm M}} TB.M.$ where  $\chi_{\rm M}$  is the molar susceptibility.

Preparation of ligand complexes

 $[Fe(salen)]_2O$ ,  $[Cr(salen)]_2O$ ,  $[Fe(salophen)]_2O$  and  $[Cr(salophen)]_2O$  were prepared by adding concentrated ammonia solution (26% w/w) to a stirred hot EtOH

solutions of [Fe/Cr(salen)Cl] or [Fe/Cr(salophen)Cl], respectively, until they were became alkaline [13, 32, 33].

The synthesis of 2,4,6-tris(3,4-dihydroxybenzimino)-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, three equivalent amount of 3,4-dihydroxybenzaldehyde (20.70 g, 150 mmol) was added. The mixture was stirred for 6 h. The white product was collected by filtration and dried in vacuo. The obtained mixture was purified using Combi Flash Cromotography and using 1:1 acetone/chloroform mixture as eluent. The product was recrystallized from a mixture of methanol and water (1:1). Yield for the compound is 2.28 g (94%). <sup>1</sup>H-N.M.R. (DMSO-d<sub>6</sub>)  $\delta$  9.70 (s, 3H),  $\delta$  = 6.09 (s, 6H) and 6.90–7.28 (9H, m). The IR data and yield for **II** is given in Tables 1–2.

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

A solution of L (0.49 g, 2.0 mmol) and  $[Fe(salen)]_2O/[Cr(salen)]_2O/[Fe(salophen)]_2O/[Cr(salophen)]_2O (3.0 mmol) in 100 mL of absolute ethanol were refluxed for 3 h. The mixture was allowed to cool to room temperature. Then, the solid product was filtered and purified using Combi Flash Cromotography and using 1:2 ethanol/ diethylether mixture as eluent.$ 

Yield for the complexes are 2.47 g (85%), 2.55 g (80%), 1.73 g (60%) and 1.97 g (62%), respectively.

The magnetic moment values (B.M.) for **III**, **IV**, **V** and **VI** are 1.86, 1.79, 3.57 and 3.53, respectively.

The IR (cm<sup>-1</sup>) data for **III**:  $1598^{a}$ ,  $1670-1624^{b}$ ,  $1540^{c}$  (C=N), 1125 (C–N), 1443, 1558 (C–C<sub>ar</sub>), 3025as, 1508w (C–H<sub>ar</sub>), 2897 (C–H<sub>aliph</sub>), 3418s, 1385w (O–H), 1288, 1253 (C–O<sub>ph</sub>), 545 (M–N), 473 (M–O).

The IR (cm<sup>-1</sup>) data for **IV**: 1579<sup>a</sup>, 1654–1607<sup>b</sup>, 1534<sup>c</sup> (C=N), 1125 (C–N), 1445 (C–C<sub>ar</sub>), 3056 (C–H<sub>a</sub>), 2874 (C–H<sub>aliph</sub>), 3417s, 1378 (O–H), 1281, 1257 (C–O<sub>ph</sub>), 558 (M–N), 483 (M–O).

The IR (cm<sup>-1</sup>) data for V: 1620<sup>a</sup>, 1638<sup>b</sup>, 1572<sup>c</sup> (C=N), 1125, 1119 (C–N), 1437, 1520 (C–C<sub>ar</sub>), 3127as (C–H<sub>ar</sub>), 2870, 2898 (C–H<sub>aliph</sub>), 3396s, 1378w (O–H), 1288 (C–O<sub>ph</sub>), 532 (M–N), 466 (M–O).

The IR (cm<sup>-1</sup>) data for VI:  $1656-1615^{a}$ ,  $1722-1687^{b}$ ,  $1575^{c}$  (C=N), 1115 (C–N), 1435, 1484 (C–C<sub>ar</sub>), 3127as (C–H<sub>ar</sub>), 2875 (C–H<sub>aliph</sub>), 3347s, 1396w (O–H), 1285 (C–O<sub>ph</sub>), 542 (M–N), 475 (M–O), respectively. The elemental analysis, physical properties and magnetic moment values of the complexes are given in Table 1.

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

Compounds	$\mu_{\rm B}$	M.p. (°C)	Yield (g) %	Color $M_{\mathbf{W}}$ (g/mol)	Found Calculated % of				
					С	Ν	Н	Fe	Cr
C <sub>24</sub> H <sub>18</sub> N <sub>6</sub> O <sub>6</sub> L ( <b>II</b> )	Dia.	145	(2,28) 94	White	59,18	17,24	3,69	_	_
				[486,45]	59,20	17,27	3,70		
$C_{72}H_{57}N_{12}O_{12}Fe_3 L(FeSalen)_3$ (III)	1.86	336 <sup>a</sup>	(2,47) 85	Red-brown	59,55	11,57	3,90	11,55	-
				[1449,92]	59,59	11,59	3,93	11,56	
$C_{84}H_{57}N_{12}O_{12}Fe_3 L(FeSalophen)_3$ (IV)	1.79	318 <sup>a</sup>	(2,55) 80	Brown	63,22	10,52	3,56	10,48	-
				[1594,05]	63,24	10,54	3,58	10,51	
$C_{72}H_{57}N_{12}O_{12}Cr_3 L(CrSalen)_3$ (V)	3.57	280 <sup>a</sup>	(1,73) 60	Dark green	60,02	11,64	3,93	-	10,81
				[1438,37]	60,07	11,68	3,96		10,84
$C_{84}H_{57}N_{12}O_{12}Cr_3 L(CrSalophen)_3$ (VI)	3.53	259 <sup>a</sup>	(1,97) 62	Dark green	63,66	10,58	3,59	-	9,85
				[1582,50]	63,70	10,62	3,60		9,86

<sup>a</sup> Decomposition

Bold values are found values and unbold values are calculated values

Table 2 Characteristic FT-IR bands (cm<sup>-1</sup>) of complexes\*

Compounds	C=N	C–N	C–C <sub>ar</sub>	C–H <sub>ar</sub>	C-H <sub>aliph</sub>	O–H	C–O <sub>ph</sub>
$C_{24}H_{18}N_6O_6 L$ (II)	1,595 <sup>a</sup>	1,119	1,441	3,130as	2,872	3,419s	1,298
	1,654 <sup>b</sup>			1,537w		1,386w	
$C_{72}H_{57}N_{12}O_{12}Fe_3 L(FeSalen)_3$ (III)	1,598 <sup>a</sup>	1,125	1,443	3,025as	2,897	3,418s	1,288
	1,670–1,624 <sup>b</sup>		1,558	1,508w		1,385w	1,253
	1,540 <sup>c</sup>						
$C_{84}H_{57}N_{12}O_{12}Fe_3 L(FeSalophen)_3$ (IV)	1,579 <sup>a</sup>	1,125	1,445	3,056as	2,874	3,417s	1,281
	1,654–1,607 <sup>b</sup>					1,378w	
	1,534 <sup>c</sup>						1,257
$C_{72}H_{57}N_{12}O_{12}Cr_3 L(CrSalen)_3$ (V)	1,620 <sup>a</sup>	1,125	1,437	3,127as	2,870	3,396s	1,288
	1,638 <sup>b</sup>	1,119	1,520		2,898	1,378w	
	1,572 <sup>c</sup>						
$C_{84}H_{57}N_{12}O_{12}Cr_3 L(CrSalophen)_3$ (VI)	1,656–1,615 <sup>a</sup>	1,115	1,435	3,127as	2,875	3,347s	1,285
	1,722–1,687 <sup>b</sup>		1,484			1,396w	
	1,575 <sup>c</sup>						

\*KBr pellet—*w* wagging vibration, *as* asymmetric stretching vibration Bold values are found values and unbold values are calculated values

## **Results and discussion**

The target ligands were synthesized in one-step from melamine. The conversion of melamine to the trisubstitutedmelamine derivative was accomplished in 94% yield. The structural formula of the 2,4,6-tris(3,4-dihydroxybenzimino)-1,3,5-triazine **L** (**II**) was verified by elemental analyses, <sup>1</sup>H-N.M.R. and FT-IR [34] (Fig. 1, Tables 1, 2). The ligand is soluble in common organic solvents. Synthetic strategy for preparing trinuclear-iminocatechol uses a complex as a "ligand" that contains a potential donor group capable of coordinating to metal center of ligand complexes. Therefore, we choose [Fe(salen)]<sub>2</sub>O, [Cr(salen)]<sub>2</sub>O, [Fe(salophen)]<sub>2</sub>O and [Cr(salophen)]<sub>2</sub>O as "ligand complexes" [35]. These complexes are some of the first examples of melamine-based trinuclear complexes bridged to the iron/chromium centers by catechol groups. The other examples of complexes bridged to the iron centers by catechol group have also been published in 2008 by Uysal et al. [36]. 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 1,595 and 1,654 cm<sup>-1</sup> in accordance with reported recently by Koç and Uçan (2007). The band at 1,441 cm<sup>-1</sup> indicates presence of phenyl groups, and phenolic C–O streching vibration was observed at



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

1,298 cm<sup>-1</sup> [37, 38]. Phenolic OH streching vibration at 3,419 cm<sup>-1</sup> and the other phenolic OH wagging vibration at 1,386 cm<sup>-1</sup> were observed at the FT-IR spectra of **II** [36, 39].

IR bands at  $1,670-1,624 \text{ cm}^{-1}$  for complex III,  $1,654-1,607 \text{ cm}^{-1}$  for complex IV,  $1,638 \text{ cm}^{-1}$  for complex V and  $1,687 \text{ cm}^{-1}$  for complex VI were assigned to C=N (b) streching vibrations and band at 1540, 1534, 1572 and 1575 cm<sup>-1</sup> were assigned to C=N (c) streching vibrations for complexes III, IV, V and VI, respectively. Bands at 1598, 1579, 1620 and 1656–1615 cm<sup>-1</sup> were assigned to triazine ring (a) streching vibrations for complexes III, IV, V and VI, respectively, whereas C=N (c) streching vibration bands were found at  $1,560-1,567 \text{ cm}^{-1}$  for [Fe(salen)]<sub>2</sub>O, [Cr(salen)]<sub>2</sub>O, and [Cr(salophen)]<sub>2</sub>O complexes [17, 36–38]. Aromatic streching vibrations are between  $1,558 \text{ and } 1,435 \text{ cm}^{-1}$  for III, IV, V and VI. Phenolic C–O streching vibrations at 1288, 1281, 1288 and 1285 cm<sup>-1</sup> have clearly been observed at the IR spectra of III, IV, V and

**VI**, respectively, whereas they had been observed at  $1,277 \text{ cm}^{-1}$  for **L**<sup>1</sup>. It has been interpreted that a downward shifts of 5–11 cm<sup>-1</sup> for phenolic C–O streching vibration in the complexes indicates coordination through the oxygen atoms. A downward shifts from 1,560 and 1,567 cm<sup>-1</sup> for C=N (c) to 1,534–1,575 cm<sup>-1</sup> indicated also that capped coordination was formed [17, 32, 36–38]. In the tri-directional-trinuclear complexes, the bands in the 558–532 and 466–483 cm<sup>-1</sup> ranges can be attributed to the M–N and M–O streching modes [40].

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(3,4-dihydroxybenzimino)-1,3,5-triazine (**L**) at singlet  $\delta = 9.70$  ppm correspond to three -N=CH- groups and at singlet  $\delta = 6.09$  ppm for -OH groups [41]. At  $\delta = 6.90$ -7.28 ppm (multiplet) for ligand **II** are relating to aromatic C-**H** signals.

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 [42]. The magnetic moment per trinuclear complexes which were constructed from [{Fe(salen)}<sub>2</sub>O], [{Fe(salophen)}<sub>2</sub>O],  $[{Cr(salen)}_2O]$  and  $[{Cr(salophen)}_2O]$  of 2,4,6-tris(3,4dihydroxybenzimino)-1,3,5-triazine (L) shows paramagnetic property with a magnetic susceptibility value per atom: 1.86-1.79 B.M. and 3.57-3.53 B.M., respectively. It is seen that the [{Fe(salen)}<sub>2</sub>O], [{Fe(salophen)}<sub>2</sub>O],  $[{Cr(salen)}_2O]$  and  $[{Cr(salophen)}_2O]$  containing compounds are represented by the electronic structure of  $t_{2g}^5 e_g^0$ and  $t_{2\sigma}^3 e_{\sigma}^0$ . The magnetic data for the [{Fe(salen)}<sub>2</sub>O],  $[{Fe(salophen)}_2O], [{Cr(salen)}_2O] and [{Cr(salophen)}_2O]$ tripodal complexes show well agreement with the  $d^{5}$  and  $d^{3}$ metal ion in an octahedral structure. This consequence is supported by the results of the magnetic susceptibility and elemental analysis suggesting that these Trinuclear complexes have also an octahedral structure [13, 17, 33, 36, 42, 43].

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 [44]. 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) [17, 36, 43, 45]. Thermal decomposition of the anhydrous [Fe(salen)], [Cr(salen)], [Fe(salophen)] and [Cr(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 [Fe(salen)] capped complexes of 2,4,6-tris(3,4-dihydroxybenzimino)-1,3,5triazine (**III**), first decomposition step was started at 336.41 ° C. Although the weight loss was theoretically calculated to be 6.38%, it was observed experimentally to be 8.31%. While H<sub>2</sub>O gases left from the medium, [Fe(salen)] group was also left from the main structure. At the second decomposition step,  $C_2H_4$  and  $N_2$  gasses went away from main structure at 421.54 °C. Although the weight loss was theoretically calculated to be 17.62%, it was observed experimentally to be 23.92%. At the thirth decomposition step,  $C_6H_6$  left from the main structure at 566.10 °C. Although the total weight loss was theoretically calculated to be 59.5%, it was observed experimentally to be 64.81%. According to TGA-DTA analysis of [Fe(saloph)] capped complexes of 2,4,6-tris(3,4-dihydroxybenzimino)-1,3,5triazine (**IV**), first decomposition step was started at 318.36 °C. Although the weight loss was theoretically calculated to be 5.52%, it was observed experimentally to be 7.46%. While H<sub>2</sub>O gases left from the medium, [Fe(saloph)] group was also left from the main structure. At the second decomposition step, C<sub>6</sub>H<sub>6</sub> and N<sub>2</sub> gasses went away from main structure at 443.22 °C. Although the weight loss was theoretically calculated to be 54.96%, it was observed experimentally to be 57.70%. At the thirth decomposition step, C<sub>6</sub>H<sub>6</sub> left from the main structure at 560.30 °C. Although the total weight loss was theoretically calculated to be 79.87%, it was observed experimentally to be 85.40%.

According to TGA-DTA analysis of [Cr(salen)] capped 2,4,6-tris(3,4-dihydroxybenzimino)-1,3, complexes of 5-triazine (V), first decomposition step was started at 280.76 °C. Although the weight loss was theoretically calculated to be 6.25%, it was observed experimentally to be 8.21%. While H<sub>2</sub>O gases left from the medium, [Cr(salen)] group was also left from the main structure. At the second decomposition step, C<sub>2</sub>H<sub>4</sub> and N<sub>2</sub> gasses went away from main structure at 394.47 °C. Although the weight loss was theoretically calculated to be 50.83%, it was observed experimentally to be 54.35%. At the thirth decomposition step, C<sub>6</sub>H<sub>6</sub> left from the main structure at 563.80 °C. Although the total weight loss was theoretically calculated to be 78.54%, it was observed experimentally to be 80.07%.

According to TGA-DTA analysis of [Cr(saloph)] capped 2,4,6-tris(3,4-dihydroxybenzimino)-1,3, complexes of 5-triazine (VI), first decomposition step was started at 259.21 °C. Although the weight loss was theoretically calculated to be 5.52%, it was observed experimentally to be 7.35%. While H<sub>2</sub>O gases left from the medium, [Cr(saloph)] group was also left from the main structure. At the second decomposition step, C<sub>6</sub>H<sub>6</sub> and N<sub>2</sub> gasses went away from main structure at 412.63 °C. Although the weight loss was theoretically calculated to be 54.96%, it was observed experimentally to be 59.68%. At the thirth decomposition step, C<sub>6</sub>H<sub>6</sub> left from the main structure at 572.50 °C. Although the total weight loss was theoretically calculated to be 79.87%, it was observed experimentally to be 83.15%.

# Conclusions

In this study, novel tri-directional and melamine based Schiff bases "2,4,6-tris(3,4-dihydroxybenzimino)-1,3,5triazine" were synthesized. Synthetic strategy for preparing Tripodal-Trinuclear uses a complex as a "ligand" that





contains a potential donor group capable of coordinating to metal center of ligand complexes. We have chosen [Fe(salen)]<sub>2</sub>O, [Fe(saloph)]<sub>2</sub>O, [Cr(salen)]<sub>2</sub>O and [Cr(saloph)]<sub>2</sub>O as "ligand complexes" because they can coordinate to the other ligand. These complexes are the examples of Tripodal-Trinuclear complexes bridged by catechol group to the iron and chromium centers. Their structures were characterized by means of elemental analysis, <sup>1</sup>H N.M.R., FT-IR spectroscopy, LC-MS spectroscopy, thermal analyses and magnetic susceptibility measurements. The magnetic data for the tripodal-trinuclear complexes show well agreement with the d<sup>5</sup> and d<sup>3</sup> metal ion in an octahedral structure.

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