

A novel Schiff base compound was synthesized, and its complexation properties with Fe(III) and Cr(III) were investigated. Tripodal ligand was synthesized by the reaction of s-triazine and 4-hydroxybenzaldehyde. Then a Schiff base involving 8-hydroxyquinoline was synthesized by the reaction of 5-aminomethyl-8-hydroxyquinoline (QN) and 2,4,6-tris(p-formylphenoxy)-1,3,5-triazine (TRIPOD) in methanol/chloroform media. The obtained Schiff base (QN-TRIPOD) was then reacted with four trinuclear Fe(III) and Cr(III) complexes including tetradentate Schiff bases *N,N'*-bis(salicylidene)ethylenediamine (salenH<sub>2</sub>)/bis(salicylidene)-*o*-phenylenediamine (SalophenH<sub>2</sub>). The synthesized ligand and complexes were characterized by means of elemental analysis carrying out <sup>1</sup>H NMR, FTIR spectroscopy, thermal analyses, and magnetic susceptibility measurements. Finally, metal ratios of the prepared complexes were determined by using atomic adsorption spectrometry.

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## INTRODUCTION

1,3,5-Triazine (or s-Triazines) derivatives are known for a long time and widely used as herbicides, dyestuffs, explosive, surface active agents, drugs, or polymer-like melamine–formaldehyde that has excellent thermal and electrical properties [1–5]. Furthermore, a very large number of supramolecular noncovalently bond assemblies between melamine and barbituric or cyanuric acid derivatives have been reported [6,7]. The chemistry of these compounds has been studied intensively, and they are the subject of many reviews [8–10].

8-Hydroxyquinoline, a monoprotic bidentate chelating agent, has received increased attention recently because its most representative metalloquinolate, aluminum tris(8-hydroxyquinoline), has served as the emitting material in organic light emitting diodes [11]. 8-Hydroxyquinoline metal chelates are presently considered as one of the most reliable electro-transporting and emitting materials applied in molecular-based organic light emitting diodes for their thermal stability, high fluorescence, and excellent electron-transporting mobility [12]. In addition, a large number of 8-hydroxyquinoline derivatives have been synthesized and shown to be bioactive. For example, it was reported that some 8-hydroxyquinoline derivatives possess antitumor and antimicrobial activities [13].

Metal complexes of the “Salen-type” ligands have been considered as interesting species in many fields of chemical research because of some specific properties. Fe-Salen complexes have been extensively studied in the solid state and in solution [14,15]. Kessel and Hendrickson [16] have studied Schiff base complexes of iron(III) incorporating *p*-quinine bridges. The reactions of {[Fe(Salen)]<sub>2</sub>O} with carboxylic acids have been described by Wollmann and Hendrickson [17]. Uysal and Ucan have reported the synthesis and characterization of 1,3,5-tricarboxylato and monocarboxylato bridges with [Salen or SalophenFe(III)/Cr(III)] [18]. Chromium(III) complexes of Salen-type Schiff bases have been found to enhance insulin activity. And also, various metal-Salen complexes such as manganese(III), chromium(III), and nickel(II)-Salen complexes have been used for the epoxidation of olefins [19–21].

The aim of the present study is synthesis of novel tridirectional–trinuclear systems and to present their effects on magnetic behavior of [Salen/SalophenFe(III)] and [Salen/SalophenCr(III)] capped complexes. The reaction of trimeric cyanuric (C<sub>3</sub>N<sub>3</sub>Cl<sub>3</sub>) with 3 equivalent of 4-hydroxybenzaldehyde in benzene media has given the desired trisaldehyde in a single step, coded as 2,4,6-tris(p-formylphenoxy)-1,3,5-triazine (TRIPOD) [22]. Trisaldehyde was then converted to a novel Schiff base [5-aminomethyl-8-hydroxyquinoline (QN)-TRIPOD] with QN. Finally, its

complexation properties investigated with Fe(III) and Cr(III). It may be useful to stress at this point that the new products mentioned previously are the main results of this work.

## RESULT AND DISCUSSION

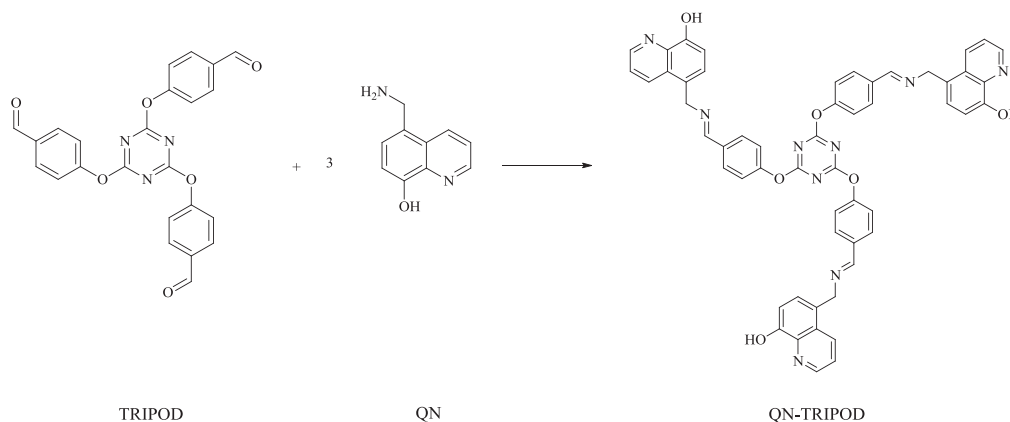
2,4,6-Tris(p-formylphenoxy)-1,3,5-triazine (TRIPOD) was prepared through the reaction of 2,4,6-Trichloro-1,3,5-triazine and 4-hydroxybenzaldehyde in the presence of  $\text{Na}_2\text{CO}_3$ . Then, the tripodal Schiff base (QN-TRIPOD) was synthesized using TRIPOD and 5-aminomethyl-8-hydroxyquinoline in methanol/chloroform media (Scheme 1). The result of this reaction, "Tripodal Oxy-Schiff base" was obtained as a new Schiff base. QN-TRIPOD was characterized by elemental analyses,  $^1\text{H}$  NMR, and FTIR spectroscopy. The FTIR spectrum of QN-TRIPOD is depicted in Figure 2. As can be seen in Figure 2, the obtained QN-TRIPOD was confirmed by the disappearance of aldehyde band ( $1702\text{ cm}^{-1}$ ) in TRIPOD and the appearance of the imine bands belong to a QN-TRIPOD compound ( $\text{CH}=\text{N}$ ) at  $1636\text{ cm}^{-1}$ . QN-TRD prepared in this way was obtained in nearly quantitative yield and high purity. Synthetic strategy for preparing Tripodal-Trinuclear uses a complex as a "ligand" that contains a potential donor group capable of coordinating to another ligand.  $\{[\text{Fe}/\text{Cr}(\text{Salen})]_2\text{O}\}$  and  $\{[\text{Fe}/\text{Cr}(\text{Salophen})]_2\text{O}\}$  have been chosen as ligand complex because they can coordinate to another ligand [26]. These complexes are the first examples of tripodal-trinuclear complexes bridged by catechol groups and pyridine to the iron and chromium centers (Scheme 2). The vibrations of three strong bands the azomethine  $-\text{C}=\text{N}$ , triazine  $\text{C}=\text{N}$ , and OH of the free ligand are observed at  $1636$ ,  $1562$ , and  $3407\text{ cm}^{-1}$  range in the FTIR spectra. FTIR spectra of all trinuclear complexes show  $\text{C}=\text{N}$  stretching bands for the azomethine group at  $1601\text{--}1623\text{ cm}^{-1}$ , for the triazine bands at  $1565\text{--}1574\text{ cm}^{-1}$ , for the cap parts of the complexes

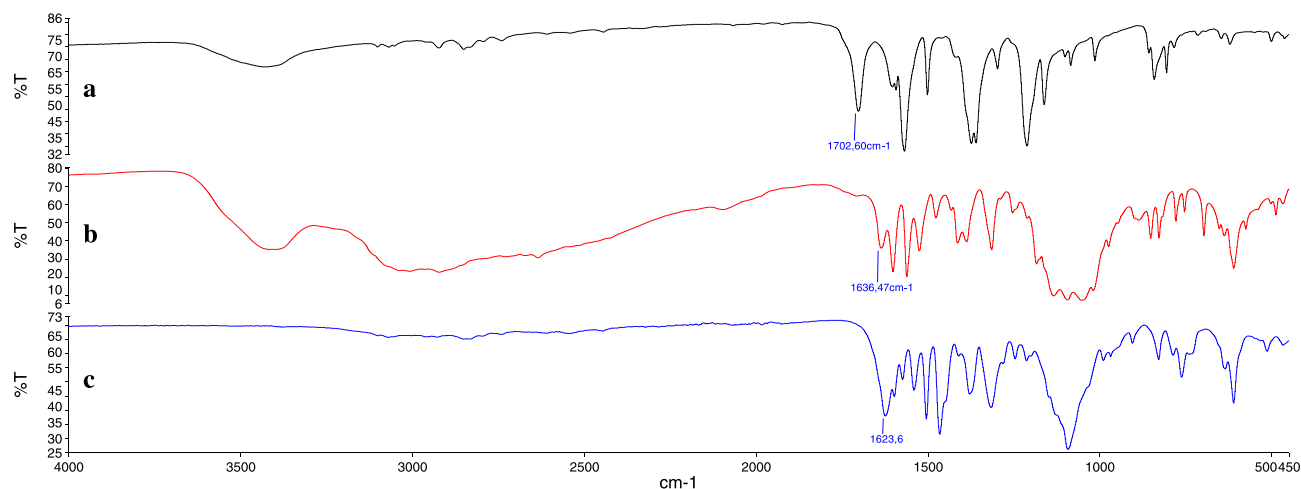
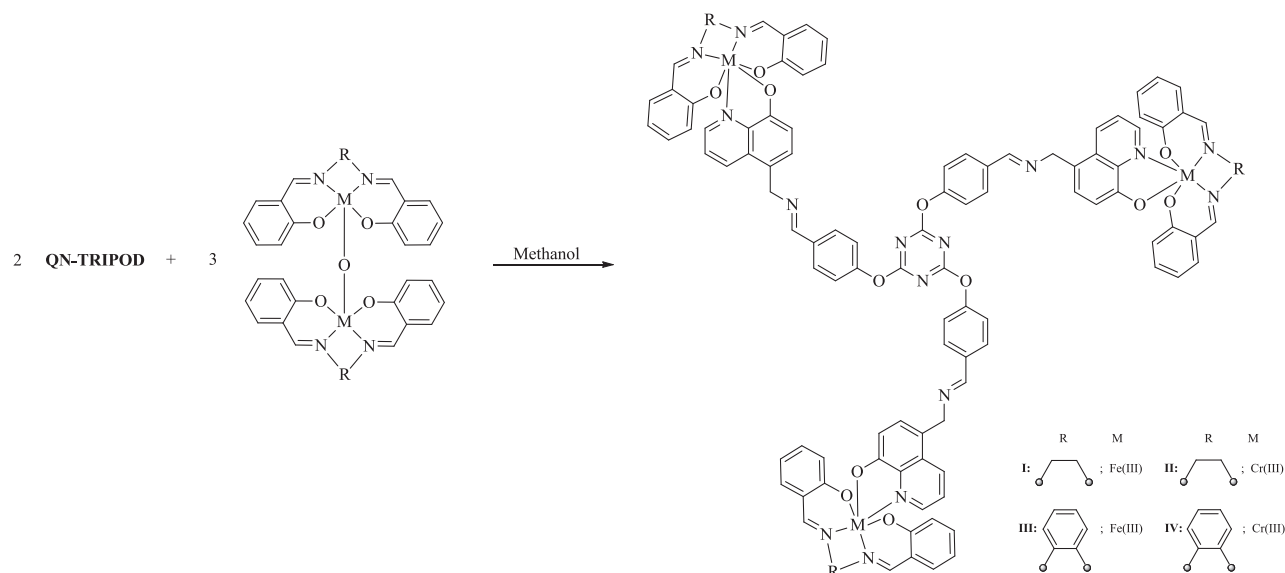
at  $1534\text{--}1552\text{ cm}^{-1}$ . In the complexes, these  $\text{C}=\text{N}$  bands shift to lower frequencies for cap parts of the complexes, indicating that the OH groups of the tripodal oxy-Schiff base ligand are coordinated to metal center of the tripodal-trinuclear complexes [18]. In the ligand QN-TRIPOD, the band at  $3407\text{ cm}^{-1}$  can be assigned to the  $-\text{OH}$  group vibrations. In the tripodal-trinuclear complexes, which these bonds disappear, is evidence that  $-\text{OH}$  group coordinated to metal center of ligand complexes. We have suggested that oxygen atom (for complexes **I**, **II**, **III**, and **IV**)  $-\text{OH}$  group coordinated to metal centers. In the tripodal-trinuclear complexes, the bands in the  $547\text{--}565$  and  $464\text{--}475\text{ cm}^{-1}$  ranges can be attributed to the  $\text{M}\text{--}\text{N}$  and  $\text{M}\text{--}\text{O}$  stretching modes (Fig. 1) [25,27].

In order to identify the structures of the new Tripodal Oxy-Schiff base and TRIPOD,  $^1\text{H}$  NMR were used. Spectra were recorded in  $\text{DMSO-}d_6$ . And the expected chemical shifts were observed. The obtained compound was confirmed by the disappearance of aldehyde protons ( $9.97\text{ ppm}$ ) in TRIPOD, and the appearance of the imine protons belongs to a QN-TRIPOD compound ( $-\text{CH}=\text{N}$ ) at  $8.91\text{ ppm}$ .  $^1\text{H}$  NMR spectra also confirmed the structure of the synthesized Schiff base. And also, the other  $^1\text{H}$  NMR spectrum values of QN-TRIPOD were observed as expected. For the complex structures because the Fe(III) and Cr(III) metals are paramagnetic  $^1\text{H}$  NMR spectra did not be taken (Fig. 2).

The magnetic behavior of Fe(III) and Cr(III) complexes is in accord with proposed trinuclear structures. The magnetic moments of the trinuclear complexes indicate paramagnetic properties with magnetic susceptibility values per atom of 1.63, 1.72, 3.51, and 3.63 BM, respectively. The equation simplifies to:  $\mu_{\text{eff.}} = 2.84\sqrt{\chi_M T}$  BM. The measured  $\mu_{\text{eff}}$  can be compared with the calculated value. The spinning of unpaired electrons in paramagnetic complexes of d-block metal ions creates a magnetic field, and these spinning electrons are in effect small magnets. The magnetic susceptibility,  $\mu$ , because of the spinning of the electrons is given by the spin-only formula:  $\mu_s = \sqrt{n(n+2)}$ , where  $n$  is the

**Scheme 1.** Synthesis of a novel Schiff base bearing 5-aminomethyl-8-hydroxyquinoline (QN-TRIPOD).



**Scheme 2.** Synthesis of tripodal–trinuclear complexes of QN-TRIPOD.**Figure 1.** FTIR spectra of (a) 2,4,6-tris(p-formylphenoxy)-1,3,5-triazine (**TRIPOD**), (b) 2,4,6-tris(2,5-aminomethyl-8-hydroxyquinoline-4'-formylphenoxy)-1,3,5-triazine (**QN-TRIPOD**), (c) tripodal–trinuclear complex compound (**II**). [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

number of unpaired electrons. When  $n$  is 1 and 3, the calculated values of  $\mu_s$  are 1.73 and 3.87 BM. The measurements and calculated values of  $\mu$  show us that the  $\{[\text{Fe}(\text{Salen}/\text{saloph})]_2\text{O}\}$  and  $\{[\text{Cr}(\text{Salen}/\text{Salophen})]_2\text{O}\}$  containing compounds appear to be represented by the electronic structures of  $t_2g^5eg^0$  and  $t_2g^3eg^0$ . The magnetic data for the  $\{[\text{Fe}(\text{Salen})]_2\text{O}\}$ ,  $\{[\text{Fe}(\text{Salophen})]_2\text{O}\}$ ,  $\{[\text{Cr}(\text{Salen})]_2\text{O}\}$ , and  $\{[\text{Cr}(\text{Salophen})]_2\text{O}\}$  tripodal complexes demonstrate strong 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 also suggesting that these trinuclear complexes have an octahedral structure [1,18,27].

The thermal properties (TG, DTA) of all tripodal complexes  $\{[\text{Fe}/\text{Cr}(\text{Salen}/\text{Salophen})]$  capped complexes of **QN-**

**TRIPOD} were investigated. And their plausible degradation schemes are presented in Table 1. Complexes at 22–100°C are because of the loss of moisture [23]. It was determined in two steps. Firstly,  $[\text{Fe}/\text{Cr}(\text{Salen}/\text{Salophen})]$  caps have been left the main structure at 244–556°C. After, when the sample was heated to 556°C, main body started to decompose. At this step, the weight loss carried out the temperature at 556–703°C. Thermal measurement has been taken until 1000°C. The second steps have corresponded to the decompositions of the aromatic groups and the residual ligand. The final decomposition products were metaloxides, quinoline, and the triazine ring. The observed weight losses for complexes are in good agreement with the calculated values [1,25,27] (Fig. 3).**

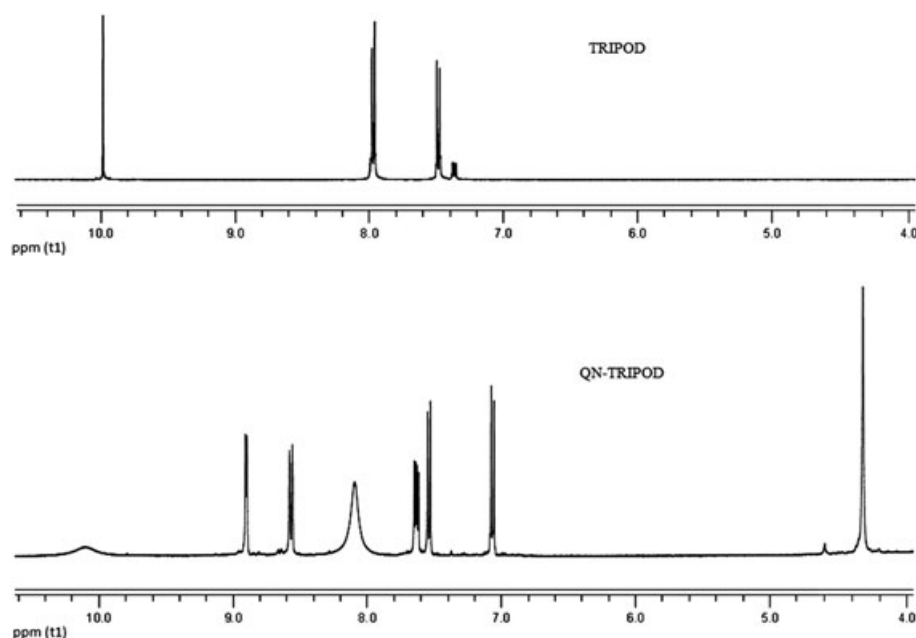


Figure 2. The  $^1\text{H}$  NMR spectra of TRIPOD and QN-TRIPOD.

Table 1

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

Compound	Step no	Temp. range ( $^{\circ}\text{C}$ )	Weight loss found (Calcd.) (%)	Fragment <sup>a</sup>
<b>I</b>	1	101–244	49.41 (49.44)	$\text{N}_2$ , $\text{CO}_2$ , $\text{C}_2\text{H}_4$ , $\text{CO}$ , $\text{NH}_3$ , $\text{H}_2$ , $\text{C}_6\text{H}_6$
	2	244–469		
	3	469–703		
	4	703–980		
<b>II</b>	1	100–304	46.11 (49.10)	$\text{N}_2$ , $\text{CO}_2$ , $\text{C}_2\text{H}_4$ , $\text{CO}$ , $\text{NH}_3$ , $\text{H}_2$ , $\text{C}_6\text{H}_6$
	2	304–556		
	3	556–664		
	4	664–998		
<b>III</b>	1	105–292	50.98 (53.03)	$\text{N}_2$ , $\text{CO}_2$ , $\text{CO}$ , $\text{NH}_3$ , $\text{H}_2$ , $\text{C}_6\text{H}_6$
	2	292–534		
	3	534–692		
	4	692–987		
<b>IV</b>	1	103–256	51.78 (53.91)	$\text{N}_2$ , $\text{CO}_2$ , $\text{CO}$ , $\text{NH}_3$ , $\text{H}_2$ , $\text{C}_6\text{H}_6$
	2	256–497		
	3	497–689		
	4	689–976		

<sup>a</sup>These are conjectural data.

## CONCLUSION

In this study, a new tripodal *s*-triazine based Schiff base “2,4,6-tris(2,5-amminomethyl-8-hydroxyquinoline-4'-formylphenoxy)-1,3,5-triazine” was synthesized and its complexes were prepared. 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  $[\text{Fe}(\text{Salen})_2\text{O}]$ ,  $[\text{Fe}(\text{Salophen})_2\text{O}]$ ,  $[\text{Cr}(\text{Salen})_2\text{O}]$ , and  $[\text{Cr}(\text{Salophen})_2\text{O}]$  as “ligand complexes” because they can coordinate to the other ligands. These complexes are the examples of tripodal–trinuclear complexes bridged by catechol groups to the iron and chromium

centers. Their structures were characterized by means of elemental analysis carrying out  $^1\text{H}$  NMR, FTIR spectroscopy, thermal analyses, and magnetic susceptibility measurements. Finally, metal ratios of the prepared complexes were determined by using atomic adsorption spectrometry. The magnetic data for tripodal complexes show good harmony with the  $d^5$  and  $d^3$  metal ions in an octahedral structure.

## EXPERIMENTAL

All chemicals were purchased from Aldrich, Merck, and Alfa-Easer. The linking agent, 2,4,6-trichloro-1,3,5-triazine

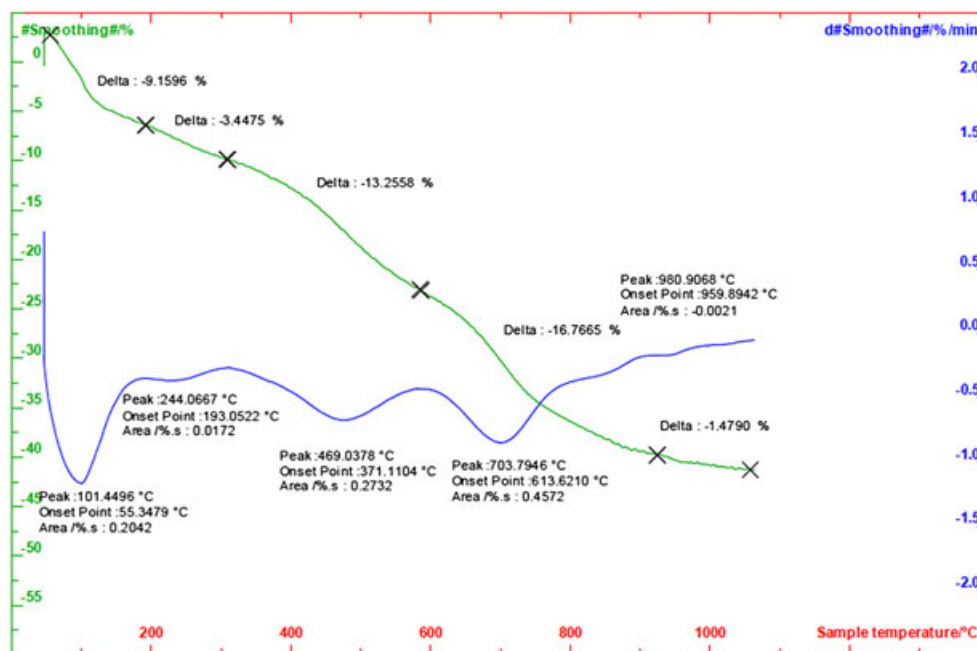


Figure 3. The TGA-DTA diagram of I. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

(abbreviated as cyanuric chloride, mp 145–146°C), was obtained from Aldrich. Cyanuric chloride was purified by recrystallizations from pure petroleum ether (60–90°C) [23].

<sup>1</sup>H NMR spectra were recorded by the Varian, 400 MHz spectrometer at room temperature. FTIR spectra were recorded using a Perkin Elmer Spectrum 100 with Universal ATR Polarization Accessory. Melting points were determined by Büchi Melting Point B-540 instrument. Elemental analyses were carried out using a LECO-CHNS-932 elemental analyzer. Thermal analysis (TG) was carried out with a Seteram thermogravimetric analyzer. The sample weight was 15–17 mg. TG curves were obtained at the heating rate of 10°C/min from 22 to 1000°C under argon atmosphere with a gas flow rate of 20 mL/min. Metal contents in complexes were determined using Unicam 929 AAS spectrometer. Magnetic susceptibilities of metal complexes were measured at 296 K using a Sheerwood Scientific MX Gouy magnetic susceptibility apparatus and carried out using the Gouy method with Hg[Co(SCN)<sub>4</sub>] 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.

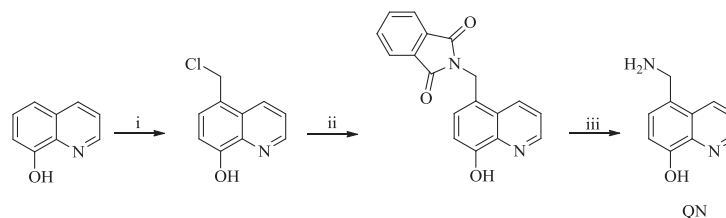
**Synthesis of 5-chloromethyl-8-hydroxyquinoline.** 5-Chloromethyl-8-hydroxyquinoline was synthesized according to the literature [12] with a slightly modification. Thirty-seven percent of formaldehyde (6.4 mL, 0.08 mol) was added to a solution of 8-hydroxyquinoline (5.84 g, 0.04 mmol) in concentrated hydrochloric acid (50 mL). The mixture was treated with hydrogen chloride gas until the solid formed and allowed to stand overnight at the room temperature. The yellow

solid was collected on a filter and washed with condensed hydrochloric acid three or four times instead of acetone. Dried in vacuo at 40°C. Yield: 91.4%, mp: 280°C. FTIR (cm<sup>-1</sup>): 3298 (–OH), 1595 (pyridine ring), 1550, 1493 (aromatic), 1388 (C–N), 695 (C–Cl).

**Synthesis of 5-aminomethyl-8-hydroxyquinoline.** QN and 2-(hydroxyquinoline-5-ylmethyl)-isoquinoline-1,3-diketone were synthesized according to the procedure [24]. 2-(Hydroxyquinoline-5-ylmethyl)-isoquinoline (2.0 g, 6.5 mmol) was added to the concentrated hydrochloric acid (50 mL) stirred at room temperature 1 h, then refluxed until the mixture become transparent. After cooling to room temperature, the resulting solid was filtered, and the filtrate was concentrated on a rotary evaporator. The resulting yellow residue was dissolved in 10 mL water, then triethylamine was added, until greenish solid formed, filtered. The solid formed was washed with cold water, dried under vacuum cabinets at room temperature to give compound QN. FTIR (cm<sup>-1</sup>): 3292, 2974, 2650, 1558, 1508, 1454, 1298, 1077. <sup>1</sup>H-NMR (DMSO-*d*<sub>6</sub>, ppm):  $\delta$ =4.03 (s, 2H, –CH<sub>2</sub>), 7.01 (d, 1H, Ar–H), 7.42 (d, 1H, Ar–H), 7.56 (m, 1H, Ar–H), 8.53 (d, 1H, Ar–H), 8.82 (d, 1H, Ar–H) (Scheme 3).

**Synthesis of TRIPOD.** TRIPOD was synthesized according to the literature procedure [22]. p-Hydroxybenzaldehyde (1.6 g, 1.31 mmol) and 2,4,6-Trichloro-1,3,5-triazine (0.6 g, 3.25 mmol) were added to a suspension of Na<sub>2</sub>CO<sub>3</sub> (10 g) in 50 mL of benzene. The mixture was refluxed for 20 h. The reaction mixture was then cooled, and the solid was removed by filtration and washed with hot AcOEt twice. The filtrate was extracted

**Scheme 3.** Synthetic route for 5-aminomethyl-8-hydroxyquinoline (QN). Reagents and reaction condition: (i) formaldehyde, gaseous HCl, room temperature 12 h; (ii) potassium phthalimide, DMF, 150°C 5 h, (iii) concentrated HCl, reflux.



**Table 2**

Some physical properties, effective magnetic moments, elemental analyses, and AAS analyses of the ligands and complexes.

Compound	Formula	Color	$\mu_{eff}$ (BM)	mp (°C)	Yield (%)	Found (Calcd.)(%)			
						C	N	H	M
TRIPOD	C <sub>24</sub> H <sub>15</sub> N <sub>3</sub> O <sub>6</sub>	White	—	174	85	65.26 (65.31)	9.61 (9.52)	3.39 (3.43)	—
QN	C <sub>10</sub> H <sub>10</sub> N <sub>2</sub> O	Greenish	—	96	80	68.84 (68.95)	16.12 (16.08)	5.72 (5.79)	—
QN-TRIPOD	C <sub>54</sub> H <sub>39</sub> N <sub>9</sub> O <sub>6</sub>	Cream	—	156	70	71.22 (71.28)	13.91 (13.85)	4.28 (4.32)	—
I	C <sub>102</sub> H <sub>78</sub> N <sub>15</sub> O <sub>12</sub> Fe <sub>3</sub>	Dark Brown	1.63	325 <sup>a</sup>	75	65.34 (65.40)	11.31 (11.22)	4.12 (4.20)	8.83 (8.94)
II	C <sub>102</sub> H <sub>78</sub> N <sub>15</sub> O <sub>12</sub> Cr <sub>3</sub>	Dark Green	3.51	350 <sup>a</sup>	65	65.72 (65.80)	11.34 (11.28)	4.11 (4.22)	8.43 (8.38)
III	C <sub>114</sub> H <sub>78</sub> N <sub>15</sub> O <sub>12</sub> Fe <sub>3</sub>	Brown	1.72	330 <sup>a</sup>	70	67.73 (67.87)	10.49 (10.41)	3.83 (3.90)	8.22 (8.30)
IV	C <sub>114</sub> H <sub>78</sub> N <sub>15</sub> O <sub>12</sub> Cr <sub>3</sub>	Green	3.63	365 <sup>a</sup>	60	68.19 (68.26)	10.52 (10.47)	3.83 (3.92)	7.69 (7.78)

<sup>a</sup>Decomposition temperature.

with 10% Na<sub>2</sub>CO<sub>3</sub> twice and with H<sub>2</sub>O once. The organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and then concentrated. The white powder was recrystallized from 20 mL of AcOEt.

**Synthesis of QN-TRIPOD.** 5-Aminomethyl-8-hydroxyquinoline (0.522 g, 3 mmol) was added drop by drop to a solution of 2,4,6-tris(p-formylphenoxy)-1,3,5-triazine (0.442 g, 1 mmol) in 30 mL methanol/chloroform solution (1/1), and then three drops of concentrated H<sub>2</sub>SO<sub>4</sub> was added as catalyst in 50 mL methanol/chloroform solution (1/1). The mixture was then stirred under reflux for 24 h. The mixture was cooled to room temperature and yellow powder product was filtered. The precipitate was washed with cool ethanol. FTIR (cm<sup>-1</sup>): 3407 (–OH), 1636 (–CH=N), 1562 (C=N<sub>triazine</sub>); <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>):  $\delta$ =4.41 (s, 6H, –CH<sub>2</sub>), 7.09 (d, 3H, ArH), 7.56 (d, 3H, ArH), 7.63–7.67 (m, 15H, ArH), 8.1 (s, 3H, –CH), 8.57 (d, 3H, ArH), 8.89 (d, 3H, ArH), 10.10 (s, 3H, OH).

**Preparation of ligand complexes.** [Fe(Salen)]<sub>2</sub>O, [Fe(Salophen)]<sub>2</sub>O, [Cr(Salen)]<sub>2</sub>O, and [Cr(Salophen)]<sub>2</sub>O were prepared by addition of concentrated ammonia solution until alkaline. It was to the hot EtOH solutions of [Fe(Salen)]Cl, [Fe(Salophen)]Cl, [Cr(Salen)]Cl, and [Cr(Salophen)]Cl, respectively [25].

**General procedure for the reaction of tripodal complexes (I, II, III, and IV).** A solution of QN-TRIPOD (1 mmol) in 20 mL absolute methanol was added to the drop wise suspension of {[Fe/Cr(Salen)]<sub>2</sub>O}/[Fe/Cr(Salophen)]<sub>2</sub>O (1.6 mmol) in 20 mL of absolute methanol, and the

mixture were refluxed for 4 h. The mixture was allowed to cool to room temperature, and the obtained product was filtered, washed with water, ethanol and diethyl ether, and dried in vacuo (0.4 bar) at 60°C. The FTIR (cm<sup>-1</sup>) data for **I**: 2859 (CH), 1620 (–CH=N), 1574 (C=N triazine), 1377 (COC), 547 (M–N), 469 (M–O). **II**: 2862 (CH), 1603 (–CH=N), 1575 (C=N triazine), 1369 (COC), 551 (M–N), 475 (M–O). **III**: 2824 (CH), 1615 (–CH=N), 1578 (C=N triazine), 1368 (COC), 549 (M–N), 464 (M–O). **IV**: 2829 (CH), 1600 (–CH=N), 1578 (C=N triazine), 1368 (COC), 565 (M–N), 471 (M–O). The elemental analyses, magnetic, and physical properties of the synthesized complexes are given in Table 2.

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