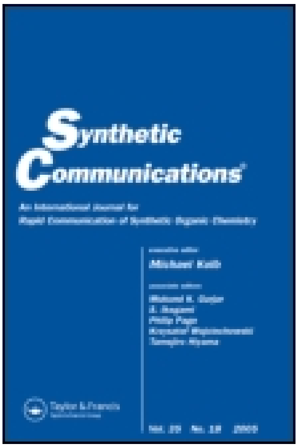

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Synthesis and Characterization of S-Triazine Cored Tripodal Keto-oxime and Its Trinuclear Complexes

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SYNTHESIS AND CHARACTERIZATION OF S-TRIAZINE CORED TRIPODAL KETO-OXIME AND ITS TRINUCLEAR COMPLEXES

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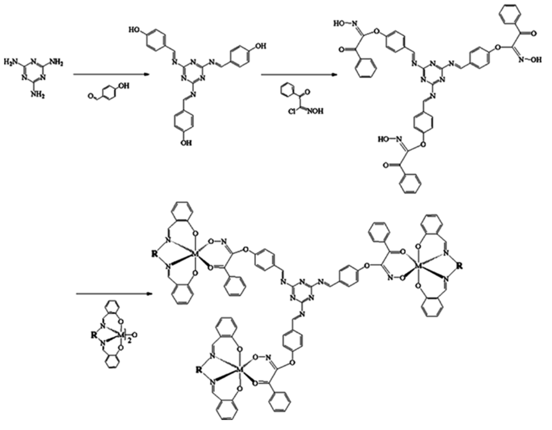
GRAPHICAL ABSTRACT



Abstract *2,4,6-Tris(4-hydroxybenzimino)-1,3,5-triazine (**II**) has been synthesized by the reaction of 1 equiv. melamine **(I)** and 3 equiv. 4-hydroxybenzaldehyde and characterized by means of elemental analysis, ^1H NMR, Fourier transform–infrared spectroscopy, and liquid chromatography–mass spectrometry (LC-MS). 2,4,6-tris(4-(2-phenyl-2-keto-hydroxyiminoyloxy)benzimino)-1,3,5-triazine **L (III)** has been synthesized by the reaction of 1 equiv. **II** and 3 equiv. chloroisonitrozoacetophenone and characterized by means of the same methods. Four novel trinuclear Fe(III) and Cr(III) complexes involving tetradentate Schiff bases N,N′-bis(salicylidene)ethylenediamine-(salenH_2) or bis(salicylidene)-o-phenylenediamine-(salophen H_2) with **L (III)** have been synthesized and characterized by means of elemental analysis, Fourier transform–infrared spectroscopy, LC-MS (ESI^+), and thermal analyses. The metal ratios of the prepared complexes have been determined using atomic absorption spectroscopy. The aim of the present study is to synthesize novel tridirectional-trinuclear systems and to present their effects on magnetic behavior of [salen or salophenFe(III)/Cr(III)] capped complexes. The complexes have also been characterized as low-spin distorted octahedral Fe(III) and as distorted octahedral Cr(III) bridged by keton-oxime group.*

Keywords Keton-oxime groups; melamine; salen; salophen; Schiff bases

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INTRODUCTION

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] and spolymers,[3] such as melamineformaldehyde, which has excellent thermal and electrical properties.[4] Interest in the metal coordination environment has prompted the study of oxime ligands because of their variable geometries and the tunability of their substituents. Oxime ligands are used as analytical reagents and serve as models for biological systems (e.g., vitamin B12 and dioxygen carriers) as well as catalysts in chemical processes.[5]

The anions of salen or salen-type ligands and their complexes have been regarded as interesting varieties in many fields of chemical research because of certain specific properties. Fe(salen) complexes have been extensively studied in solid state and in solution. Barone et al.[6] showed that such systems exhibited catalytic activity toward the blend oxidation of hydrocarbons and their electron-transfer reactions, mimicking the catalytic functions of peroxidases. Woldemariam et al.[7] synthesized a water soluble Fe(III)-salen complex and studied its biochemical effects on DNA in vitro and on cultured human cells. We have also reported that the synthesis and characterization of mono-/tri-/multi-carboxylato or mono-/tri-catechol bridges with [salen or salophenFe(III)/Cr(III)].[8–13] Singh and Gupta[14] have also studied synthesis, structure, and physicochemical properties of melamine-based dendrimers. We have especially preferred chromium complexes because chromium is a unique transition-metal ion, which is biologically significant at all the levels of living organisms.[15] Chromium(III)-salen complexes in the homogeneous phase have also been used for the epoxidation of olefins.[16,17]

The aim of the present study is to synthesize novel tridirectional-trinuclear systems and to present their effects on magnetic behavior of [salen or salophenFe(III)/Cr(III)] capped complexes. We also report that synthesized tridirectional melamine Schiff bases present a new scaffold. Melamine ($C_3N_6H_6$) was reacted with 3 equiv. of 4-hydroxybenzaldehyde in benzene and bolied under reflux, and the desired tris-iminophenol (**II**) 2,4,6-tris(4-hydroxybenzimino)-1,3,5-triazine was obtained in a one step.[18,19] Then, as dry Cl_2 gas was bubbled through a suspension of isonitrosoacetophenone in chloroform under sunlight for 30 min, chloroisonitrozoacetophenone was obtained. Compound **II** was reacted with 3 equiv. of chloroisonitrozoacetophenone in acetone at room temperature, and the targed ligand (**L**) 2,4,6-tris(4-(2-phenyl-2-ketohydroxyiminoyloxy)benzimino)-1,3,5-triazine was obtained in one step. Their structures are characterized by Fourier transform–infrared (FT-IR), ^{1}H NMR, liquid chromatography–mass spectroscopy (LC-MS), thermal gravimetric analysis (TGA), and magnetic suscebtibility. The metal ratios of the prepared complexes have been determined using atomic absorption spectroscopy (AAS).

RESULTS AND DISCUSSION

The target ligand **L (III)** was synthesized in two steps from melamine. The conversion of melamine to the triarmed-melamine-oxime derivative was accomplished with 68% yield. The structural formula of **L (III)** was verified by elemental analyses, ^{1}H NMR, FT-IR, and mass spectral data[20] (Fig. 1, Tables 1 and 2). The ligand is

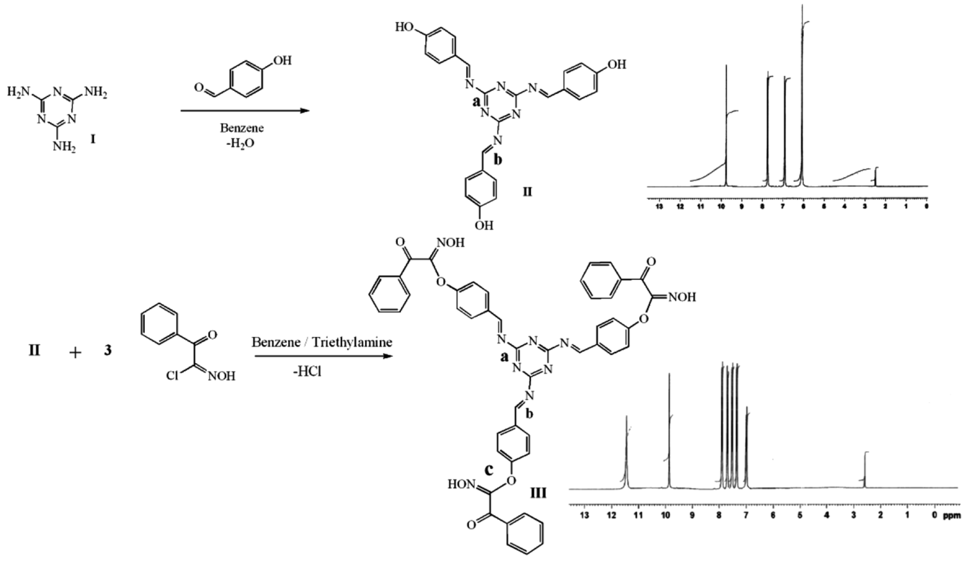

Figure 1. Synthetic routes for the preparation of ligand **III**.

soluble in common organic solvents. The synthetic strategy for preparing **L (III)** utilizes a complex as a ligand that contains a potential donor group capable of coordinating to another ligand. Therefore, we chose [Fe/Cr(salen or salophen)]$_2$O as ligand complexes.[21] These complexes are the first examples of melamine-based trinuclear complexes bridged to the iron/chromium centers by keton and oxime groups (Fig. 2). All compounds are stable at room temperature in the solid state, and they are only soluble in organic solvents such as ethylacetate, dimethylsulfoxide (DMSO), dimethylformamide (DMF), and are insoluble in water. The results of

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

Compounds	μ_B	Mp (°C)	Yield (%)	Color [M_W (g/mol)]	**Found** and Calculated (%)				
					C	H	N	Fe	Cr
$C_{24}H_{18}N_6O_6$	—	142	72	White	**65.67**	**4.12**	**19.15**	—	—
(II)				[438]	65.75	4.14	19.17		
$C_{48}H_{33}N_9O_9$	—	196	68	White	**64.98**	**3.56**	**14.21**		
L **(III)**				[880]	65.53	3.75	14.33	—	—
$C_{96}H_{72}N_{15}O_{15}Fe_3$	1.73	243^a	54	Brown	**61.94**	**4.61**	**11.27**	**9.02**	—
L(FeSalen)$_3$ **(IV)**				[1842]	62.56	4.91	11.40	9.10	
$C_{108}H_{72}N_{15}O_{15}Fe_3$	1.75	276^a	56	Brown	**65.03**	**3.48**	**10.36**	**8.29**	—
L(FeSalophen)$_3$ **(V)**				[1986]	65.27	3.63	10.58	8.44	
$C_{96}H_{72}N_{15}O_{15}Cr_3$	3.72	226^a	45	Green	**62.43**	**3.85**	**11.35**	—	**9.23**
L(CrSalen)$_3$ **(VI)**				[1830]	62.95	3.93	11.48		9.62
$C_{108}H_{72}N_{15}O_{15}Cr_3$	3.78	248^a	48	Green	**65.12**	**3.44**	**10.36**	—	**8.71**
L(CrSalophen)$_3$ **(VII)**				[1974]	65.65	3.65	10.64		8.92

[a] Decomposition.

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

Compound	Step	Temp. range (°C)	Weight loss found (**calcd.**) (%)	Fragment
$C_{96}H_{72}N_{15}O_{15}Fe_3$	**I**	165–260	25.76 (**26.34**)	$3C_6H_6$, $3NO_2$, 3CO
L(FeSalen)$_3$ **(IV)**	**II**	295–365	42.54 (**43.48**)	$3C_2H_4$, $3N_2$, 6CO, $6C_6H_6$
	III	560–675	15.98 (**16.71**)	$3/2N_2$, $3/2C_2H_2$, $3C_6H_6$
$C_{108}H_{72}N_{15}O_{15}Fe_3$	**I**	170–275	23.86 (**24.44**)	$3C_6H_6$, $3NO_2$, 3CO
L(FeSalophen)$_3$ **(V)**	**II**	315–375	46.65 (**47.57**)	$3N_2$, 6CO, $9C_6H_6$
	III	560–672	14.89 (**15.50**)	$3/2N_2$, $3/2C_2H_2$, $3C_6H_6$
$C_{96}H_{72}N_{15}O_{15}Cr_3$	**I**	125–240	25.82 (**26.55**)	$3C_6H_6$, $3NO_2$, 3CO
L(CrSalen)$_3$ **(VI)**	**II**	275–325	42.91 (**43.74**)	$3C_2H_4$, $3N_2$, 6CO, $6C_6H_6$
	III	560–676	16.44 (**16.81**)	$3/2N_2$, $3/2C_2H_2$, $3C_6H_6$
$C_{108}H_{72}N_{15}O_{15}Cr_3$	**I**	132–248	23.97 (**24.62**)	$3C_6H_6$, $3NO_2$, 3CO
L(CrSalophen)$_3$ **(VII)**	**II**	286–335	47.11 (**47.83**)	$3N_2$, 6CO, $9C_6H_6$
	III	560–670	14.93 (**15.59**)	$3/2N_2$, $3/2C_2H_2$, $3C_6H_6$

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.

When chloroisonitrosoacetophenone was added to tripodal Schiff base ligand **II**, the phenolic OH band at 3338 cm^{-1} disappeared. In the FT-IR spectra of **L III**, the vibrations of the triazine C=N (**a**), azomethine C=N (**b**), oxime C=N (**c**), carbonyl C=O, and C−O−C of the free ligands were observed at 1576, 1622, 1653, 1694, and 1372 cm^{-1} range, respectively.[22–24] The bands at 3258 cm^{-1} and 958 cm^{-1} can be assigned to the oxime-O-H and oxime N-O group vibrations, respectively.[25] IR bands at 1658–1654 cm^{-1} for complex **IV–VII** were assigned to C=N (**c**) stretching vibrations, 1008–1004 cm^{-1} were assigned to N-O stretching vibrations, and 1538–1544 cm^{-1} were assigned to C=N (**d**) stretching vibrations for complexes **IV–VII**, whereas C=N (**d**) stretching vibration bands were found at 1560–1567 cm^{-1} for [Fe/Cr(salen or salophen)]$_2$O complexes.[8–13,22,23,26] In the FT-IR spectra of tripodal-trinuclear complexes **IV–VII**, the oxime OH band disappeared, and oxime N-O bands shifted upward, suggesting chelation of oxygen atoms to the metal. Bands

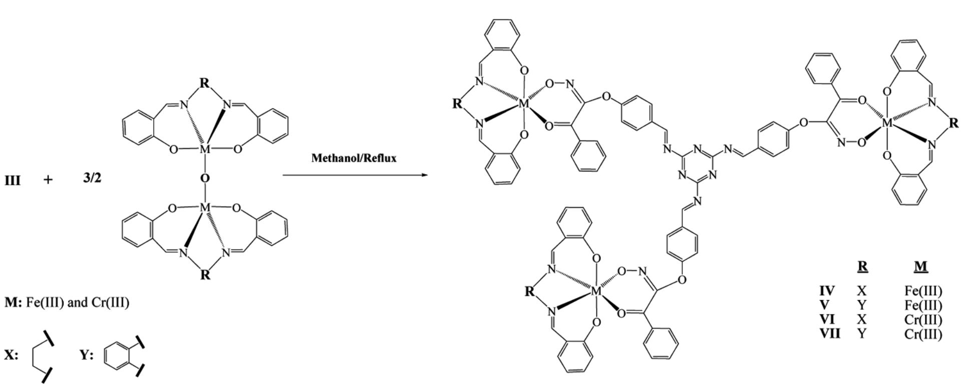

Figure 2. Synthetic routes for the preparation of complexes.

at 1709–1704 cm^{-1} for complexes **IV–VII**, in which the IR bands for C=O vibrations of tripodal complexes have shifted upward, were also assigned to C=O groups because of coordination of [Fe/Cr(salen or salophen)]$_2$O to keton-oxime groups. For the tripodal-trinuclear complexes **IV–VII**, the bands in the 544–532 and 478–468 cm^{-1} ranges can be attributed to the M-N and M-O stretching modes.[27]

To identify the structures of the ligand **III**, the ^{1}H NMR spectra were recorded in DMSO-d$_6$.[28,29] In the ^{1}H NMR spectra of chloroisonitrosoacetophenone, the absence of a singlet at $\delta = 8.40$ ppm showed that this compound was formed. In the ^{1}H NMR spectra of **II**, the presence of a singlet at $\delta = 6.08$ ppm for three phenolic OH and of a singlet at $\delta = 9.77$ ppm for Mel–N=**CH**-Ph showed that a three-directional link to melamine had occurred.[18,19,24] Then, also ^{1}H NMR spectra of the confirmed structures of the synthesized compounds **III** revealed that the value (at $\delta = 6.08$ ppm) of OH chemical shift disappeared. The signal in ^{1}H NMR spectrum of **III** at δ 11.50 ppm corresponded to the oxime –OH proton resonances.

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 an octahedral environment. The magnetic behavior of Fe(III) and Cr(III) complexes are good harmony with the proposed trinuclear structures.[29] The magnetic moment per trinuclear complexes, which were constructed from [Fe/Cr(salen or salophen)] and **III**, shows a paramagnetic property with a magnetic susceptibility value per atom of 1.73–1.75 B.M. and 3.72–3.78 B.M., respectively. It was seen that the [Fe/Cr(salen or salophen)]–containing compounds were represented by the electronic structure of $t_{2g}^5e_g^0$ and $t_{2g}^3e_g^0$. The magnetic data for the [Fe/Cr(salen or salophen)] triarmed complexes showed good harmony with the low-spin d^5 (S = 1/2) Fe(III) and d^3 (S = 3/2) Cr(III) ion in an octahedral structure. This consequence was supported by the results of the elemental analyses suggesting that these trinuclear complexes are six coordinate structures.[8–13,25,29,30]

All the complexes have also been thermally investigated, and their plausible degradation schemes are presented in Table 2.[31,32] Thermal decomposition of the three-armed complexes **IV–VII** have started in range of the first step 125–275 °C, and the anhydrous [Fe/Cr(salen or salophen)] complexes detached from ligand **III**. The thermal analyses (TG, DTA) of complexes **IV–VII** showed weight losses theoretically calculated to be 26.34%, 24.44%, 26.55%, and 24.62%; experimentally they were observed to be 25.76%, 23.86%, 25.82%, and 23.97%, respectively. It can be attributed to decomposition of keton-oxime groups in this step. The second decomposition step was observed in the range 275–375 °C. The thermal analyses (TG, DTA) of complexes **IV–VII** showed weight losses theoretically calculated to be 43.48%, 47.57%, 43.74%, and 47.83%; experimentally they were observed to be 42.54%, 46.65%, 42.91%, and 47.11%, respectively. It can be attributed to decomposition of salen or salophen groups in this step. The final decomposition step was completed in the range of 560–676 °C. The final decomposition products were metals and triazine ring. The observed weight losses for all ligands and complexes are in good harmony with the calculated values.

LC-MS (ESI$^+$) spectrum of all ligand and their complexes were investigated. The calculated molecular weights of all ligand and complexes have been given in Table 1. Molecular peaks of the cations are observed with the same isotope distribution as the calculated ones, theoretically. That is, from the investigation of LC-MS spectra of the compounds, it has been seen that molecular weights of ligands and complexes are in good harmony with the intensity values observed in LC-MS spectra.

CONCLUSIONS

In this study, novel tri-directional and melamine cored Schiff base **L III** was synthesized. The strategy of synthesis for preparing tripodal-trinuclear used a complex as a ligand that contained a potential donor group capable of coordinating to the other ligand. We chose [Fe/Cr(salen or salophen)]$_2$O as ligand complexes because they could coordinate to the other ligand. These complexes were the first examples of tripodal-trinuclear complexes bridged by keton-oxime groups to the iron and chromium centers. Their structures were characterized by means of elemental analysis, ^{1}H NMR, FT-IR spectroscopy, LC-MS, thermal analyses, and magnetic susceptibility measurements. The magnetic data for the tripodal-trinuclear complexes showed good harmony with the low spin d^5 ($S = 1/2$) Fe(III) and d^3 ($S = 3 \times 1/2$) Cr(III) ion in an octahedral structure.

EXPERIMENTAL

Materials and Methods

Melamine, 4-hydroxybenzaldehyde, and all other reagents were purchased from Merck and used without further purification. 2,4,6-Tris(4-hydroxybenzimino)-1,3,5-triazine used in this study was synthesized in a previous study.[18,19] [Fe/Cr(salen or salophen)]$_2$O was prepared according to previously published methods.[30,33,34] ^{1}H NMR spectra were taken using a Varian-Mercury 200 NMR spectrometer. The chemical shifts for NMR spectra were ascribed in relation to the external tetramethylsilane (TMS) standard. IR spectra were recorded using a Perkin-Elmer 1600 FT-IR spectrometer using KBr pellets. Elemental analyses were carried out using a Hewlett-Packard 185 analyzer. Metal contents in complexes were determined using a Unicam 929 AAS spectrometer. Mass spectra of the compounds were obtained on a Varian MAT 711 spectrometer. pH values were measured on a WTW 537 pH meter. 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, μ_{eff}, per metal atom were calculated based on the expression $\mu_{eff} = 2.84\sqrt{\chi_M T}$ B.M., where χ_M is the molar susceptibility.

Preperation of Ligand Complexes

[Fe/Cr(salen or salophen)]$_2$O was prepared by adding concentrated ammonia solution (26% w/w) to a stirred hot EtOH solution of [Fe/Cr(salen or salophen)Cl], until it became alkaline.[30,33,34]

Synthesis of Chloroisonitrozoacetophenone

Dry Cl$_2$ gas was bubbled through a suspension of isonitrosoacetophenone (3.58 g, 24 mmol) in chloroform under sunlight for 30 min, and the suspended material dissolved and turned yellow. Bubbling of the Cl_2 gas was sustained for a further 1 h under ultraviolet (UV) radiation (254 nm), and the temperature of the mixture reached 40 °C. When the mixture was cooled to room temperature, white crystals formed. Excess Cl_2 was expelled in vacuum and then filtered, and the solid was washed with diethylether. FT-IR (cm^{-1}) 3256 (OH), 1656 (C=N c), 1696 (C=O), 956 (N-O). ^1H NMR (DMSO-d_6) δ 11.50 (s, broad, 1H), 7.94–7.91 (d, 2H), 7.66–7.63 (dd, 1H), 7.55–7.52 (dd, H). LC-MS (ESI^+) $m/z = 183$ [100%, chloroisonitrozoacetophenone]. Molecular peaks of the cations are observed with the same isotope distribution as the theoretical ones.

Synthesis of 2,4,6-Tris(4-(2-phenyl-2-keto-hydroxyiminoyloxy) benzimino)-1,3,5-triazine L (III)

Choloroisonitrosoacetophenone (3 equiv., 27.54 g, 150 mmol) in 50 mL acetone was added to a suspension of 2,4,6-tris(4-hydroxybenzimino)-1,3,5-triazine (21.92 g, 50 mmol) and triethylamine (8.93 g, 150 mmol) in 40 mL of acetone. The product obtained was collected by filtration and dried in vacuo. The obtained mixture was purified using colon chromotography and using 1:4 ethylacetate/n-hexane mixture as eluent. The product was recrystallized from a mixture of methanol and water (1:2). FT-IR (cm^{-1}) 3258 (OH), 2886 (CHar), 1576 (triazine C=N a), 1622 (CH=N b), 1653 (CH=N c), 1694 (C=O), 958 (N-O), 1372 (C–O–C). ^1H NMR (DMSO-d_6) δ 11.50 (s, broad, 3H), δ 9.79 (s, 3H), 7.94–7.91 (d, 6H), 7.76–7.73 (d, 6H), 7.66–7.63 (dd, 3H), 7.55–7.52 (dd, 6H), 6.93–6.90 (d, 6H). LC-MS (ESI^+) m/z: 879 [100%, III]. Molecular peaks of the cations are observed with the same isotope distribution as the theoretical ones. Some physical properties and yield for **III** are given in Table 1.

Preparation of L(FeSalen)$_3$ (IV), L(FeSalophen)$_3$ (V), L(CrSalen)$_3$ (VI), and L(CrSalophen)$_3$ (VII) Complexes

A solution of **L** (1.76 g, 2.0 mmol) and [Fe/Cr(salen or salophen)]$_2$O (3.0 mmol) in 100 mL of absolute methanol was refluxed for 3 h. The mixture was allowed to cool to room temperature. Then the mixture was filtered and dried in vacuum. LC-MS (ESI^+) m/z: 1849 [100%, IV], m/z: 1993 [100%, V], m/z: 1837 [100%, VI], and m/z: 1981 [100%, VII]. Molecular peaks of the cations are observed with the same isotope distribution as the theoretical ones. Some physical properties and yields for **IV, V, VI** and **VII** are given in Table 1. FT-IR (cm^{-1}) for complex **IV**: 2884 (CHar), 1575 (triazine C=N a), 1625 (CH=N b), 1658 (C=N c), 1540 (C=N d), 1709 (C=O), 1008 (N-O), 1380 (C–O–C), 540 (M-N), 475 (M-O); for complex **V**: 2885 (CHar), 1568 (triazine C=N a), 1622 (CH=N b), 1654 (C=N c), 1538 (C=N d), 1708 (C=O), 1004 (N-O), 1378 (C–O–C), 542 (M-N), 477 (M-O); for complex **VI**: 2888 (CHar), 1572 (triazine C=N a), 1623 (CH=N b), 1656 (C=N c), 1542 (C=N d), 1704 (C=O), 1005 (N-O), 1381 (C–O–C), 543 (M-N), 474 (M-O); and for complex **VII**: 2882 (CHar), 1574 (triazine C=N a), 1622 (CH=N b), 1655 (C=N c), 1544 (C=N d), 1706 (C=O), 1006 (N-O), 1382 (C–O–C), 544 (M-N), 478 (M-O).

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