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# The first example of calix[4]pyrrole functionalized *vic*-dioxime ligand: Synthesis, characterization, spectroscopic studies and redox properties of the mononuclear transition metal complexes

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## 1. Introduction

The coordination chemistry of vic-dioxime compounds has been becoming increasingly interesting and has been studied from a variety of perspectives by many different research groups [1–7]. Because of the potential applications of metal complexes of these compounds, they have been shown to be useful in many research areas such as medicine [8], catalysis [9], electrooptical sensors [10] and trace metal analysis [11]. Calix[4]pyrroles and their derivatives have recently become the subject of intensive research aimed at the development of novel ligands [12], because, the compounds are capable of selective binding of anionic [13], cationic [14] and neutral moeities [15] both in the solution and the solid states. In the research of second generation calixpyrrole [16], Sessler and co-workers employed three fluorescent calix[4]pyrroles prepared by attaching commercial dyes to 3-aminophenylcalix[4]pyrrole. The unique character of these compounds is that they support additional binding sites that can dramatically increase the binding capability to anions. Anion binding is a key process in many chemical and biochemical phenomena, as evidenced by the fact that many substrates of structurally characterized enzymes are anionic in nature [17]. Even though a great number of calix[4]pyrrole derivatives have been synthesized and reviews have been published giving an overview of the synthesis and prop-

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## ABSTRACT

Novel calix[4]pyrrole bearing *vic*-dioxime ligand (**LH**<sub>2</sub>) of the general formula,  $R_1R_2C_2N_2O_2H_2$  (where,  $R_1 = C_6H_5$ - and  $R_2 = C_{39}H_{50}N_5$ -) has been synthesized by the reaction of *anti*-chlorophenylglyoxime with 3-aminophenylcalix[4]pyrrole at room temperature. The mononuclear Cu(II), Ni(II) and Co(II)complexes of this *vic*-dioxime ligand were prepared and their structures were confirmed by elemental analysis, FT-IR, TGA and magnetic susceptibility measurements; the HMBC, DEPT, <sup>1</sup>H and <sup>13</sup>C NMR spectra of the LH<sub>2</sub> ligand were also reported. The electrochemical property of the complexes was investigated in DMSO by cyclic voltammetry at 200 mV s<sup>-1</sup> scan rate. The cyclic voltammetric measurements clearly indicated that Co(LH)<sub>2</sub>·2H<sub>2</sub>O complex differs from the Ni(LH)<sub>2</sub> and Cu(LH)<sub>2</sub> complexes upon the exhibition of quasi-reversible one-electron transfer reduction process in the negative region instead of an irreversible process.

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erties of calix[4]pyrrole derivatives, there are no reports cited in the chemical literature including structural analyses of calix[4]pyrrole functionalized vic-dioxime ligands and their transition metal complexes. For that reason, we think that synthesis of vic-dioxime compounds that functionalized with calix[4]pyrrole derivative can generate new materials with interesting properties due to their above mentioned specific complexation abilities with different anions, metal cations, and neutral compounds.

The aim of the present study is to obtain and characterize calix[4]pyrrole bearing *vic*-dioxime ligand and to prepare its nickel(II), cobalt(II) and copper(II) complexes. The newly prepared ligand was characterized fully in order to confirm the proposed structure using NMR (<sup>1</sup>H, <sup>13</sup>C, HMBC, DEPT), IR and elemental analysis. The composition of Co(II), Ni(II) and Cu(II) complexes have been identified by elemental analysis, FT-IR and magnetic susceptibility measurements. Thermal decomposition of related complexes was investigated by thermogravimetric analysis (TGA) and the cyclic voltammetric measurements of the complexes have also been conducted to investigate electrochemical behavior of their reduced or oxidized species in nonaqueous solution.

### 2. Experimental

### 2.1. Materials and measurements

All chemicals were of reagent grade quality and were used without purification; *anti*-chlorophenylglyoxime (1) [18] and



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3-aminophenylcalix[4]pyrrole (2) [16] were prepared according to a described procedures. All solvents were of reagent grade and were purified according to standard procedures. Elemental analyses (C, H, N) were determined using a LECO-932 CHNSO model analyzer. <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on a Bruker GmbH Dpx-400 MHz High Performance Digital FT-NMR spectrometer with DMSO-d<sub>6</sub> as the solvent with Me<sub>4</sub>Si as internal reference. The IR spectra of solid samples (KBr tablets) were recorded in the range from 200 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> on a Perkin–Elmer Model 1605 FT-IR spectrophotometer. Magnetic moments of the complexes were measured using a Sherwood Scientific Model MX1 Gouy magnetic susceptibility balance at room temperature using Hg[Co(SCN)<sub>4</sub>] as calibrant; diamagnetic corrections were calculated from Pascal's constants. Melting points were determined using an electrothermal apparatus and were uncorrected. Thermal measurements were carried out on a Setaram Labsvs TG-DTA Instruments thermal analysis system in dinitrogen atmospheres. applying a heating rate of 10 °C min<sup>-1</sup> in a temperature range of 25-1073 °C All the electrochemical experiments were performed using a CH Instruments electrochemical analyzer (model 600C series) equipped with BAS C3 cell stand. Working electrode was a bare or modified glassy carbon disk (BAS) with a geometric area of  $0.027 \text{ cm}^2$ . The reference electrode was a Ag/AgCl/KCl<sub>(sat.)</sub> and the counter electrode was a Pt wire.

### 2.2. Synthesis of the ligand $[LH_2]$ (3)

A mixture of amino-modified calixpyrrole (2) (0.1 mmol, 59 mg), anti-chlorophenylglyoxime (0.1 mmol, 19 mg), Et<sub>2</sub>O (15 mL) and  $Et_3N(0.1 \text{ mL})$  was stirred overnight at room temperature. Then the solution was washed three times with saturated NaHCO<sub>3</sub>  $(3 \times 15 \text{ mL})$ . The organic phase was collected, dried with Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed under reduced pressure and recrystallized from warm ethanol, giving bright yellow needles. Yield: 25%; m.p.: 168 °C, Elemental analysis Anal. Calc.: C, 75.10; H, 7.59; N, 13.05. Found: C, 74.95; H, 7.47; N, 12.96%. IR (KBr) v<sub>max</sub>/ cm<sup>-1</sup>: 3415 (N-H), 3368 (O-H), 3022-3059 (C-HAr), 1610 (C=N), 978 (N–O), <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>, ppm): 0.45–0.56 (m, 18H, CH<sub>3</sub>), 1.31-1.69 (m, 15H, CH<sub>2</sub>), 2.01 (s, 3H, CH<sub>3</sub>), 5.63-6.09 (br m, 8H, pyr-CH), 6.80-7.80 (br m, 9H, Ar-CH), 8.24 (br s, 2H, NH), 8.55 (br s, 2H, NH), 10.17 (s, 1H, N-OH), 11.05 (s, 1H, N-OH). <sup>13</sup>C NMR (400 MHz, DMSO-d<sub>6</sub>, ppm): 7.65, 8.32, 8.74, 8.89, 8.93, 9.35, 9.42, 27.30, 28.90, 29.20, 29.70, 30.66, 31.79, 42.59, 43.31, 43.38, 44.78, 103.11, 104.65, 104.72, 105.18, 112.13,



Fig. 1. (a) <sup>13</sup>C NMR and (b) DEPT spectra of ligand.

113.71, 115.54, 127.92, 128.96, 129.90, 130.42, 135.92, 136.39, 138.56, 147.78, 149.61.

then filtered and washed with water, ethanol and ether and dried *in vacuo*.

# 2.3. Preparation of mononuclear complexes, [Ni(LH)<sub>2</sub>] (**4**), [Cu(LH)<sub>2</sub>](**5**), [Co(LH)<sub>2</sub>·2H<sub>2</sub>O](**6**)

A solution of 0.05 mmol metal salt [NiCl<sub>2</sub>· $GH_2O$  (0.0118 g), CuCl<sub>2</sub>· $2H_2O$  (0.0084 g) CoCl<sub>2</sub>· $GH_2O$  (0.0116 g)] in 5 mL of hot ethanol, was added dropwise to a stirred solution of 3 (0.1 mmol, 75 mg) in 5 mL of ethanol. The pH of the reaction mixture was around 3.5–4.0 and was then adjusted to 5.5–6.0 by adding 1% NaOH solution. The mixture was stirred on a water bath at 60 °C for 2 h in order to complete the precipitation. The precipitates were 2.3.1. Data for (4)

Yield: 58%, m.p.: >200 °C; Elemental analysis *Anal.* Calc.: C, 72.37; H, 7.19; N, 12.58. Found: C, 72.25; H, 7.08; N, 12.45%. IR (KBr)  $v_{max}/cm^{-1}$ : 3318 (N–H), 3060–3010 (C–HAr), 1725 (O–H···O), 1595 (C=N), 982 (N–O), <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>, ppm): 0.24–0.54 (m, 36H, CH<sub>3</sub>), 1.05–1.86 (m, 36H, CH<sub>2</sub> + CH<sub>3</sub>), 5.44–5.88 (br m, 16H, pyr-CH), 6.04–7.35 (br m, 18H, Ar-CH), 8.80 (br s, 4 , NH), 9.22 (br s, 4H, NH), 15.93 (s, 2H, O–H···O), MS(ES-MS), *m/z*: 1559 [M+H]<sup>+</sup>,  $\mu_{eff}$ : diamagnetic.



Fig. 3. The <sup>1</sup>H NMR spectrum of the nickel(II) complex (2) in DMSO-d<sub>6</sub> at 25 °C (400 MHz).

### 2.3.2. Data for (5)

Yield: (52%); m.p.: >200 °C, Elemental analysis *Anal.* Calc.: C, 72.15; H, 7.16; N, 12.54. Found: C, 71.95; H, 7.07; N, 12.47%. IR (KBr)  $v_{\text{max}}/\text{cm}^{-1}$ : 3373 (N–H), 3060–3010 (C–HAr), 1740 (O–H···O), 1580 (C=N), 985 (N–O).  $\mu_{\text{eff}}$ : 1.59.

### 2.3.3. Data for (6)

Yield: (46%); m.p.: >200 °C, Elemental analysis *Anal.* Calc.: C, 70.73; H, 7.27; N, 12.29. Found: C, 70.67; H, 7.20; N, 12.37%. IR (KBr)  $v_{max}/cm^{-1}$ : 3435 (H<sub>2</sub>O), 3370 (N–H), 3067–3017 (C–HAr), 1740 (O–H…O), 1587 (C=N), 983 (N–O).  $\mu_{eff}$ : 2.14.

### 3. Results and discussion

### 3.1. Synthesis and characterization of LH<sub>2</sub> ligand and its complexes

3-Aminophenylcalix[4]pyrrole was reacted with *anti*-chlorophenylglyoxime in diethyl ether to yield the desired *vic*-dioxime ligand and which crystallized in warm ethanol for further purification. Mononuclear Ni(II), Cu(II) and Co(II) complexes (**4**– **6**) were synthesized in ethanol by reacting NiCl<sub>2</sub>·6H<sub>2</sub>O, CuCl<sub>2</sub>·2H<sub>2</sub>O and CoCl<sub>2</sub>·6H<sub>2</sub>O with new ligand (**3**) in the presence of NaOH. The routes employed in the synthesis of the *vic*-dioxime ligand and its nickel(II), cobalt(II) and copper(II) complexes are outlined in Scheme 1.

The structural formula of **3** was deduced from FT-IR, NMR (<sup>1</sup>H, <sup>13</sup>C, HMBC, DEPT) and elemental analysis data. In the IR spectrum of **3**, the strong v(NH), v(OH), v(C=N) and v(NO) characteristic stretching vibrations bands were observed at 3415, 3368, 1610 and 978 cm<sup>-1</sup>, respectively. The IR spectra of complexes showed a weak deformation band at 1725–1740 cm<sup>-1</sup>, indicative of intramolecular hydrogen bonded bending vibrations (O–H···O) associated with the square-planar *vic*-dioxime complexes. As v(NO)stretching vibrations of LH<sub>2</sub> shift to slightly higher frequencies, the v(C=N) stretching vibrations shift to slightly lower frequencies, and it indicates the coordination of the azomethine nitrogen to the metal center [19]. Deveci et al. [3] and Yuksel et al. [5] showed that *vic*-dioxime ligand form square-planar Ni(II) and Cu(II) complexes in which the ligands chelate *via* nitrogen and oxygen donor atoms. In the IR spectrum of the Co(II) complex, coordinated H<sub>2</sub>O molecules were identified by a strong broad OH absorption around 3435 cm<sup>-1</sup> [19]. The FT-IR spectra of all of the compounds exhibited two medium intensity absorption signals at between 3000 cm<sup>-1</sup> and 3100 cm<sup>-1</sup> result from phenyl group. Additionally, the strong and very strong CH<sub>2</sub> stretching bands were observed at 2944 cm<sup>-1</sup> and 2824 cm<sup>-1</sup>.

According to magnetic susceptibility measurements, while complex **5** and **6** were paramagnetic with a magnetic susceptibility values 1.59 and 2.14 BM, respectively, complex **4** was diamagnetic, consistent with the formation of a low spin d<sup>8</sup> square planar geometry. On the basis of the magnetic and spectral evidence an octahedral geometry for Co(II) complex was proposed and it is consistent with the formation of octahedral structure with water molecules occupying axial positions. The suggested structures of the complexes are shown in Scheme 1.

When the <sup>1</sup>H NMR spectra of the ligand in DMSO was examined, peaks corresponding to N–OH protons (11.46 ppm and 10.59 ppm (s, 2H)) was observed downfield. This assignment was further substantiated by disappearance of the corresponding OH peaks when ligand underwent deuterium exchange. These values are in accord with the previously reported oxime derivatives [3,5,19,20]. This compound showed various doublets and quartet (not resolvable) at 6.80 and 7.80 ppm for aromatic protons. Two broad singlets were observed one at 8.24 ppm and the other at 8.55 ppm for pyrrole N–H groups. In addition, the two quaternary hydroxyimino carbon atoms (C=N–O), appeared at 149.61 and 147.78 ppm, which was confirmed by their disappearence in DEPT NMR spectrum (Fig. 1b). Observation of dioxime carbons in <sup>13</sup>C NMR spectra at two different frequencies indicates that the *vic*-dioxime ligands have the *anti*-structure [19].

By HMBC spectrum, specific assignments of protons and carbons are made as follows. HMBC of the ligand shows correlations that support the proposed structure. HMBC spectrum shows the  $\delta$  10.59 proton corresponds to  $\delta$  147.78 carbon, and  $\delta$  11.46 proton corresponds to  $\delta$  149.61 carbons (Fig. 2). Other correlations are also in accordance with the expected structure.



Fig. 4. Mass spectrum of the nickel(II) complex (2).

Although the solubility of the Ni(II) complex in organic solvents was limited, we was able to obtain <sup>1</sup>H NMR spectra for this complex (Fig. 3). The <sup>1</sup>H NMR spectra of the Ni(II) complex (**2**) was characterized by the existence of intra-molecular D<sub>2</sub>O-exchangeable H-bridge (O-H···O) protons which were observed by a new signal at low field,  $\delta$  = 15.93 ppm. The other protons observed for complex are very similar to those which are found for ligand.



Fig. 5. Thermal curves of the (a)  $Ni(LH)_2$  (b)  $Cu(LH)_2$  (c)  $Co(LH)_2 \cdot 2H_2O$ .

The mass spectrum of this compound (Fig. 4) was measured using the ES/MS technique and the Ni(II) complex (**2**) displayed a significant peak corresponding to  $[M]^+$  at m/z = 1559.74. The elemental analysis data of this compound were also in accordance with the proposed formulation.

The thermal behaviour of Cu(II), Ni(II) and Co(II) complexes have been studied by DTA and TG. Thermal curve of the complexes are seen in Fig. 5. In the TG curve of the Ni(II), Cu(II) and Co(II) complexes (calc: 95.21%, exp: 94.18% for [Ni(LH)<sub>2</sub>], calc: 94.91%, exp: 94.01% for [Cu(LH)<sub>2</sub>] and (calc: 95.30%, exp: 95.04% for [Co(LH)<sub>2</sub>·2H<sub>2</sub>O]) weight loss was observed in the range 25– 1100 °C. It was concluded from TG studies that all the Ni(II), Cu(II) and Co(II) complexes examined were converted to the corresponding oxides [ref verelim]. The final decomposition products were identified by IR spectroscopy with corresponding spectra obtained



**Fig. 6.** Cyclic voltammograms of 1 mM (a) nickel(II) complex (**4**) (b) copper(II) (**5**) complex (c) cobalt(II) complex (**6**) of **3** in DMSO containing 0.1 M TBATFB as supporting electrolyte at the glassy carbon electrode versus Ag/AgCl/KCl<sub>(sat.)</sub>. Scan rate is 200 mV s.

under the same conditions as the pure oxides. Its thermal decomposition reveals that the Cu(II) and Ni(II) complexes do not contain the adsorbed or coordinated water or solvent molecules, which are consistent with the analytical and spectroscopic data. [19]. The presence of coordinated water molecules in Co(II) complex further corroborates the assumption made on the basis of the infrared spectral studies [20].

## 3.2. Electrochemical behavior of complexes of LH<sub>2</sub> ligand

The ability of oxime-aminophenylcalix[4]pyrrole ligands to stabilize reduced and oxidized forms of metal ions has sparked interest in their role in bioinorganic systems [21]. Hence, for a better understanding of their properties, the investigation of redox behavior has a vital importance. Although various oximes and their metal and non-metal compounds have been studied extensively and practical applications in many important chemical processes have been found, electrochemistry of vic-dioximes is scarce. Therefore, the redox properties of the complexes were investigated in a solution of 1 mM (4-6) in 0.1 M TBATFB in DMSO versus Ag/AgCl/ KCl<sub>(sat.)</sub> reference electrode using cyclic voltammetry (CV) with a scan rate of 200 mV s<sup>-1</sup> between 0 V and -1.6 V at the glassy carbon electrode. The cyclic voltammograms for 4 and 5 display almost similar redox potentials associated with M(II)/M(I) (M = Ni, Cu). In both complexes irreversible reduction wave was observed at  $E_c = -743$  mV for **4** and  $E_{c1} = -638$  mV for **5** (Fig. 6). A second reversible wave at  $E_{c2}$  = -800 mV (with an anodic counterpart at  $E_{a2} = -640 \text{ mV}$ ) was also observed in the voltammogram which may be ascribed to the Cu(I)/Cu(0) reduction due to the less stable Cu(I) ions in solution. The cyclic voltammogram of Co(II) complex is shown in Fig. 6c, the reduction process refers to the metal-based  $Co^{II}/Co^{I}$  couples. The anodic,  $E_{pa}$  and cathodic,  $E_{pc}$ , potentials appear at -570 and -750 mV, respectively, at a scan rate of 200 mV s. On the basis of comparison with ferrocenium/ferrocene couple ( $\Delta E_{\rm P}$ , 70 mV,  $I_{\rm pa}/I_{\rm pc}$ , **1** at 200 mV s), the reduction process for cobalt complex ( $\Delta E_P$ , 180 mV,  $I_{pa}/I_{pc}$ , 0.53 at 200 mV s) is appropriately described as quasi-reversible [22].

Although the vic-dioximes are electroactive [23,24], the lack of the oxidation or reduction signals of the oxime moieties either in LH<sub>2</sub> ligand or its complexes is probably due to the increasing polarization effect by means of calixpyrrole groups attached to the oxime moieties. Similar behavior was observed for some phthalocyanine complexes having four sulfonates groups on the phthalocyanine moiety [25]. Although vic-dioxime ligands have been shown to be good ligands for the stabilization of uncommon high oxidation states of transition metals, in this study we could not see any oxidation peaks for  $Cu^{III}$ ,  $Ni^{III}$ ,  $Ni^{IV}$  and  $Co^{III}$  ions, and it can be taken as indication that, none of the complexes in this work, have the ability of stabilize these ions.

### 4. Conclusions

In this work, we have synthesized and characterized a new vicdioxime ligand and three new Ni(II). Co(II) and Cu(II) complexes. Also, we have investigated redox behaviors of Ni(II), Co(II) and Cu(II) complexes of ligand by cyclic voltammetry at glassy carbon electrode. Electrochemical data have shown that nickel and copper complexes exhibit almost similar electrochemical behavior, with the irreversible reduction processes based on metal cations, while the Co(II) complex displays guasi-reversible one-electron transfer reduction process in the cathodic region based on metal.

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