



Synthesis, characterization and spectroscopy studying of some metal complexes of a new Schiff base ligand; X-ray crystal structure, NMR and IR investigation of a new dodecahedron Cd(II) complex

Reza Golbedaghi ^{a,*}, Majid Rezaeivala ^b, Maryam Khalili ^a, Behrouz Notash ^c, Javad Karimi ^d

^a Chemistry Department, Payame Noor University, 19395-4697, Tehran, Iran

^b Department of Chemical Engineering, Hamedan University of Technology, Hamedan, 65155, Iran

^c Department of Chemistry, Shahid Beheshti University, G.C., Evin, Tehran, 1983963113, Iran

^d Department of Chemistry, Ferdowsi University of Mashhad, Iran

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ABSTRACT

Some new $[\text{Cd}(\text{H}_2\text{L}^1)(\text{NO}_3)]\text{ClO}_4$ (**1**), $[\text{Mn}(\text{H}_2\text{L}^1)](\text{ClO}_4)_2$ (**2**), $[\text{Ni}(\text{H}_2\text{L}^1)](\text{ClO}_4)_2$ (**3**) and $[\text{Cu}(\text{H}_2\text{L}^1)](\text{ClO}_4)_2$ (**4**) complexes were prepared by the reaction of a Schiff base ligand and M (II) metal ions in equimolar ratios (M = Cd, Mn, Ni and Cu). The ligand H_2L^1 was synthesized by reaction of 2-[2-(3-formyl phenoxy)propoxy]benzaldehyde and ethanol amine and characterized by IR, ^1H , ^{13}C NMR spectroscopy and elemental analysis. The synthesized complexes were characterized with IR and elemental analysis in all cases and ^1H , ^{13}C NMR, and X-ray in the case of Cd(II) complex. The X-ray crystal structure of compound **1** showed that all nitrogen and oxygen atoms of Schiff base ligand (N_2O_4) and a molecule of nitrate with two donor oxygen atom have been coordinated to the metal ion and the Cd(II) ion is in an eight-coordinate environment that is best described as a distorted dodecahedron geometry.

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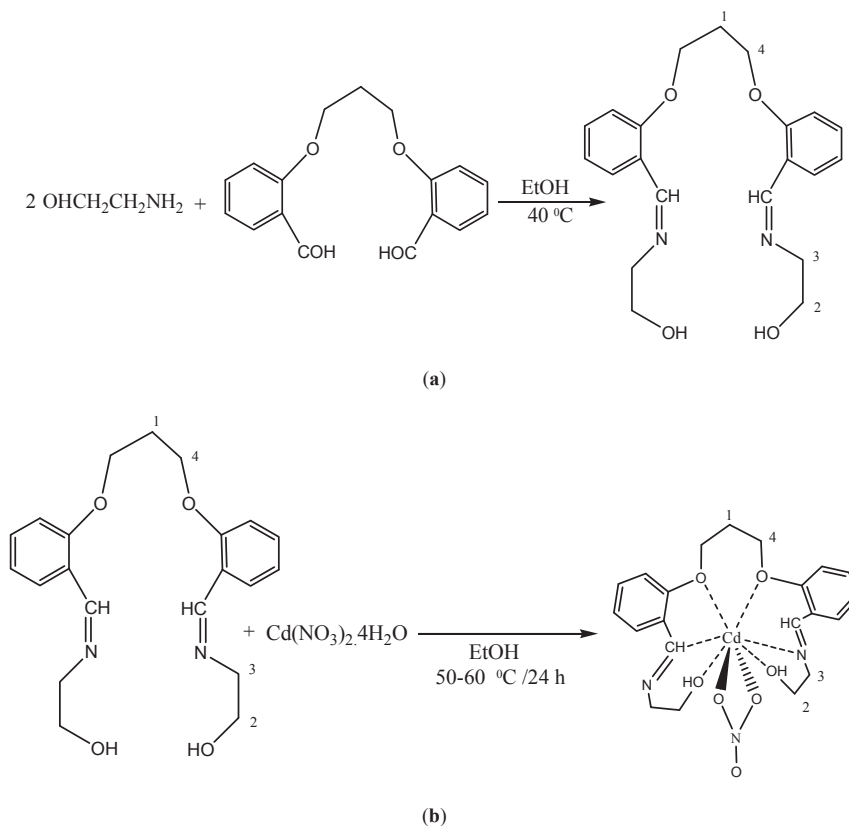
1. Introduction

We are interested in the polydentate coordination chemistry of different metal ions and we have a number of publications of macrocyclic [1,2] and macroacyclic [3–7] Schiff base complexes. Recently Schiff base compounds have been extensively employed as ligands for mono- or multinuclear macrocyclic or macroacyclic metal complexes, which are applicable to biomimetic catalysts, transporter agents, magnetic or electronic functional materials, building materials in nano-space construction and so on [8–10]. In addition, during the last few decades there has been great interest in synthesis of new Schiff base ligands and their coordination chemistry. This interest comes from the fact that Schiff base ligands and their complexes show remarkable biological activities including antibacterial, tumor, fungal, microbial, oxidant, cancer, diabetic, analgesic, and anti-HIV properties [11–17]. Herein, we

continue the study of the various complexes which show characteristic architecture and properties depending on the conformation of the Schiff base ligand. In addition, we are interested in the polydentate coordination chemistry of different metal ions specially Cd(II), Mn(II), Ni(II) and Cu(II) complexes and we have studied their complexes containing multidentate ligands [1–7]. In the current work, we report the synthesis and characterization of Cd(II), Mn(II), Ni(II) and Cu(II) complexes with a new macroacyclic Schiff base ligand derived from the 2-[2-(3-formyl phenoxy)propoxy] benzaldehyde. After synthesis the Schiff base ligand and preparation some new complexes, we tried to get appropriate crystals of them. Finally, the suitable crystals of Cd(II) complex were prepared and we succeeded to take its X-ray analysis data. The X-ray crystal structure shows distorted dodecahedron geometry for Cd(II) metal ion.

* Corresponding author.

E-mail address: golbedaghi82@gmail.com (R. Golbedaghi).



Scheme 1. Syntheses of the Schiff base ligand H₂L₁ (a) and related complex **1** and NMR numbering (b).

2. Experimental

2.1. General information

Hydrated metal salts and ethanol amine were obtained from Aldrich and were used without further purification. 2-[2-(3-formylphenoxy)propoxy]benzaldehyde (H₂L₁) was prepared via literature method [18]. IR and NMR spectra were measured on a Perkin Elmer FT-IRGX and a Bruker DPX 300 spectrometer, respectively.

2.2. X-ray crystal structure determination

Single crystal X-ray data on complex **1** were collected at 120 K on a Bruker SMART APEX CCD diffractometer using graphite monochromated Mo K_α radiation (λ = 0.71073 Å). The linear absorption coefficients, scattering factors for the atoms, and the anomalous dispersion corrections were taken from the International Tables for X-ray Crystallography [19]. The data integration and reduction were carried out with SAINT [20] software. Empirical absorption correction was applied to the collected reflections with SADABS [21] and the space group was determined using XPREP [22]. The structure was solved by the direct methods using SHELXTL-97 [23] and refined on F² by full-matrix least-squares using the SHELXL-97 program [24] package. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms attached to carbon atoms were positioned geometrically and treated as riding atoms using SHELXL default parameters.

2.3. Synthesis

2.3.1. Preparation of aldehydes

1, 3-dibromo propane (0.1 mol) and NaOH (0.2 mol) were mixed

and heated under reflux in 500 ml equals ethanol (300 ml H₂O and 200 ml EtOH). Salsylaldehyde (0.2 mol) in 30 ml EtOH was added and heated under reflux for 4 days. After cooling the resulting yellow crystals were filtered, washed and recrystallized [18].

2.3.2. Preparation of ligand H₂L₁

Aldehyde (0.284 g, 1 mmol) and ethanol amine (0.122 g, 2 mmol) were mixed and heated in the 40 °C for 30 min in EtOH (30 ml). The solution was filtered and the filtrate was reduced to ca 10 cm³. Yellow crystalline compound was obtained washed with cooled ethanol and dried. Yield: 54%. Anal. Calc. for C₂₁H₂₆N₂O₄: C, 67.85; H, 6.84; N, 7.91. Found: C, 67.44; H, 6.79; N, 7.81. IR (KBr, cm⁻¹): 1638 (νC=N Schiff base), 1490 (νC=C), 3365 (νOH). ¹HNMR (DMSO, ppm, 300 MHz): δ 2.36 (m, 2H), 3.71 (t, 4H), 3.82 (t, 4H), 4.25 (t, 4H), 6.99 (m, 2H), 7.40 (m, 2H) 7.96 (m, 2H), 7.99 (m, 2H), 8.75 (s, 2H), ¹³C NMR (DMSO, ppm, 300 MHz): δ 62.48 (C-1), 63.57 (C-2), 65.03 (C-3), 76.73(C-4), 77.05(C-5), 112.20, 121.09, 125.62, 125.74, 12684, 127.40, 132.16 (Aromatic Rings), 158.88 (Schiff base).

2.3.3. Preparation of complex [CdH₂L₁(NO₃)]ClO₄ (**1**)

This complex was prepared by reaction of ligand H₂L₁ and Cd(NO₃)₂·4H₂O. Ligand H₂L₁ (0.370 g, 1 mmol) was solved in 10 ml methanol and the solution was stirred at 30–40 °C. Cd(NO₃)₂·4H₂O (0.308 g, 1 mmol) dissolved in MeOH (5 ml) was subsequently added. The mixture was stirred at 50–60 °C for 24 h, then NaClO₄ (0.28 g, 2 mmol) was added. The solution was filtered and the filtrate was reduced to ca. 10 cm³. A crystalline compound was obtained by slow diffusion of Et₂O vapor into this solution. Yield of the complex [CdH₂L₁(NO₃)]ClO₄ (**1**), 75%. Anal. Calc. for (C₂₁H₂₅CdClN₃O₁₁): C, 39.10; H, 4.10; N, 6.50. Found: C, 38.78; H, 3.89; N, 3.76%. IR (KBr, cm⁻¹): 1682 (νC=N Schiff base), 1456 (νC=C), 3445 (νOH), 1384 (νN–O), 1111 (νClO₄). ¹HNMR (DMSO, ppm,

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