

Synthesis and Characterization of [CrLCl₂]Cl Complexes (where L = S₄ Macrocyclic Ligands): A Photoelectron Spectroscopic Study

KRISHNA KUMAR YADAV¹, ANIL KUMAR¹, MOHD KASHIF AZIZ², RAFAT SABA², ASHISH RAJAK² and SHEKHAR SRIVASTAVA^{2,*}

¹Department of Chemistry, Bipin Bihari P.G. College, Jhansi-284002, India

²Department of Chemistry, University of Allahabad, Allahabad-211002, India

*Corresponding author: E-mail: shekhsri@rediffmail.com

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In this study, five complexes of the type [CrLCl₂]Cl [where L = S₄ macrocyclic ligands] were prepared and analyzed by elemental analysis, molar conductivity, infrared and X-ray photoelectron spectroscopy and their geometry was established as octahedral.

Keywords: Chromium, S₄ macrocyclic ligands, X-ray photoelectron spectroscopy.

INTRODUCTION

The coordination chemistry of macrocyclic ligands and their metal complexes has current interest among chemists and biochemists [1-5]. They have wide applications since they provide stable and selective complexes with many organic and inorganic cations; anions and with neutral organic and biological substrates to form supramolecular compounds with specific properties [6,7]. Schiff base macrocyclic ligands have been proved very useful in inorganic biochemistry material science, catalysis, encapsulation, activation, electron-transport [8,9]; non-linear optical materials [10,11]; biosites modeling [12]; catalysis for many organic reactions [13]; as catalyst in asymmetric synthesis [14] and as luminescence materials [15,16].

Recently asymmetric heptaza Schiff base macrocyclic Mn(II) complexes was synthesized by template [1+1] cyclocondensation of N,N,N',N'-tetrakis(2-aminothyl) propane-1,2-diamine-1,2-diamine with 2,6-diacetylpyridine-1,2-diamine with 2,6-diacetylpyridine [17,18]. Various tetraazamacrocyclic ligands and their metal complexes have been also synthesized e.g. [14]aneN₄ and [12]aneN₄ [19-24]. Keypour *et al.* [25,26] reported a synthesis of a novel series of pentaaza macrocyclic complexes. The hexaazamacrocyclic macrocyclic were also synthesized and found to be versatile since they can coordinate with one or two metal ions [27]. Recently, a hexaazamacrocyclic ligand having four ethyldioxolane pendant groups and their

metal complexes with Zn(II), Cd(II), La(III), Ce(III), Nd(III), Sm(III), Eu(III), Ho(III) have been synthesized [27]. Hexaazamacrocyclic complexes having pendant arm by template condensation of 1,2-phenylenediamine and 1,4-phenylenediamine through formaldehyde in the presence of transition metal have been synthesized [28]. Octaazamacrocyclic were also synthesized and have been proved useful since it can form mono or dinuclear metal complexed due to having large cavity [29]. A variety of macrocyclic complexes were formed by condensation of hydrazine or hydrazone with dicarbonyl compounds [30,31]. Covalently linked azamacrocyclic have been known for several decades but only a modest number of these ligands and their complexes had been synthesized prior to 2000. Since that time, a number of new synthetic methods for their preparation have been discovered, yielding a growing collection of these ligands. During last few decades synthesis and applications of linking azamacrocyclic ligands and their metal complexes have been studied comprehensively [2].

Synthesis and characterization of neutral macrocyclic tetraimine complexes of transition metals; super-structured macrocyclic aldehyde or ketone functionalized tetraimines; superstructured neutral macrocyclic functionalized tetraimines and synthesis and characteristics of mixed *bis*-macrocyclic with neutral and charged macrocyclic components have been studied comprehensively [4]. The transition and post-transition metal ion chemistry of pentadentate dibenzo-substituted macrocyclic ligands having nitrogen, sulphur or oxygen donors have

been well studied [5]. The hydrogen-bonded nanometer scale capsules formation from *C*-methylresorcin[4]arene have arisen a new area of research in macrocyclic chemistry and studied well [3]. These macrocyclic metal complexes may be useful in study electrochemical behaviour, stereochemistry, effect on antiviral, anticancer, antituberculosis, antibacterial and antifungal, catalytic and biophysical properties, sensor devices, fluorescence and chemoluminescence properties.

The sulphur atom as a donor to transition metals is very well studied [32]. The sulphur atom act as sulfide ion (S^{2-}) or as mercaptide ion (RS^-) or as thioether (RSR) [32-34]. Macrocyclic tetrathioethers such as [16]ane S_4 , [16]ane S_4 , [14]ane S_4 and [12]ane S_4 have become more significant in last decade because they give low oxidation state metal sulphur sites in metal catalyzed processes since more diversity in chemical [35-37] and biological systems [38,39]. During last few decades complexes with tetraaza [40-42], dioxotetraaza [43,44], tetraoxotetraaza [45], tetraoxooctaaza [46] and trioxotetraaza were well studied. The amide macrocyclic ligands are significant since their metal complexes work as porphyrin analogues in catalyzing organic oxidations. Comparatively less research work has been done on macrocyclic complexes having N_4S_4 potential donors [47-49]. Recently macrocyclic complexes of Cr(II) [50,51], Cr(III) [52-54] and Cr(V) [55] are reported with N_4 macrocyclic ligands but Cr(III) complexes with S_4 macrocyclic ligands are scarce. This paper deals with synthesis and characterization of $[CrLX_2]X$ complexes with tetradentate ligands with thioethers as donor as *i.e.* [14]ane S_4 ; 13,14-benzo-1,4,8,11-tetrathiacyclopentadecane [14]ane S_4 -diol; 4,5-benzo-3,6,10,13-tetrathiacyclohexadecane-1,8-diol and 6,18-dihydroxy-1,4,8,11-tetrathiacyclohexadecane-1,8-diol.

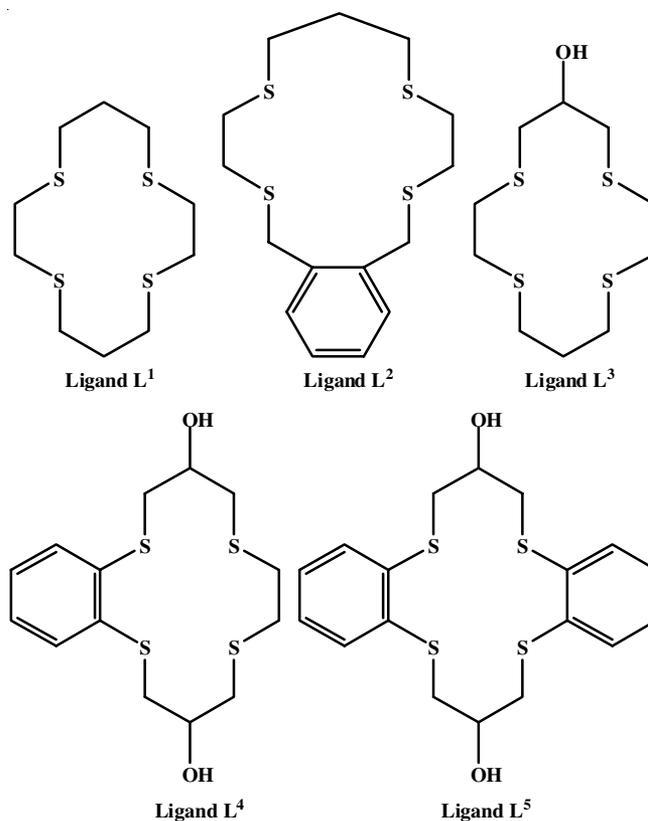
EXPERIMENTAL

All used solvents were purchased from E. Merck (AR grade) or Ranbaxy (AR grade) and used after purification and drying by conventional methods. $CrCl_3 \cdot xH_2O$ were purchased from Aldrich (AR grade). Other chemicals *e.g.* 2-chloroethanol (Loba), thiourea (Aldrich), 3,7-dithianonane-1-dithiol (Aldrich), 1,3-propanedithiol, 1,11-dioxa-4,8-dithiaundecane, anhydrous $MgSO_4$, 1,3-dibromopropane, 1,4,8,11-tetrathiaundecane, 1,3-dichloro-2-propanol, α, α' -dibromo-*o*-xylene, epichlorohydrin, 1,2-ethanedithiol, triethylamine, 1,10-dichloro-4,7-dithiadecane 2,9-diol and 5,6-benzo-1,10-benzene-1,2-dithiol (all procured from BDH) were used after purification and dried by conventional methods.

The melting point of the synthesized complexes were recorded on electrically operated melting point apparatus (M/s Toshniwal) and value recorded are uncorrected. The elemental analysis were conducted at C.D.R.I., Lucknow, India. The molar conductance of the complexes were calculated at room temperature in DMF by Century CC-601 Conductivity bridge. The IR spectra of the complexes and the ligands were recorded on Shimadzu IRAffinity-1S spectrometer at room temperature in KBr or CsI. The X-ray photoelectron spectra *i.e.* XPS were recorded on JEOL-JPS-9030 photoelectron spectrometer. The $AlK\alpha$ X-ray line (1486.6 eV) was used for photo excitation.

Preparation of ligands: All the ligands *viz.*, 1,4,8,11-Tetrathiacyclohexadecane (L^1); 13,14-benzo-1,4,8,11-tetrathia-

cyclopentadecane (L^2); 1,4,8,11-tetrathiacyclohexadecanol (L^3); 4,5-benzo-3,6,10,13-tetrathiacyclohexadecane-1,8-diol (L^4) and dibenzo substituted 6,18-dihydroxy-1,4,8,11-tetrathiacyclohexadecane-1,8-diol (L^5) were synthesized by reported procedure [56,57]. The structure of the synthesized ligands is shown below:



Synthesis of $[CrLX_2]X$ complexes (where $L = L^1$ or L^2 or L^3 or L^4 or L^5): To a solution of $CrCl_3 \cdot xH_2O$ (0.1 mmol) in dry C_2H_5OH was mixed (0.1 mmol) in a above synthesized ligand (L^1 or L^2 or L^3 or L^4 or L^5) in dry C_2H_5OH solution and refluxed for 2-5 h. The solid product was obtained, filtered, washed with dry C_2H_5OH and dried over P_4O_{10} .

RESULTS AND DISCUSSION

All the newly synthesized $[CrLX_2]X$ complexes were yellowish white solid and stable at room temperature. The melting points, yields, elemental analysis and molar conductance data are listed in Table-1. The molar conductance in DMF 100-120 $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$ of these complexes (Table-1) indicated that all these were electrolyte (1:1) [58]. All the prepared ligands exhibited $\nu(C-S)$ band at 1050-1040 cm^{-1} , which have shown one band in all prepared these $[CrLX_2]X$ metal complexes with high intensity towards higher side (1100-1080 cm^{-1}) [59]. The presence of new bands in metal complexes at 380 and 355 cm^{-1} attributed as due to $\nu(Cr-Cl)$ and at 350 cm^{-1} as due to $\nu(Cr-S)$ [60].

The $Cr 2p_{1/2, 3/2}$; $Cl 2p$ and $S 2p$ binding energies (eV) data of $[CrCl_3 \cdot xH_2O]$ and $[CrLX_2]X$ where $L = (L^1 \text{ or } L^2 \text{ or } L^3 \text{ or } L^4 \text{ or } L^5)$ are listed in Tables 2 and 3 (Fig. 1). It might be seen that $Cr 2p_{1/2, 3/2}$ photoelectron peaks binding energy value were observed in $CrCl_3 \cdot xH_2O$ at $Cr 2p_{1/2} \approx 585.6 \text{ eV}$ and $Cr 2p_{3/2} =$

TABLE-1
ELEMENTAL ANALYSIS AND MOLAR CONDUCTANCE [CrLX₂]X COMPLEXES (WHERE L¹ TO L⁵)

| Complexes | m.p. (°C) | Yield (%) | Elemental analysis (%): Found (calcd.) | | | | | Molar conductance (ohm ⁻¹ cm ² mol ⁻¹) |
|---------------------------------------|-----------|-----------|--|-----------|-------------|-------------|-------------|--|
| | | | C | H | S | Cr | Cl | |
| [CrL ¹ Cl ₂]Cl | 180 | 75 | 42.4 (42.5) | 4.6 (4.8) | 20.4 (20.6) | 12.2 (12.0) | 24.9 (24.8) | 112 |
| [CrL ² Cl ₂]Cl | 178 | 84 | 44.2 (44.4) | 5.0 (5.2) | 19.6 (19.7) | 10.6 (10.4) | 21.8 (21.4) | 120 |
| [CrL ³ Cl ₂]Cl | 185 | 87 | 42.4 (42.6) | 4.4 (5.2) | 20.4 (20.6) | 14.7 (14.6) | 30.2 (30.0) | 112 |
| [CrL ⁴ Cl ₂]Cl | 192 | 88 | 44.2 (44.4) | 5.2 (5.2) | 19.6 (19.7) | 10.6 (10.4) | 21.8 (21.6) | 114 |
| [CrL ⁵ Cl ₂]Cl | 196 | 86 | 43.2 (43.2) | 5.2 (5.3) | 18.2 (18.4) | 10.0 (10.0) | 20.6 (20.4) | 118 |

576.6 eV and in metal complexes at Cr 2p_{1/2} = 584.6 eV and Cr 2p_{3/2} = 575.6 eV, which is less about ~1 eV than CrCl₃·xH₂O (Fig. 1a-b and Table-2). It suggested that chromium ion had more electron density in [CrLX₂]X complexes than [CrCl₃·xH₂O] due to involvement of metal ion in coordination [61]. Further, S 2p photoelectron peak have shown only one symmetrical peak in [CrLX₂]X complexes at ~BE 167.8 eV while in free ligands at ~166.4 eV, which was lower than in metal complexes, suggestion all four sulphur atoms are coordinated with chromium metal ion (Table-3 and Fig. 1c). The Cl 2p photoelectron peaks in each ligand have shown one single photoelectron peak at ~202.4 eV, while in each metal complex has shown two peaks with intensity ratio 2:1 at 203.8 eV and 202.6 eV which were assigned as inner and outer sphere chloride ion photoelectron peaks respectively. These observations concluded that two chloride ions were coordinated to chromium metal ion and one chloride ion was in outer sphere of each metal complex [61].

Conclusion

On the basis of elemental analysis, molar conductance measurements, infrared and X-ray photoelectron spectral data and the subsequent discussion for the complexes given above, the structure and an octahedral geometry may be proposed

TABLE-2
Cr 2p_{1/2, 3/2} BINDING ENERGIES (eV) IN CrCl₃·xH₂O AND [CrLCl₂]Cl COMPLEXES

| Compound | Cr 2p _{1/2} | Cr 2p _{3/2} |
|---------------------------------------|----------------------|----------------------|
| CrCl ₃ ·xH ₂ O | 585.60 | 576.6 |
| [CrL ¹ Cl ₂]Cl | 584.6 | 575.6 |
| [CrL ² Cl ₂]Cl | 284.6 | 575.6 |
| [CrL ³ Cl ₂]Cl | 284.6 | 575.6 |
| [CrL ⁴ Cl ₂]Cl | 284.6 | 575.6 |
| [CrL ⁵ Cl ₂]Cl | 284.6 | 575.6 |

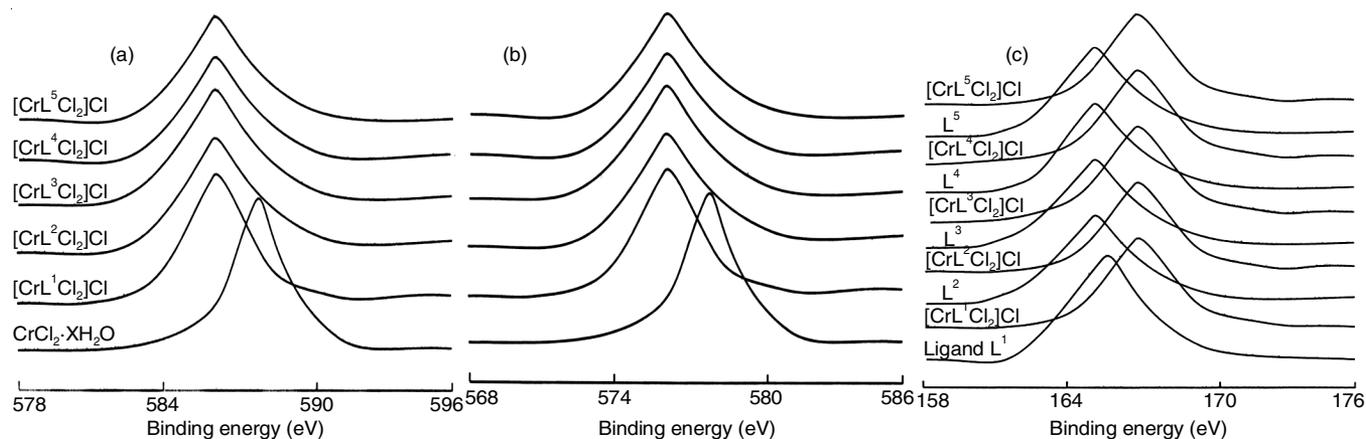


Fig. 1. Cr 2p_{1/2} BE (eV) in CrCl₂·xH₂O (a); Cr 2p_{3/2} BE (eV) in CrCl₃·xH₂O (b) and S 2p BE (eV) in ligands (c) and their [CrLCl₂]Cl complexes

TABLE-3
S2p BINDING ENERGIES (eV) IN LIGAND AND [CrLCl₂]Cl

| Ligand and complex | S2p | Cl 2p |
|---------------------------------------|-------|-------------|
| Ligand L ¹ | 166.4 | 202.4 |
| [CrL ¹ Cl ₂]Cl | 167.8 | 202.6-203.8 |
| Ligand L ² | 166.4 | 202.4 |
| [CrL ² Cl ₂]Cl | 167.8 | 202.6-203.8 |
| Ligand L ³ | 166.4 | 202.4 |
| [CrL ³ Cl ₂]Cl | 167.8 | 202.6-203.8 |
| Ligand L ⁴ | 166.4 | 202.4 |
| [CrL ⁴ Cl ₂]Cl | 167.8 | 202.6-203.8 |
| Ligand L ⁵ | 166.4 | 202.4 |
| [CrL ⁵ Cl ₂]Cl | 167.8 | 202.6-203.8 |

for these complexes as shown in Fig. 2 for all these prepared [CrLCl₂]Cl complexes.

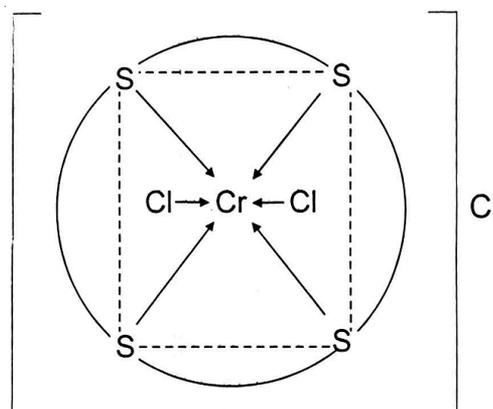


Fig. 2. Proposed octahedral geometry structure of [CrLCl₂]Cl complexes

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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