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Synthesis and Spectral Studies of Ruthenium(II) Metal Complexes with Macrocyclic Ligands: XPS Study

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In this work, bivalent ruthenium macrocyclic complexes of the general formula $[RuCl_2(MacL^{1-2})]$, where $MacL^1$ and $MacL^2$ denote macrocyclic ligands, were synthesised *via* a template condensation approach. The complexes $[\{RuCl_2(MacL^1)\}$ and $\{RuCl_2(MacL^2)\}]$ were obtained through the reaction of 1,3-diaminopropane with oxalic acid and succinic acid, respectively, in the presence of a Ru(II) precursor, maintaining a molar ratio of 2:2:1. The resulting complexes were characterised using a combination of physico-chemical and spectroscopic techniques including molar conductance measurements, elemental analysis, magnetic susceptibility and spectral studies such as UV-visible, FT-IR, ^{1}H NMR and X-ray photoelectron spectroscopy (XPS). Based on the analytical and spectral results, an octahedral geometry was proposed for both ruthenium(II) macrocyclic complexes.

Keywords: Ru(II) complexes, Macrocyclic ligands, Template condensation, XPS study.

INTRODUCTION

Studies on macrocyclic transition metal complexes has expanded rapidly owing to their pivotal roles in coordination chemistry and bioinorganic systems [1]. Studies of these complexes emphasise their significance in a wide range of biological and functional processes including photosynthesis, oxygen transport and catalytic activity, as well as their potential applications as metal extractants and as agents in radiotherapy and medical imaging [2,3]. In particular, the synthesis of aza-macrocyclic complexes has attracted considerable attention due to their strong relevance to biomimetic and catalytic systems, along with their diverse applications in biology and medicine [4-6].

Researchers in the field of coordination chemistry have successfully developed a wide range of macrocyclic ligands like polyazamacrocycles, both unsaturated and saturated crown ethers, polyamines, porphyrins, N_4S_2 donor macrocyclic ligands and Robson-type tetraiminodiphenol [7-9]. There are many transition metal ion function not only in biological systems but also as carriers of enzymes in Schiff base ligand environments, providing models for the active sites of metalloenzymes [10]. The molecular consideration of RNA, DNA and related biomolecules by compounds with N_4 donor macrocyclic ligands has also become a highly intriguing subject [11,12]. The macrocyclic Schiff base ligands are used alternatively systematically due to their thermodynamic factors and kinetic factors, which affect the reactivity patterns of macrocyclic transition metal complexes [13]. Macrocyclic complexes have attracted considerable interest due to their tunable hard–soft donor characteristics, which enable flexible coordination behaviour and significantly influence their biological activity [14]. This adaptable coordination environment facilitates effective interaction with biomolecular targets, contributing to their observed antimicrobial properties against bacteria and fungi. In particular, macrocyclic Schiff base complexes containing azomethine (–C=N–) linkages exhibit enhanced biological activity. The azomethine functionality plays a key role in facilitating metal coordination and modulating electronic distribution within the complex, thereby enhancing its interaction with biological systems. As a result, such complexes demonstrate a wide range of pharmacological effects, including herbicidal, antifungal, antibacterial and antitumor activities [15-17].

The ruthenium coordination compounds have expanded considerably in the recent decades years, due to their wide range

of applications in biological systems, catalysis and analytical methodologies, basically involving Ru(II)/Ru(III) Schiff base ligands and macrocyclic ligand compounds [18,19]. Earlier investigations have firstly addressed the structural and spectroscopic analysis features of *trans*-ruthenium(II)/Ru(III) di(CN^-) compounds stabilised through N_4 donor 3º-amines macrocyclic ligands, while four nitrogen donor macrocyclic complexes of ruthenium(II)/ruthenium(III) were reported with catalytic and spectroscopic behaviour [19-21]. XPS studies of transition metal complexes with macrocyclic ligands have been reported [22]; however, ruthenium(II) macrocyclic complexes remain comparatively less explored. Existing literature highlights the versatility of macrocyclic ligands, particularly their ability to stabilise multiple oxidation states and accommodate diverse coordination geometries [23]. Such ligands are commonly synthesised through template condensation reactions between aliphatic diamines and a bifunctional organic precursor. Several studies have reported that condensation reactions involving diamines and multifunctional organic compounds lead to the formation of stable macrocyclic complexes with interesting structural and biological properties [24].

In particular, 1,3-diaminopropane serves as a flexible nitrogen donor, while oxalic acid and succinic acid act as bifunctional precursors suitable for macrocycle formation. These components enable the construction of ligands capable of effectively coordinating transition metal ions and stabilising octahedral geometries around the metal centre. Although a wide range of macrocyclic transition metal complexes has been reported, systems based on Ru(II) incorporating dicarboxylic acids and aliphatic diamines remain comparatively less explored. Accordingly, the present study focuses on the template synthesis and characterisation of Ru(II) macrocyclic complexes derived from oxalic acid or succinic acid and 1,3-diaminopropane, with the aim of investigating their structural and physico-chemical properties.

EXPERIMENTAL

The reagents employed in these experiments were of AnalaR grade. The precursor complex [$RuCl_2(DMSO)_4$] was synthesised following a reported procedure [25]. All solvents were of analytical grade and were distilled over suitable drying agents prior to use [26].

Characterization: Elemental analysis of carbon, hydrogen and nitrogen was conducted using a Thermo Scientific FLASH 2000 analyzer. Electronic absorption spectral data were obtained with a Varian Cary 100 Bio UV-Vis spectrophotometer and FT-IR spectral data (4000-400 cm^{-1}) of the complexes were obtained using KBr pellets on a Bruker Tensor 27 spectrophotometer. Magnetic susceptibility measurements were measusred at ambient temperature by a Gouy balance calibrated *via* Hg[$Co(NCS)_4$] instrument. Molar conductance was measured on 10^{-3} M in DMF at 24 ºC on a Digisun digital conductivity meter (model DL-909). ^1H NMR spectra were obtained using a JEOL JNM-ECZ500R/S1 spectrometer in DMSO-d_6 and X-ray photoelectron spectral data were recorded on a PHI 5000 VersaProbe III system. Binding energies were correlated to the carbon 1s peak at 284.8 eV to correct for surface charging and peak deconvolution was performed with Origin software.

Synthesis of [$RuCl_2(MacL^{1-2})$] macrocyclic complexes: A methanolic solution of 1,3-diaminopropane (2 mmol) was placed on a magnetic stirrer in a 100 mL round-bottom flask, following the gradual addition of methanolic solutions of the dicarboxylic acids {oxalic acid or succinic acid (2 mmol)} along with the metal precursor [$RuCl_2(DMSO)_4$] (1 mmol). The reaction mixture was stirred magnetically for 5-7 h under reflux. After completion, the solution was reduced to approximately half its volume and left in a desiccator overnight at room temperature. The resulting solid products were filtered, washed successively with ether, methanol and dried in a desiccator. The synthetic route for the formation of the macrocyclic complexes is outlined in **Scheme-I**.

[$RuCl_2(MacL^1)$] complex: Yield:70%; coloir: brown; m.w.: 428.23, Anal. of $C_{10}H_{16}Cl_2N_4O_4Ru$; calcd. (found) %: C: 28.00 (27.53); H: 3.77 (3.43); N: 13.08 (12.68); molar conductivity: 12.5 Ω^{-1} mol^{-1} cm^2.

[$RuCl_2(MacL^2)$] complex: Yield: 73%; colour: black; m.w.: 484.34; Anal. of $C_{14}H_{24}Cl_2N_4O_4Ru$; calcd. (found) %: C: 34.72 (33.58); H: 5.00 (4.30); N: 11.57 (10.22); molar conductivity: 14.5 Ω^{-1} mol^{-1} cm^2.

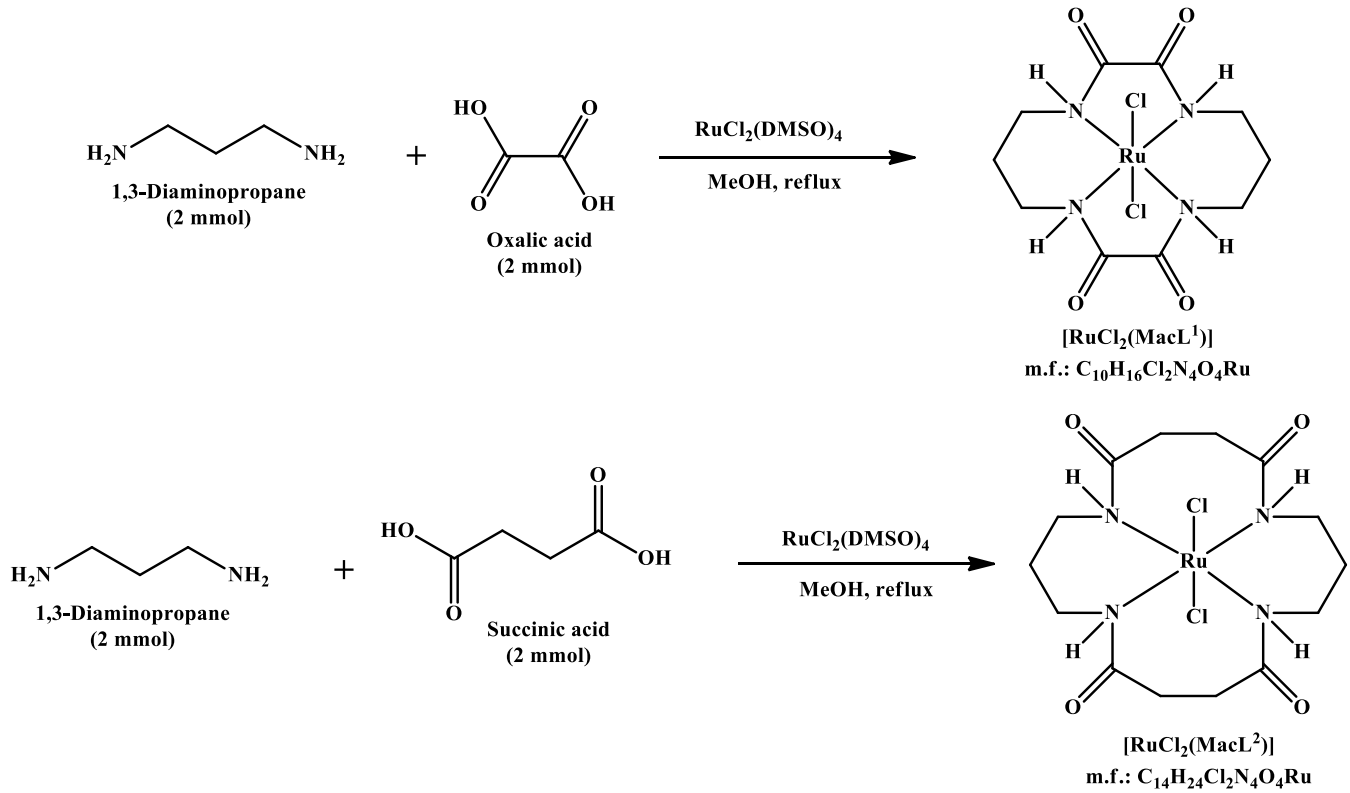
RESULTS AND DISCUSSION

Bivalent Ru(II) macrocyclic complexes were synthesised by reacting [$RuCl_2(DMSO)_4$] with two N_4-donor macrocyclic ligands. The resulting complexes are air-stable and exhibit good solubility in chloroform, DMSO and DMF. Elemental analysis data are consistent with the proposed compositions of the Ru(II) complexes. Both complexes display diamagnetic behaviour, which is characteristic of Ru(II) in a low-spin d^6 electronic configuration.

Infrared spectral studies: The FT–IR spectra of both Ru(II) macrocyclic complexes provide clear evidence for macrocycle formation (Fig. 1). The absence of characteristic –OH stretching bands of the dicarboxylic acids and the disappearance of free $–NH_2$ vibrations from 1,3-diaminopropane indicate successful condensation and coordination. A broad absorption band observed in the range 3265-3260 cm^{-1} is assigned to N–H stretching vibrations [27]. Additional peaks are also consistent with those reported for N_4-donor macrocyclic systems [24,27-29]. The strong absorption band at 1684-1680 cm^{-1} is attributed to C=O stretching vibrations, while the peak at 1580-1576 cm^{-1} is associated with C=N stretching, confirming the formation of the macrocyclic framework [30]. Medium intensity bands in the region 3131-2800 cm^{-1} correspond to C–H stretching vibrations of aliphatic groups [31]. Symmetric and asymmetric bending modes are observed around 1480-1477 cm^{-1} and 1385-1380 cm^{-1}, respectively.

Peaks in the 1290-1272 cm^{-1} region are assigned to N–H bending vibrations of amide functionalities and C–N stretching vibrations appear within the 1340-1000 cm^{-1} range [32]. Peaks observed between 950-650 cm^{-1} are attributed to N–H wagging modes within the coordinated macrocyclic structure. Furthermore, peaks in the 455-453 cm^{-1} region correspond to ν(Ru–N) vibrations, confirming coordination of the ligand to the Ru(II) centre through nitrogen donor atoms [33].

UV-Vis spectral studies: Ruthenium(II) in a low-spin octahedral field (t_{2g}^6 configuration) exhibits a $^1A_{1g}$ ground state,



Scheme-I: Synthetic route of $[RuCl_2(MacL^{1-2})]$ complex derived from N_4 donor macrocyclic ligands

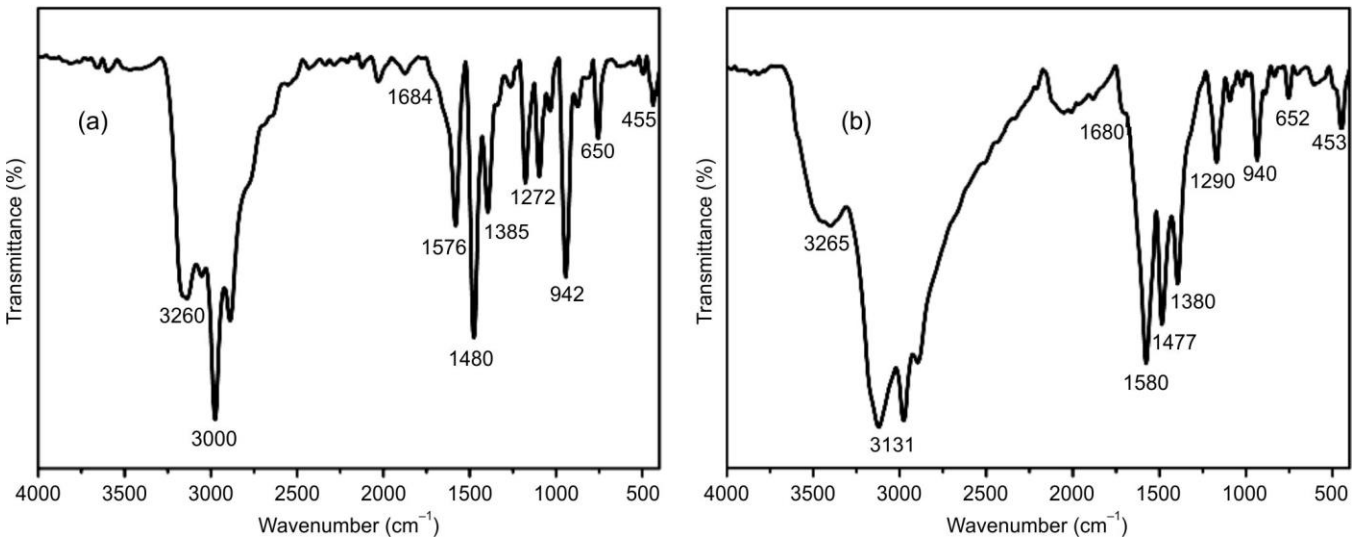


Fig. 1. Infrared spectra of (a) $[RuCl_2(MacL^1)]$ and (b) $[RuCl_2(MacL^2)]$ macrocyclic complex

from which four electronic transitions are possible, including $^1A_{1g} \rightarrow {}^3T_{1g}$, $^1A_{1g} \rightarrow {}^3T_{2g}$, $^1A_{1g} \rightarrow {}^1T_{1g}$, and $^1A_{1g} \rightarrow {}^1T_{2g}$. The UV-Vis spectra of the complexes display two prominent absorption bands in the 200-520 nm region (Fig. 2).

The lower-energy band, observed in the range 440-520 nm, is assigned to the spin-allowed $^1A_{1g} \rightarrow {}^1T_{1g}$ transition, consistent with the molar extinction coefficients reported for similar Ru(II) complexes [34]. The more intense high-energy band in the 250-290 nm region is attributed to a metal-to-ligand charge transfer (MLCT) transition, involving excitation from metal-centred t_{2g} orbitals to ligand π*-orbitals. This assignment aligns with reported spectral features of low-spin octahedral Ru(II) systems [34,35].

NMR spectra: The ^1H NMR spectra of the Ru(II) complexes provide clear evidence for macrocycle formation. The signals corresponding to the $–NH_2$ groups of 1,3-diaminopropane and the –OH groups of the dicarboxylic acids are absent indicating successful condensation and formation of the $[RuCl_2(MacL^{1-2})]$ macrocyclic framework *via* a template approach. Both Ru(II) complexes exhibit a sharp resonance in the δ 8.7-8.1 ppm region, attributed to amide (–CO–NH–) protons, as shown in Fig. 3. Additional signals observed in the

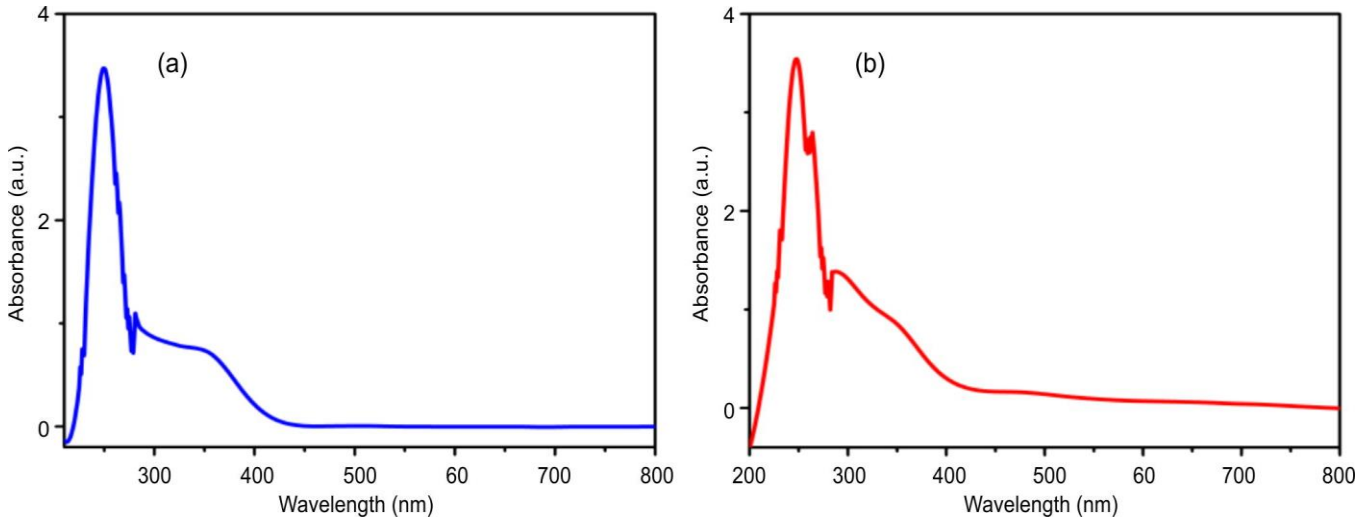


Fig. 2. UV-Vis spectra of (a) [$RuCl_2(MacL^1)$] and (b) [$RuCl_2(MacL^2)$] macrocyclic complex

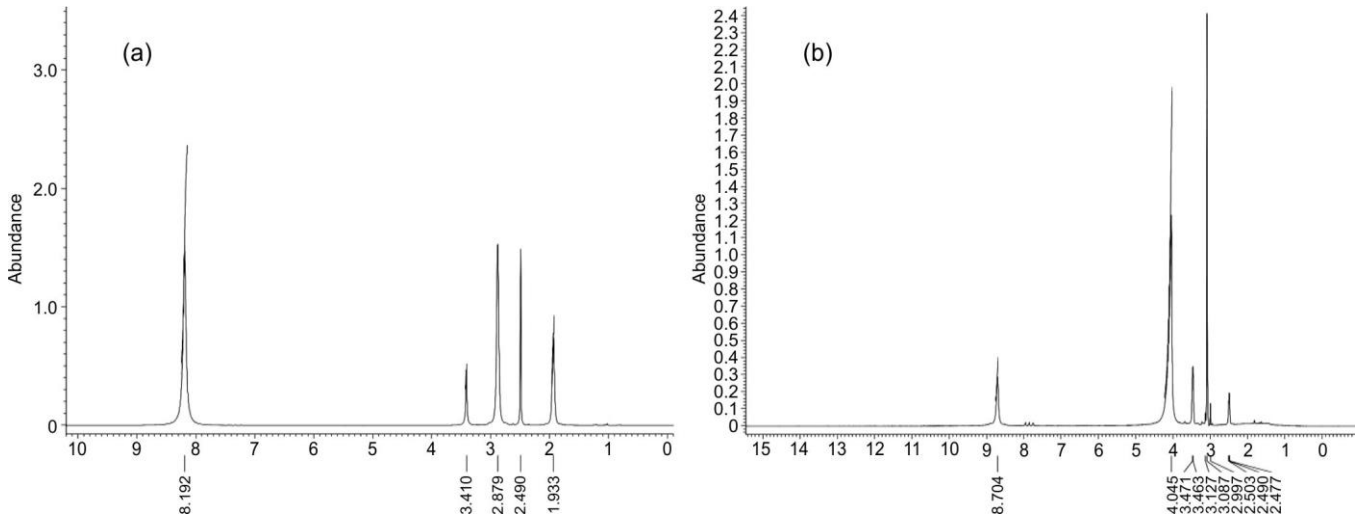


Fig. 3. ^1H NMR spectra of (a) [$RuCl_2(MacL^1)$] and (b) [$RuCl_2(MacL^2)$] macrocyclic complex

δ 4.0-2.8 ppm and δ 2.8-1.9 ppm ranges correspond to N–CH_2 and aliphatic –CH_2 protons derived from the ligand backbone [36]. The absence of extraneous or unexpected signals further supports the formation and purity of the bivalent ruthenium macrocyclic complexes.

XPS study: The XPS was employed to determine the elemental composition and oxidation state of the Ru(II) macrocyclic complexes. The survey spectra confirm the presence of Ru, N, C, O and Cl. High-resolution analysis of the Ru 3*d* region reveals two distinct components assigned to Ru $3d_{5/2}$ and Ru $3d_{3/2}$. The Ru $3d_{5/2}$ peak appears at approximately 279.8 eV, consistent with ruthenium in the +2 oxidation state, while the Ru $3d_{3/2}$ signal overlaps with the C 1s peak at around 283.8 eV.

The N 1*s* peak located at 399.4 eV indicates coordination of nitrogen donor atoms to the Ru(II) centre (N→Ru) [37,38]. The C 1*s* spectrum is characterised by contributions from aliphatic C–C environments near 283.8 eV. The O 1*s* peak observed at 530.5 eV is attributed to coordinated oxygen species. The Cl 2*p* region exhibits characteristic doublet peaks at 196.4 eV (Cl $2p_{3/2}$) and 198.4 eV (Cl $2p_{1/2}$), confirming the presence of chloride ligands bound to ruthenium [39].

The Ru $3p_{3/2}$ and Ru $3p_{1/2}$ peaks appear in 460.2-482.4 eV range, further supporting the assignment of Ru in the +2 oxidation state, in agreement with reported data [40-42]. These spectral features (Figs. 4 and 5) are consistent with the formation of [$RuCl_2(MacL^{1-2})$] macrocyclic complexes and substantiate coordination of the ligand framework to the Ru(II) centre.

Conclusion

Ruthenium(II) complexes containing tetraaza macrocyclic ligands having general formula [$RuCl_2(MacL^{1-2})$] were synthesised *via* a template condensation approach. The comprehensive characterisation including molar conductance, magnetic susceptibility, elemental analysis, UV-Vis, FT-IR, ^1H NMR and XPS, supports the formation of Ru(II) macrocyclic complexes. The combined analytical and spectroscopic data are consistent with an octahedral co-ordination environment around the Ru(II) centre. Both Ru(II) complexes exhibit good stability at ambient conditions. XPS analysis shows Ru $3p_{3/2}$ and Ru $3p_{1/2}$ peaks in the range 460.2-482.4 eV, confirming the +2 oxidation state of ruthenium.

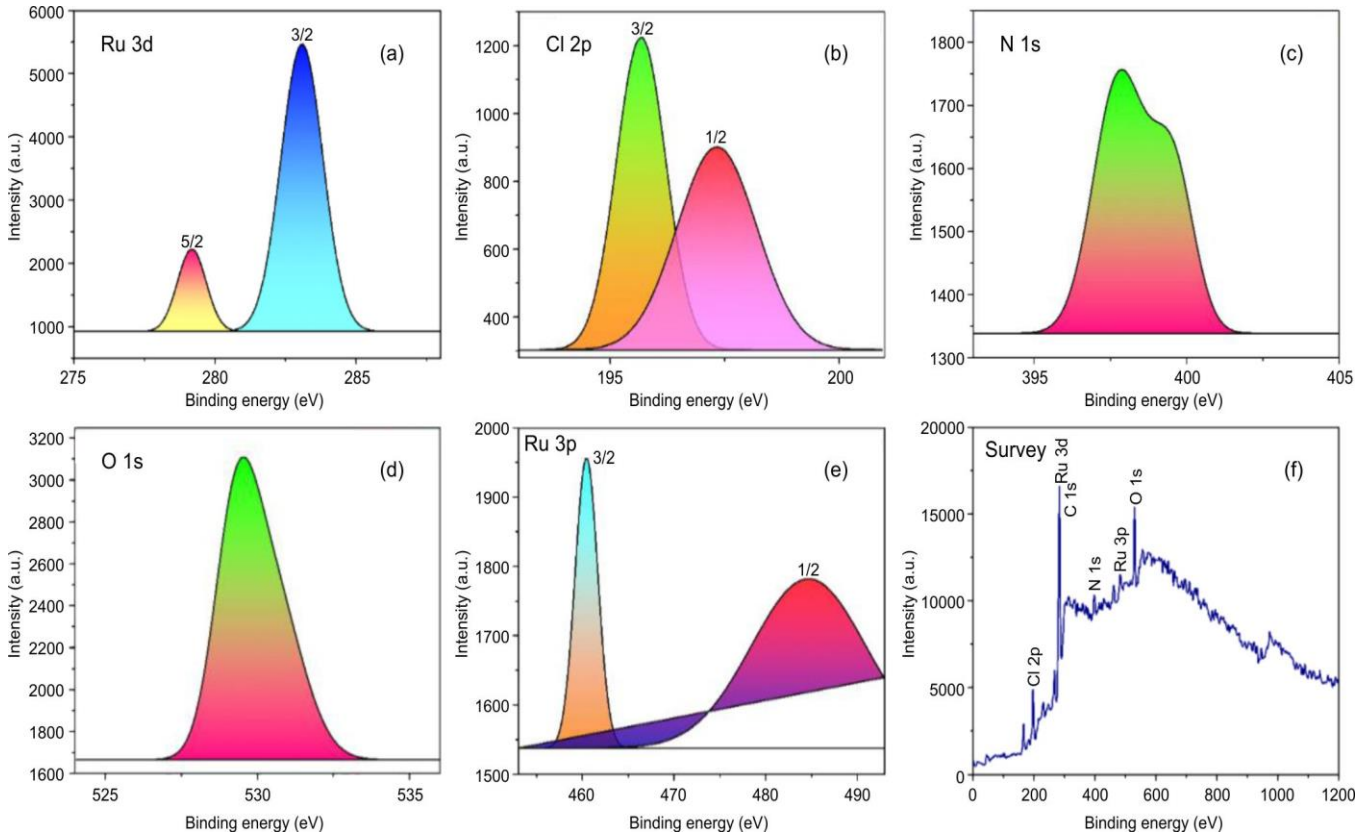


Fig. 4. High-resolution XPS spectra of (a) Ru 3d, (b) Cl 2p, (c) N 1s, (d) O 1s, (e) Ru 3p and (f) survey of essential elements of $[RuCl_2(MacL^1)]$ macrocyclic complex

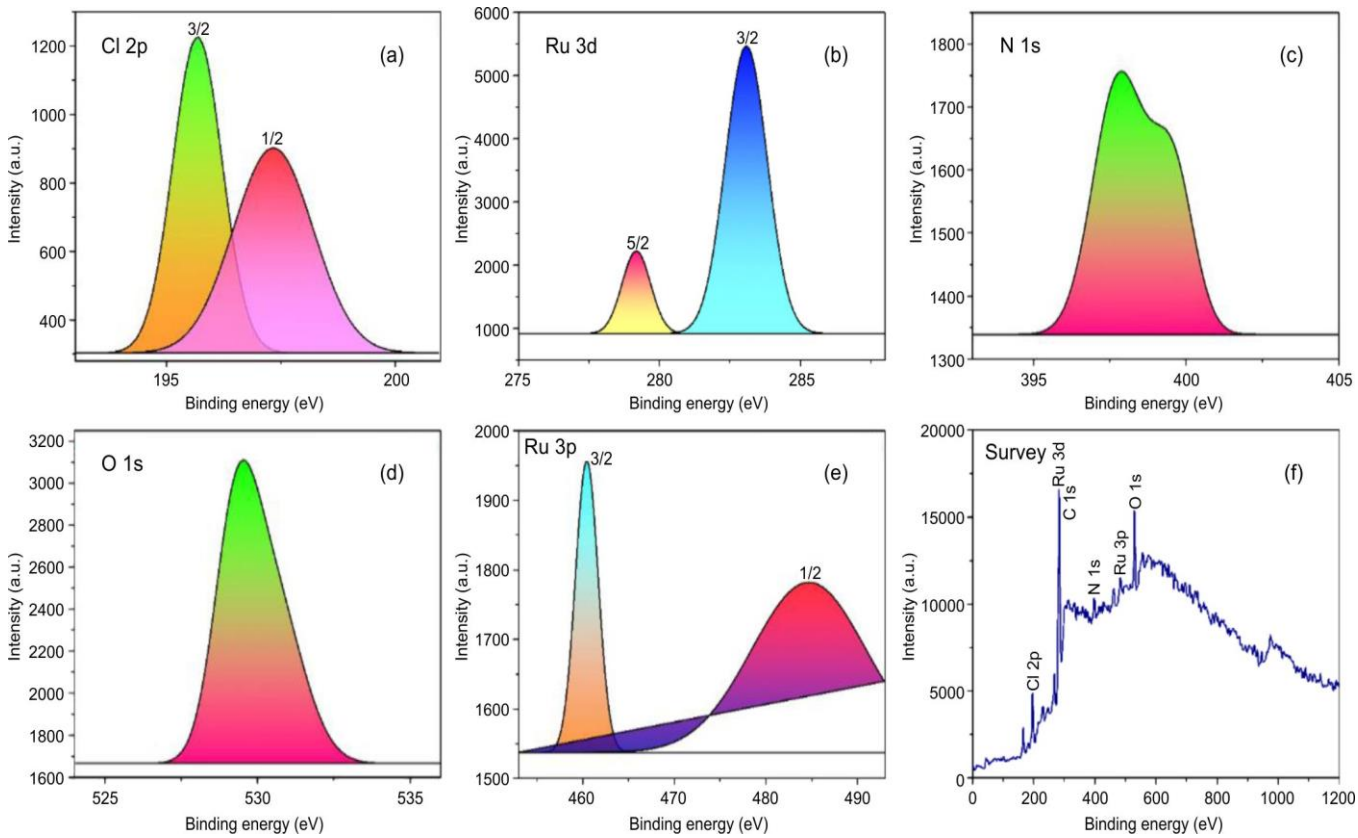


Fig. 5. High-resolution XPS spectra of (a) Cl 2p, (b) Ru 3d, (c) N 1s, (d) O 1s, (e) Ru 3p and (f) survey of essential elements of $[RuCl_2(MacL^2)]$ macrocyclic complex

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CONFLICT OF INTEREST

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

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language. The authors reviewed and edited the content and take full responsibility for the published work.

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