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Synthesis and characterization of Cu^{II} and Co^{II} complexes derived from 2,4,6-tri-(2-pyridyl)-1,3,5-triazine (TPT) by chemical and tribochemical reactions

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Four Cu^{II} and Co^{II} complexes – [Cu(L₁)Cl₂(H₂O)]₃/2H₂O · 1/2EtOH, [Cu(L₁)₂Cl₂]6H₂O, [Co(L₁)Cl₂]3H₂O · EtOH, and [Co₂(L₁)(H₂O)Cl₄]1.5H₂O · EtOH (L₁ = 2,4,6-tri(2-pyridyl)-1,3,5-triazine; TPT) – were synthesized by conventional chemical method and used to synthesize another four metal complexes – [Cu(L₁)I₂(H₂O)]6H₂O, [Cu(L₁)₂I₂]6H₂O, [Co(L₁)I(H₂O)₂] · 2H₂O, and [Co₂(L₁)I₄(H₂O)₃] – using tribochemical reaction, by grinding it with KI. Substitution of chloride by iodide occurred, but no reduction for Cu^{II} or oxidation of Co^{II}. Oxidation of Co^{II} to Co^{III} complexes was only observed on the dissolution of Co^{II} complexes in d₆-DMSO in air while warming. The isolated solid complexes (Cu^{II} and Co^{II}) have been characterized by elemental analyses, conductivities, spectral (IR, UV-Vis, ¹H-NMR), thermal measurements (TGA), and magnetic measurements. The values of molar conductivities suggest non-electrolytes in DMF. The metal complexes are paramagnetic. IR spectra indicate that TPT is tridentate coordinating *via* the two pyridyl nitrogens and one triazine nitrogen forming two five-membered rings around the metal in M:L complexes and bidentate *via* one triazine nitrogen and one pyridyl nitrogen in ML₂ complexes. In binuclear complexes, L is tridentate toward one Co^{II} and bidentate toward the second Co^{II} in [Co₂(L₁)Cl₄]2.5H₂O · EtOH and [Co₂(L₁)I₄(H₂O)₃]. Electronic spectra and magnetic measurements suggest a distorted-octahedral around Cu^{II} and high-spin octahedral and square-pyramidal geometry around Co^{II}.

Keywords: Copper(II); Cobalt(II); Tribochemistry; Triazine

1. Introduction

In the continuation of our work on tribochemical reactions for the synthesis and characterization of new metal complexes [1–8], we include metal complexes of Cu^{II} and Co^{II} derived from 2,4,6-tri-(2-pyridyl)-1,3,5-triazine (TPT). Complexes prepared by tribochemical reactions are accompanied by the substitution of Cl[−] by Br[−] and/or I[−] ions on grinding the reactants in an agate mortar with the alkali or alkaline earth halides with the reduction of Cu^{II} to Cu^I and oxidation of Co^{II} to Co^{III} in

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some cases. Papers concerning synthesis and characterization of metal complexes derived from 2,4,6-tri-(2-pyridyl)-1,3,5-triazine have been published [9–20].

2. Experimental

2,4,6-Tri-(2-pyridyl)-1,3,5-triazine; TPT was purchased from BDH and used without purification. Solvents (EtOH, Et₂O, DMF, and DMSO) and metal salts (CuCl₂ · 2H₂O, and CoCl₂ · 6H₂O) were of BDH quality.

2.1. Metal complexes

2.1.1. Preparation of Cu^{II} complex by chemical method. [Cu(L₁)Cl₂(H₂O)]₃/2H₂O · 1/2EtOH was prepared by adding a solution of TPT (0.31 g; 10⁻³ mol) dissolved in ~25 cm³ EtOH to a solution of CuCl₂ · 2H₂O (0.17 g; 10⁻³ mol) dissolved in 30 cm³ EtOH. The reaction mixture was refluxed on a water bath for 2 h and then left to cool at 25°C. The product was filtered off, washed several times with EtOH and Et₂O, and finally dried in a desiccator over P₄O₁₀.

[Cu(L₁)₂Cl₂]₆H₂O was prepared by adding a solution of CuCl₂ · 2H₂O (0.17 g; 10⁻³ mol) dissolved in 20 cm³ EtOH to a solution of TPT (0.62 g; 10⁻³ mol) dissolved in ~50 cm³ EtOH. The complexes were filtered off and washed with EtOH and Et₂O.

2.1.2. Preparation of Co^{II} complexes by chemical method. [Co(L₁)Cl₂]₃H₂O · EtOH was synthesized by dissolving 0.31 g (10⁻³ mol) of TPT in 50 cm³ EtOH and adding to a solution of 0.24 g (10⁻³ mol) of CoCl₂ · 6H₂O in EtOH (50 cm³) with constant stirring for 2 h. The reaction mixture was refluxed on a water bath for 3 h and then left to cool at 25°C. The product was filtered off, washed several times with EtOH and Et₂O, and finally dried in a desiccator over P₄O₁₀. [Co₂(L₁)(H₂O)Cl₄]_{1.5}H₂O · EtOH was prepared by adding a solution of 0.31 g (10⁻³ mol) of TPT in 50 cm³ EtOH to a solution of 0.48 g (10⁻³ mol) of CoCl₂ · 6H₂O in EtOH (75 cm³) with constant stirring for 1 h. The reaction was refluxed on a water bath for 1.5 h and then left to cool at 25°C. The product was filtered off and washed with EtOH and Et₂O.

2.1.3. Preparation of Cu^{II} and Co^{II} complexes by tribochemical reaction. [Cu(L₁)I₂(H₂O)]₆H₂O, [Cu(L₁)₂I₂]₆H₂O, [Co(L₁)I₂(H₂O)]₃H₂O and [Co₂(L₁)I₄(H₂O)]₃ were prepared by grinding ~0.5 g of the Cu^{II} and Co^{II} complexes derived from the metal chloride with excess KI (6 g) in agate mortar till the color changed. A mixture of MeOH (50 cm³) and EtOH (50 cm³) was then added, refluxed for 3 h, and then left overnight. The products were filtered off and dried at 80°C for 2 h. All the isolated solid complexes were kept in a desiccator over silica gel.

2.2. Physical measurements

The Cu^{II} and Co^{II} contents were determined by complexometric titration using xylenol orange [11]. Carbon, hydrogen, and nitrogen were determined at the Microanalytical Unit, Center of King Fahad Institute, Jeddah, KSA. Molar conductivities were carried

out using a Tacussel model CD 75 at Mansoura University, Mansoura, Egypt. IR spectra from 200 to 4000 cm⁻¹ were recorded in KBr on a Mattson 5000 FT-IR Spectrometer at Mansoura University, Egypt. Electronic spectra were recorded in Nujol mull from 200 to 900 nm on a Unicam spectrometer model UV2 at Mansoura University, Egypt. ¹H-NMR spectra of the ligand were recorded on Jeol-90Q Fourier transform (200 MHz) in CDCl₃ at Cairo University, Egypt and ¹H-NMR in d₆-DMSO Spectrometer (400 MHz) at King Saud University, Riyadh, KSA. Magnetic moments were determined using a Sherwood balance at room temperature (25°C) with Hg[Co(NSC)₄] as a calibrant at Mansoura University. Diamagnetic corrections for the ligand and the metal atoms were computed using Pascal's constants [21]. Thermal analysis measurements (TGA and DTG) were recorded on a Shimadzu model 50 instrument using 20 mg for [Co(L₁)I₂(H₂O)]3H₂O. The nitrogen flow rate and heating were 20 cm³ min⁻¹ and 10°C min⁻¹, respectively.

3. Results and discussion

Analytical and physical data for the complexes are listed in table 1. All the isolated metal complexes are colored, stable in air for more than 6 months and insoluble in most common organic solvents, but freely soluble in DMF and DMSO. The molar conductivities in DMF at 25°C fall in the 4.0–28.0 Ω⁻¹ cm² mol⁻¹ range (table 1), indicating non-electrolytes [22] in this solvent. The values of molar conductivities for some metal complexes (16–28 Ω⁻¹ cm² mol⁻¹) are due to partial dissociation in DMF. The metal complexes have comparatively high melting points (>300°C) except two complexes, which have melting points in the range 270–280°C, suggesting strong bonding between the metal ion and the ligand.

IR spectrum of 2,4,6-tri-(2-pyridyl)-1,3,5-triazine (TPT) in KBr shows six bands at 1654, 1645, 1582, 1528, 1471, and 1437 cm⁻¹ assigned to ν(C=N) (triazine, free), ν(C=N) (triazine, hydrogen-bonded), ν(C=N) (Py, free), ν(C=N) (Py, hydrogen-bonded), ν(C=C) (Py, free), and ν(C=C) (Py, hydrogen-bonded), respectively. The spectrum also shows strong bands at 3393 and 3264 cm⁻¹ assigned to ν_{as}(H₂O) and ν_s(H₂O) from contamination of KBr with small amounts of H₂O. The existence of water within the ligand forms external hydrogen bonding between C=N of TPT and OH (H₂O). Weak broad bands at 2300–1800 cm⁻¹ confirm hydrogen bonding of the type O–H...N [23] as shown in **1** [24].

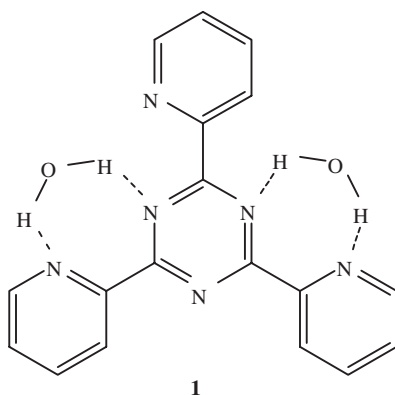


Table 1. Analytical data and some physical properties of the isolated metal complexes.

Compound	Color	m.p. (°C)	Found (Calcd) (%) (Cl; I)						Λ ($\Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$) in DMF	μ_{eff}
			C	H	N	M	X			
$[\text{Cu}(\text{L}_1)\text{Cl}_2(\text{H}_2\text{O})]_3 \cdot 2\text{H}_2\text{O} \cdot 1/2\text{EtOH}$	Green	280 d	44.3 (44.1)	3.9 (3.8)	16.4 (16.1)	12.3 (12.0)	13.8 (13.7)	23.0	1.90	
$[\text{Cu}(\text{L}_1)_2\text{Cl}_2]_6 \cdot \text{H}_2\text{O}$	Pale green	>300	49.9 (49.5)	4.2 (3.7)	19.4 (19.0)	7.3 (6.9)	8.2 (8.1)	26.0	1.79	
$[\text{Co}(\text{L}_1)\text{Cl}_2]_3 \cdot 3\text{H}_2\text{O} \cdot \text{EtOH}$	Olive green	>300	44.3 (43.8)	4.5 (4.1)	15.5 (15.1)	10.9 (10.4)	13.1 (12.9)	25.0	4.20	
$[\text{Co}_2(\text{L}_1)(\text{H}_2\text{O})\text{Cl}_4] \cdot 1.5\text{H}_2\text{O} \cdot \text{EtOH}$	Deep green	>300	36.2 (35.8)	3.5 (3.5)	12.7 (12.3)	17.8 (17.1)	21.4 (21.1)	28.0	4.10	
$[\text{Co}(\text{L}_1)_2(\text{H}_2\text{O})]_3 \cdot \text{H}_2\text{O}$	Brick red	>300	31.0 (30.7)	2.9 (2.5)	12.1 (11.6)	8.5 (8.1)	36.4 (36.0)	16.0	4.85	
$[\text{Co}_2(\text{L}_1)_4(\text{H}_2\text{O})_3]$	Chocolate brown	>300	21.8 (21.6)	1.8 (1.4)	8.5 (7.9)	11.9 (11.6)	51.2 (51.0)	5.0	5.20	
$[\text{Cu}(\text{L}_1)_2(\text{H}_2\text{O})]_6 \cdot \text{H}_2\text{O}$	Deep green	270 d	28.6 (28.1)	3.5 (3.3)	11.1 (10.8)	8.4 (8.1)	33.6 (33.5)	4.0	1.83	
$[\text{Cu}(\text{L}_1)_2]_6 \cdot \text{H}_2\text{O}$	Black	>300	41.2 (40.8)	3.5 (3.3)	16.0 (15.4)	6.1 (5.4)	24.2 (23.8)	5.0	1.88	

d, decomposed.

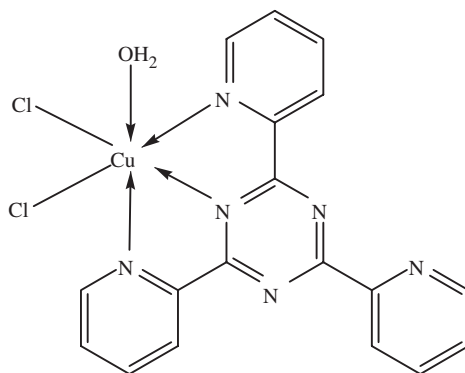
 μ_{eff} , effective magnetic moment.

In Nujol mull, the ligand shows four bands due to the C=N (triazine and Py) and C=C vibrations at 1678, 1660, 1587, and 1524 cm⁻¹; C=N bands at higher wave numbers indicate the partial destruction of hydrogen bonding between C=N and OH. The C=C vibrations remain more or less at the same positions indicating that they do not participate in hydrogen bonding. Decrease of intensity of the OH bands and observation of bands at 3371 and 3234 cm⁻¹ suggests that hydrogen bonding is weak, but still exists within the ligand. The ¹H-NMR spectrum of TPT in d₆-DMSO shows a signal at 4.77 ppm assigned to H₂O. The spectrum shows four signals (Supplementary material) with equal intensity at 7.72, 8.13, 8.75, and 8.91 ppm, with respect to TMS.

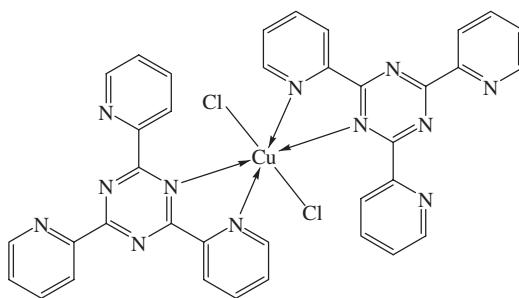
Comparing ¹H-NMR spectra of L₁ with metal complexes confirm participation of two azomethine nitrogens (C=N) and azomethine of triazine moiety, while it coordinates *via* three azomethine nitrogens (C=N) for three pyridine rings and two azomethine nitrogens of the triazine moiety in 2 : 1 (M : L) complexes.

IR spectra of complexes have been carried out in Nujol mull to prevent substitution of Cl⁻ by Br⁻ ions [7]. The complexes show bands at 3534–3455, 3408–3382, and 3297–3290 cm⁻¹ except [Cu(L₁)Cl₂]6H₂O, [Co(L₁)Cl₂]3H₂O · EtOH and [Cu(L₁)₂Cl₂]6H₂O, which exhibit only one band. This suggests that these bands are due to coordinated H₂O, since these complexes do not contain coordinated water. Spectra of metal complexes show that C=N shift to lower wave numbers indicating participation in bonding.

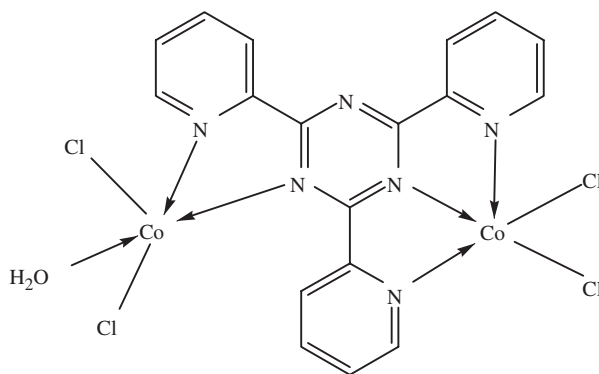
The electronic spectra of [Cu(L₁)Cl₂(H₂O)]3/2H₂O · 1/2EtOH and [Cu(L₁)₂Cl₂]6H₂O show a broad band centered at 12,690 and 12,346 cm⁻¹, respectively, assigned to ²E_g → ²T_{2g} transition in a distorted-octahedral geometry around Cu^{II}. Values of the magnetic moment, 1.9 and 1.85 B.M. (table 1), respectively, are evidence for d⁹ configuration and distorted-octahedral geometry around Cu^{II}. Values of the magnetic moments of [Co(L₁)Cl₂]3H₂O · EtOH and [Co₂(L₁)(H₂O)Cl₄]1.5H₂O · EtOH, 4.1 and 4.2 B.M., respectively, suggest either tetrahedral or five-coordinate geometry around Co^{II} [25]. Electronic spectra of the two Co^{II} complexes are identical with 17,007, 15,385 and 16,835, 15,432 cm⁻¹, respectively, suggesting square-pyramidal geometry around Co^{II} [25, 26]. The bands are assigned to ⁴A₂(F) → ⁴B₁(P) and ⁴A₂(F) → ⁴A₂(P). The spectral (UV, IR) and magnetic data suggest structures **2** and **3** for the Cu^{II} complexes and **4** and **5** are proposed for the Co^{II} complexes.



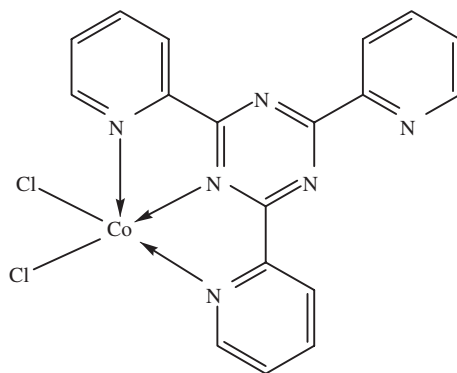
2



3



4

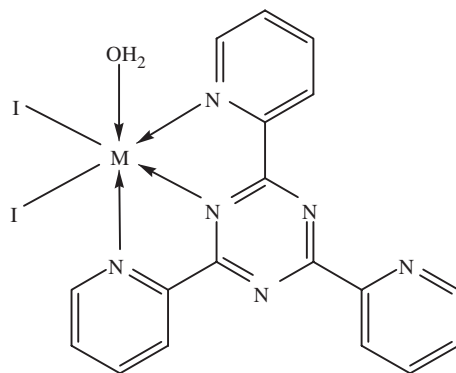


5

IR spectra of **2**, **4**, and **5** show six bands at ~ 214 , 244, 260, 280, 308, and 340 cm^{-1} . The first two bands are assigned to *cis* $\nu(\text{M}-\text{Cl})$, while one band at 250 cm^{-1} for **3** suggests two chlorides are *trans*. The other four bands are attributed to $\nu(\text{M}-\text{N})$ of the pyridine and triazine moiety. Water molecule inside the coordination sphere of **2** and **4** is determined by weight loss method using gravimetric technique.

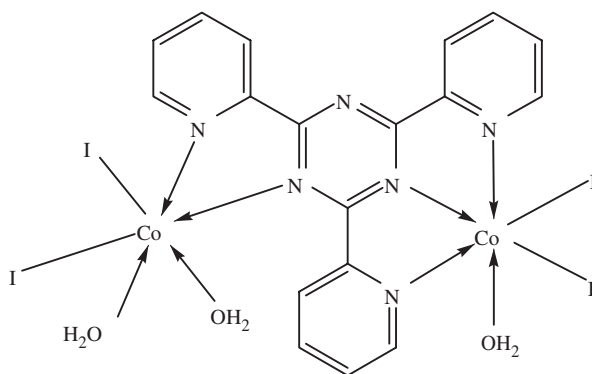
$[\text{Cu}(\text{L}_1)\text{I}_2(\text{H}_2\text{O})]6\text{H}_2\text{O}$, $[\text{Cu}(\text{L}_1)_2\text{I}_2]6\text{H}_2\text{O}$, $[\text{Co}(\text{L}_1)\text{I}_2(\text{H}_2\text{O})]3\text{H}_2\text{O}$, and $[\text{Cu}(\text{L}_1)_2\text{I}_2]6\text{H}_2\text{O}$ are deep in color, assigned to high polarization effect between iodide and metal [27]. The low molar conductivities indicate a strong bonding between the metal ion and the ligand.

IR spectra show that the ligand is tridentate *via* two nitrogens of pyridyl and triazine in 1:1 (M:L). The ligand is bidentate toward one metal and tridentate toward the second metal in complexes with 2:1 (M:L) ratio (**6** and **7**). The ligand is bidentate *via* nitrogen of pyridyl and triazine as for 1:2 (M:L) Cu^{II} complex, as shown in **8**. IR spectrum of **8** shows only one band at 316 cm⁻¹ assigned to *trans* $\nu(\text{Cu-I})$, while the other complexes exhibit two bands at 290 and 340 cm⁻¹ attributed to *cis* $\nu(\text{Cu-I})$.

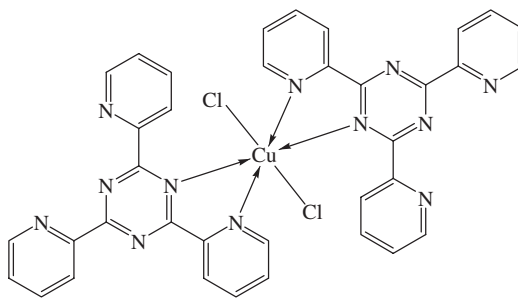


M = Cu^{II} or Co^{II}

6



7



8

H₂O and/or EtOH inside or outside the coordination sphere were determined using gravimetric technique, since their melting points are very high. Amount of the solvent outside the coordination sphere was determined by heating the metal complexes in an oven at 110°C, while the total amount of solvents (inside and outside the coordination sphere) were determined by heating the complexes to 160°C. Amounts of H₂O inside and outside the coordination sphere were determined using thermal measurements (TGA). The thermogram of the Co(II) complex, [Co(L₁)I₂(H₂O)]3H₂O, shows that at 90–110°C three water molecules of hydration are lost (estimated mass loss 7.8%, theoretical 7.3%). The second step (125–190°C) involves the loss of one water molecule (estimated mass loss 2.6%, theoretical 2.4%).

The ¹H-NMR spectrum of [Co(L₁)I₂(H₂O)]3H₂O in d₆-DMSO shows three signals with equal intensity at 16.3, 15.1, and 14.8 ppm and a weaker signal at 14.3 ppm. The observation of high-resolution signals suggests oxidation of Co^{II} to Co^{III} on dissolution in d₆-DMSO followed by oxidation by air during warming the solution. ¹H-NMR spectrum of [Co₂(L₁)I₄(H₂O)₃] shows four signals at 16.2, 15.1, 14.3, and 13.2 ppm (Supplementary material). The first three bands are equal in their intensity, while the fourth is less than the other three signals. The results suggest that the cobalt(II) complexes in the solid state are oxidized in d₆-DMSO to Co^{III}.

4. Conclusion

Complexes were prepared and characterized using chemical and tribochemical methods. Cu(II) complexes synthesized by tribochemical reactions were reduced to Cu(I) followed by the substitution of chloride by iodide ions. Some Co(II) complexes were oxidized to Co(III) followed by substitution of chloride by iodide ions. In Pd(II), Pt(II), and Ru(III), no reduction was observed on the substitution of chloride by iodide. All the isolated Cu^{II} and Co^{II} complexes are derived from 2,4,6-tri-(2-pyridyl)-1,3,5-triazine; TPT showed no reduction of Cu^{II} to Cu^I or oxidation of Co^{II} to Co^{III}. TPT coordinates to the metal ions (Cu^{II} and Co^{II}) in a bidentate and/or tridentate manner or both and highly conjugated system stabilizes the Co^{II} and Cu^{II}, preventing reduction or oxidation.

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