

Synthesis and Characterization of Co (II), Ni (II), Cu (II), and Pd(II) Complexes with Mono Sodium 2, 4, 6-Trioxotetrahydropyrimidine-1(2H)-Carbodithioate

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Abstract

Mono sodium 2,4,6-trioxotetrahydropyrimidine-1(2H)-carbodithioate (L) was prepared from barbituric acid with carbon disulfide (CS₂) in the presence of sodium hydroxide, then reacted with some chloride metals to synthesis a complexes of the types [M (L) Cl (H₂O)₃], [M (L)₂(H₂O)₂] and [M(L)₂] {M= Co(II), Ni(II), Cu(II)}, and [Pd (L)(H₂O)₂]Cl and [Pd (L)₂]. These compounds were characterized using infra-red spectra FTIR, electronic spectra UV–VIS, proton nuclear magnetic resonance ¹H-NMR spectroscopy, Micro Elemental Analysis, and Magnetic Measurements. These measurements indicate that the prepared complexes have octahedral, square planer or tetrahedral geometry. Molar Conductance measurements shows all the prepared complexes are non-electrolytic except the palladium complex of the type [Pd(L)(H₂O)₂]Cl.

Keywords: Barbituric acid, carbon disulfide, carbodithioate.

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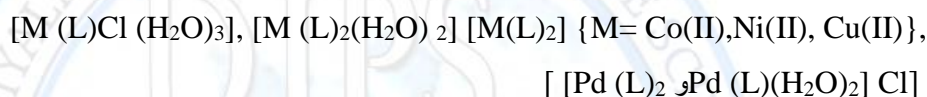
تحضير و تشخيص معقدات الكوبلت، النيكل، النحاس والبلاديوم بتكافؤهم الثنائي مع 6,4,2 ثلاثي أوكسو رباعي هيدروبيريميدين-1 كاربو ثنائي ثايويت احادي الصوديوم

أنوار عادل حميد

قسم الكيمياء- كلية العلوم- جامعة تكريت

الخلاصة

حُضِرَ المشتق 6,4,2 ثلاثي أوكسو رباعي هيدروبيريميدين-1 كاربو ثنائي ثايويت احادي الصوديوم (L) من حامض الباربيتيوريك مع ثنائي كبريتيد الكربون بوجود هيدروكسيد الصوديوم ومن ثم مفاعله مع بعض من كلوريدات الفلزات الانتقالية لتحضير المعقدات ذات الصيغ العامة:



شُخِصَت المركبات المحضرة بإستعمال تقنيات الأشعة تحت الحمراء، الأطياف الألكترونية، طيف الرنين النووي المغناطيسي للبروتون، التحليل الدقيق للعناصر والقياسات المغناطيسية. أكدت هذه القياسات إن المعقدات تمتلك بنية ثنائي السطوح، مربع مستوي، أو بنية رباعي السطوح، كما اوضحت قياسات التوصيلية المولارية إن جميع المعقدات غير الكتروليتية بإستثناء معقد البلاديوم ذو التركيب $[Pd(L)(H_2O)_2] Cl$.

الكلمات المفتاحية: حامض الباربيتيوريك، ثنائي كبريتيد الكربون، كاربو ثنائي ثايويت

Introduction

Organic chemistry of dithiocarbamates have attracted a wonderful deal of importance because of their exciting chemistry and huge effectiveness. Dithiocarbamates, are chelating agents with various applications, cytotoxic properties, chelating agent for the extraction of trace metals, have been used in against metal poisoning ^[1-3] and different various applications in agriculture, medicinal drug, industry, analytical and organic chemistry ^[2,3]. Its complexes show improved antibacterial, antimalarial, and fungicidal activities as compared to free ligands because of recede in polarity of metals after complexation ^[4,5].

Dithiocarbamate ligands can coordinate to all the transition elements in order to stabilize low- or high-valent metal ions^[6].

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Dithiocarbamates have specific binding modes to metal ions (figure 1). It acts as a chelate ligand (1) via two sulfur atoms^[6], and monodentate coordination mode (2) is also particularly common^[7]. In a few cases the two metal sulfur bonds are quite exceptional but nonetheless within the anticipated variety for a bonding interaction. That is commonly termed the anisobidentate coordination mode (3)^[8]. All other binding modes (4-6) involve the dithiocarbamate bridging two or extra metal atoms, as much as a maximum of four^[9].

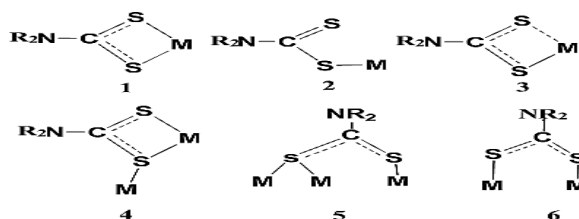


Figure 1: Binding modes of dithiocarbamate complexes^[3,4]

Experimental part

Chemicals and Instruments

Reagent grade barbituric acid (B.A) and sodium hydroxide were procured from Alfa aesar. Sodium hydroxide, Cobalt chloride hexa hydrate, Nickel chloride hexa hydrate, Copper chloride di hydrate and, while sodium chloride, hydrochloric acid 37% and palladium chloride from Sigma- Aldrich. Chloroform, Carbon disulphide, ethanol and dimethyle sulphoxide were procured from Sharlau.

The infra-red spectra of ligand and metal complexes spectra were recorded by SHIMADZU spectrophotometer within the range (400-4000 cm^{-1}). The electronic spectra of the complexes were recorded in SHIMADZU 2450 UV-VIS as 10^{-3} M DMSO solution. The magnetic susceptibilities were measured at room temperature by using Sherwood scientific instrument (Evans balance). Conductivity measurements were recorded by using dimethyl sulphoxide (DMSO) as a solvent for 10^{-3} M solution of the complexes at room temperature, using (Consort C832) instrument. The ^1H - NMR measurements were measured by using DMSO- d_6 as solvent in Bruker MHz-Avance III instrument (300 MHz) and the micro element analyses

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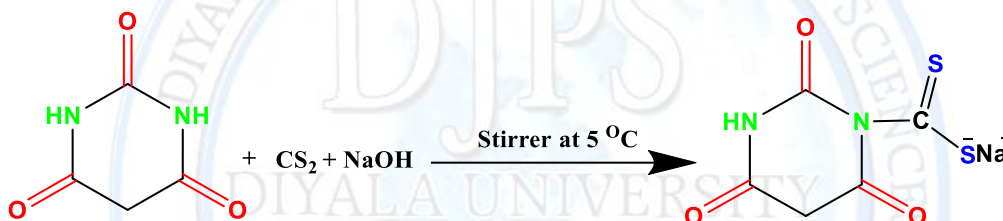
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recorded in Bruker S2 picofox, while metal percentage of some selected complexes have been measured using atomic absorption Pye-Unicam Sp.9 Spectrophotometer.

Syntheses of the ligand and complexes:

Barbituric acid (dithiocarbamate) (B.A.DTC):

The ligand was prepared according to typical method [10-13]. A solution of Barbituric acid (0.4g, 3 mmol) in (20 ml) DMSO was added to CS₂ (0.235g, 3.1mmol) in the presence of NaOH (0.125 g, 3 mmol). The reaction was maintained at 5 °C using a water-ice bath. This mixture was subjected to continuous stirring for 1 hour and maintained at room temperature for three weeks till beige solid was formed. The obtained solid was filtered, recrystallized from (3:1 (90%ethanol): Chloroform) and dried in a vacuum oven at 40 °C for 4 hours.



Preparation of [M (L) Cl (H₂O)₃] complexes. M= Co (II), Ni (II) and Cu (II).

To aqueous solution 8ml of (L) (0.6mmol), a solution of (0.6mmol) of Co (II), Ni (II) and Cu (II) salts correspondingly, in 5 ml distilled water, was added drop wise under stirring. The reaction mixture was refluxed for 1 hour on steam bath, and then was filtered; the resulting colored precipitate was washed with hot distilled water and dried at 60 °C for 4 hours.

Preparation of [M (L)₂ (H₂O)₂] complexes. M= Co (II), Ni (II) and Cu (II).

To aqueous solution 15 ml of (L) (1.2 mmol), aqueous solution of (0.6 mmol) Co (II), Ni(II) and Cu(II), correspondingly, in 5 ml distilled water, was added drop wise under stirring. The reaction mixture was refluxed for 2 hours on steam bath, filtered, the resulting colored precipitate was washed with hot distilled water in order to remove unreacted (L and metal salts) and dried at room temperature.

Preparation of [M (L)₂] complexes. M= Co (II), Ni (II) and Cu (II).

These complexes were prepared by drying complexes of the type [M (L)₂ (H₂O)₂] at 100 °C for 24 hours.

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Preparation of palladium salt (Na_2PdCl_4)^[14].

To 10ml concentrated HCl 37%, (2g, 11.3mmol) PdCl_2 was added slowly with continuously stirring by glass rod in a hot water bath, till the total volume reduced to the one-third of its volume, a saturated aqueous solution (1.32g, 22.6mmol) of NaCl was added slowly with continuously stirring by glass rod in water bath, after that the solution was left until became deep red brown powder, the obtained powder was washed three times with diethylether and dried at 60 °C for three hours in oven.

Preparation of $[\text{Pd}(\text{L})(\text{H}_2\text{O})_2]\text{Cl}$ complex.

An aqueous solution 20 ml of Na_2PdCl_4 (0.177g, 0.6 mmol) was added to an aqueous solution 30 ml of (L) (0.2g, 0.6 mmol), this mixture was refluxed for 4 hours. The obtained brown solid was then washed with hot distilled water and dried at 60°C for 4 hours.

Preparation of $[\text{Pd}(\text{L})_2]$ complex.

An aqueous solution 20 ml of Na_2PdCl_4 (0.177g, 0.6 mmol) was added to an aqueous solution 30 ml of (L) (0.4g, 1.2 mmol), this mixture was refluxed for 4 hours. The obtained brown filtered was then evaporated to gives dark brown solids, washed with hot distilled water and dried at 60°C for 4 hours.

Results and Discussion

The Dithiocarbamate ligand and its Co(II), Ni(II), Cu(II) and Pd(II) complexes have been synthesized and characterized using IR spectra, electronic spectra, magnetic susceptibility, conductivity measurements, ¹H- nuclear magnetic resonance, micro element analyses, and the atomic absorption.

The physical properties of ligand and its complexes are listed in Table 1. All complexes are thermally stable and have a good solubility in DMSO and DMF. The molar conductivities of 10⁻³ M solution of all complexes (Table1) indicate that they are non-electrolytes except $[\text{Pd}(\text{L})(\text{H}_2\text{O})_2]\text{Cl}$ ^[15].

IR Spectra:

The coordination modes of the ligand to the metal ions have been investigated by comparing the infrared spectra of the free ligand with its metal complexes (Table 2). The band of $\nu(\text{C}=\text{N})$ was appeared at 1458 cm⁻¹ in free ligand and shifted towards high values at rang in

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the spectra of the complexes ^[16-18]. The two bands of ν (C..S) of the ligand appeared at 1033 and 933 cm^{-1} , as well as ν (C..S) in all complexes shifted towards lower values. We noticed all complexes show a single peak at the range (991-1030) cm^{-1} ^[19,20], that is mean the ligand is bidentate through two sulfur atoms. The bands at (418-475) cm^{-1} could be attributed to ν (M-OH₂)^[21].

Electronic Spectra:

The electronic spectra of the complexes were recorded as 10^{-3} M solution in DMSO and the results presented in Table 3. The electronic spectra of four coordinated Co(II) complex exhibited one absorptions at (15313) cm^{-1} which was assigned to (²A_{1g} → ²E_g) in a distorted tetrahedral geometry around cobalt (II). The electronic spectra of four coordinated Ni(II) and Pd(II) complexes shows two absorption at (24194, 23818-23773) cm^{-1} and (15576, 15822-15757) cm^{-1} respectively, assigned to ¹A_{1g} → ¹B_{1g} and ¹A_{1g} → ¹A_{2g}. These bands are in agreement with square planar nickel (II) and palladium complexes ^[22, 23]. The electronic spectra of the prepared four coordinated copper (II) complex gives broad absorption band at (15124) cm^{-1} , which can be assigned to the combination of transitions (²B_{1g} → ²A_{1g}) and (²B_{1g} → ²E_g). The position of these bands is in agreement with four coordinated copper (II) complexes with square-planar geometry ^[22,24,25]. The electronic spectra of six coordinated Co(II) complex exhibited two absorptions bands at (15667-16007) cm^{-1} and (20167,20100) cm^{-1} which was assigned to (⁴T_{1g} → ⁴A_{2g}) and (⁴T_{1g} → ⁴T_{1g}(p)) in octahedral geometry around cobalt (II). The electronic spectra of six coordinated Ni(II) complexes shows three absorption bands at (11733-12001,13024-13330,27373-25100) cm^{-1} , assigned to ³A_{2g} → ³T_{2g}(\square_1), ³A_{2g} → ³T_{1g} and ³A_{2g} → ³T_{1g}(p). These bands are in agreement with octahedral nickel (II) complexes ^[22, 23]. The electronic spectra of the prepared six coordinated copper (II) complexes give absorption band at (14233-14400 and 16051-16377) cm^{-1} , which can be assigned to the combination of transitions (²B_{1g} → ²A_{1g}) and (²B_{1g} → ²E_g). The position of these bands is in agreement with six coordinated copper (II) complexes with octahedral geometry.

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Magnetic susceptibility:

The experimental magnetic moment for each complex is listed in Table 1. The magnetic moment give an idea about the electronic state of the metal ion in the complex. The magnetic moment of six coordinated complexes are, Co (II) complexes are (5.01, 5.00) B.M respectively ^[28], for Ni (II) complexes are (2.98, 3.01) B.M ^[29,30]. For Cu (II) complexes are (1.81, 1.99) B.M ^[31]. These values are due to the presence of octahedral configuration for these complexes, while the magnetic moment of four coordinated complexes are, Co (II) complex is (2.40) B.M this indicate that cobalt has distorted tetrahedral structure ^[28], but Ni (II) and Pd(II) gives no value, that is indicate that nickel and palladium complexes have square planar geometry, while Cu (II) complex is (1.66) B.M, this value is appropriate for square planar complex ^[29,30]. As shown in table 1.

Proton nuclear magnetic resonance (¹H- NMR) Spectra Measurements:

¹H-NMR spectra (figure 6-8), the NaBADTC shows a single signal at (3.505ppm) with integration 1.96 . This signal is a good proof to the existence of (CH₂), another signal at (10.92 ppm) with integration equal 1.00 refers to the proton of NH as in figure (6). The ¹H-NMR of [Pd(L)(H₂O)₂].Cl complex shows a single signal at (2.960 ppm) with integration 2.02 while the proton of NH gives integration equal 1.00 at (10.93ppm), we must mentioned that two water molecules which might possibly be replaced by DMSO molecules when the complex is dissolved in this solvent. as in figure (7), while the ¹H-NMR of the complex [Pd(L)₂] gives a single signal at (3.024ppm) with integration 4.06 attributed to two CH₂ groups from and another signal at (10.72ppm) with integration equal 1.91 due to presence of two NH group from two ligand which attached to the same palladium Figure 8.

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Table 1: Analytical, conductance and magnetic data of the ligands and their complexes

No.	Formula	Color	Yield %	M.P °C	Element & Metal analyses calc./ (found) %				Λ	μ _{eff}
					C	H	N	M		
Lig	L	Beige	78	340<	26.55 (26.42)	1.34 (1.32)	12.38 (12.33)	-	-	-
1	[Co(L)(H ₂ O) ₃ Cl]	green	50	254-261	17.08 (16.99)	2.58 (2.33)	7.97 (7.93)	16.76 (16.74)	1	4.32
2	[Ni(L)(H ₂ O) ₃ Cl]	Maroon	74	226-230	17.09 (17.02)	2.58 (2.53)	7.97 (7.99)	16.70 (16.81)	7	2.98
3	[Cu(L)(H ₂ O) ₃ Cl]	olive	87	188-193	16.86 (17.00)	2.55 (2.55)	7.86 (7.91)	17.84 (17.90)	3	1.81
4	[Co(L) ₂ (H ₂ O) ₂]	Dark green	81	158-162	23.96 (23.91)	2.01 (2.11)	11.17 (11.17)	11.75 (11.75)	5	4.91
5	[Ni(L) ₂ (H ₂ O) ₂]	Magenta	79	182-189	23.97 (23.97)	2.01 (2.09)	11.18 (11.15)	11.71 (11.77)	13	3.01
6	[Cu(L) ₂ (H ₂ O) ₂]	Sea green	72	200-201	23.74 (23.76)	1.99 (1.99)	11.07 (11.10)	12.56 (12.60)	2	1.99
7	[Co(L) ₂]	Tan	60	>300	25.81 (25.79)	1.30 (1.29)	12.04 (11.99)	12.66 (12.71)	1	2.40
8	[Ni(L) ₂]	red	63	Decomp.	25.82 (25.79)	1.30 (1.29)	12.05 (12.10)	12.62 (12.67)	0	Diamagnetic
9	[Cu(L) ₂]	black	55	>300	23.74 (23.76)	1.99 (1.99)	11.07 (11.10)	12.56 (12.60)	2	1.66
10	[Pd(L)(H ₂ O) ₂ Cl]	Brown	61	187-189	15.76 (15.76)	1.85 (1.80)	7.35 (7.55)	27.92 (27.93)	97	Diamagnetic
11	[Pd(L) ₂]	Dark brown	42	>320	23.42 (23.40)	1.18 (1.18)	10.93 (11.00)	20.75 (20.99)	0	Diamagnetic

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Table 2: ν (C-N), ν (C-S), ν (M-OH₂), and ν (C=O) frequencies

No.	Formula	ν (C-N)	ν (C-S)	ν (M-OH ₂)	ν (C=O)
L	L	1458 (s)	1033 (s) 933 (s)	-	1676 (s)
1	[Co(L)(H ₂ O) ₃ Cl]	1510(m)	1003 (s)	440(m)	1670 (s)
2	[Ni(L)(H ₂ O) ₃ Cl]	1515 (s)	1009 (s)	423(m)	1676 (s)
3	[Cu(L)(H ₂ O) ₃ Cl]	1533 (s)	1026 (m)	475(w)	1674 (s)
4	[Co(L) ₂ (H ₂ O) ₂]	1500 (s)	1022 (m)	429(w)	1674 (s)
5	[Ni(L) ₂ (H ₂ O) ₂]	1506 (s)	1014 (s)	418(m)	1683 (s)
6	[Cu(L) ₂ (H ₂ O) ₂]	1520 (s)	996 (m)	434(s)	1673 (s)
7	[Co(L) ₂]	1566 (s)	1000 (s)	-	1680 (s)
8	[Ni(L) ₂]	1582 (w)	1016 (w)	-	1674 (s)
9	[Cu(L) ₂]	1527 (s)	1024 (m)	-	1683 (s)
10	[Pd(L) (H ₂ O) ₂]	1512 (m)	991 (m)	424 (w)	1680 (s)
11	[Pd(L) ₂]	1582 (w)	1030 (w)	-	1677 (s)

Table 3: Electronic spectra and its assignment

No.	Formula	Electronic spectra	Assignment
1	[Co(L)(H ₂ O) ₃ Cl]	15667 20167	⁴ T _{1g} → ⁴ A _{2g} ⁴ T _{1g} → ⁴ T _{1g} (p)
2	[Ni(L)(H ₂ O) ₃ Cl]	11733 13024 27373	³ A _{2g} → ³ T _{2g} ³ A _{2g} → ³ T _{1g} ³ A _{2g} → ³ T _{1g} (p)
3	[Cu(L)(H ₂ O) ₃ Cl]	14233 16051	² B _{1g} → ² A _{1g} ² B _{1g} → ² E _g
4	[Co(L) ₂ (H ₂ O) ₂]	16007 20100	⁴ T _{1g} → ⁴ A _{2g} ⁴ T _{1g} → ⁴ T _{1g} (p)
5	[Ni(L) ₂ (H ₂ O) ₂]	12001,13330,25100	³ A _{2g} → ³ T _{2g} ³ A _{2g} → ³ T _{1g} ³ A _{2g} → ³ T _{1g} (p)
6	[Cu(L) ₂ (H ₂ O) ₂]	14400 16377	² B _{1g} → ² A _{1g} ² B _{1g} → ² E _g
7	[Co(L) ₂]	15313	² A _{1g} → ² E _g
8	[Ni(L) ₂]	24194 15576	¹ A _{1g} → ¹ B _{1g} ¹ A _{1g} → ¹ A _{2g}
9	[Cu(L) ₂]	15124 broad	² B _{1g} → ² A _{1g} ² B _{1g} → ² E _g
10	[Pd(L) (H ₂ O) ₂]	23818 15822	¹ A _{1g} → ¹ B _{1g} ¹ A _{1g} → ¹ A _{2g}
11	[Pd(L) ₂]	15757	¹ A _{1g} → ¹ B _{1g} ¹ A _{1g} → ¹ A _{2g}

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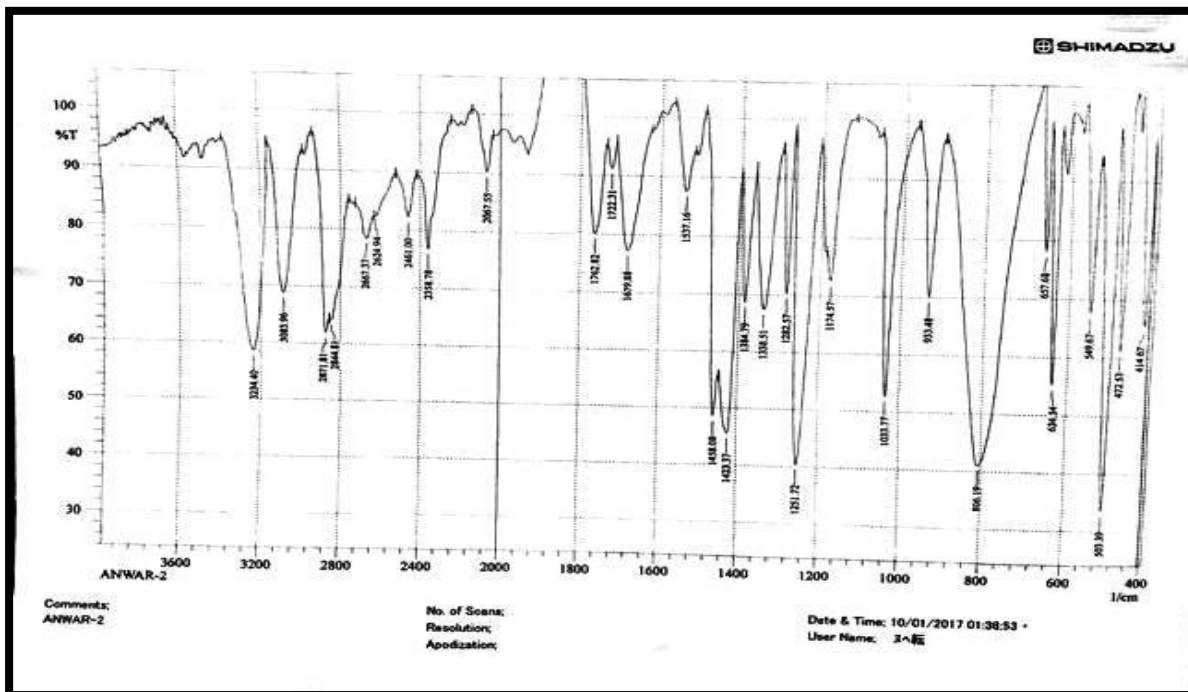


Figure 2: Infra-red spectrum of NaBADTC

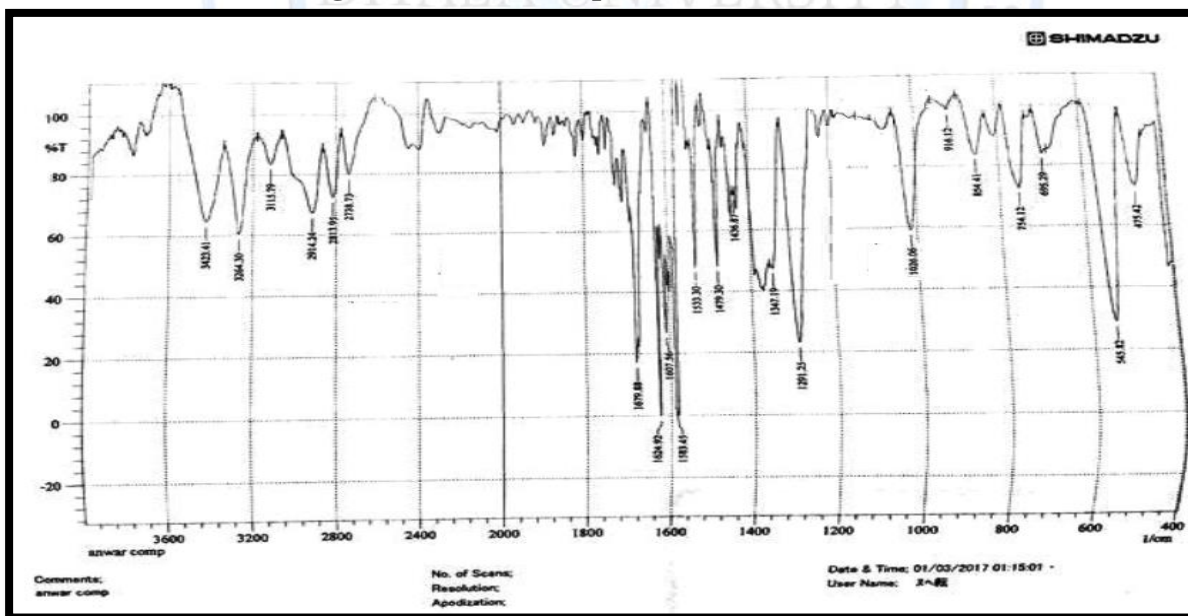


Figure 3: Infrared spectrum of [Cu(L)(H₂O)₃ Cl] complex

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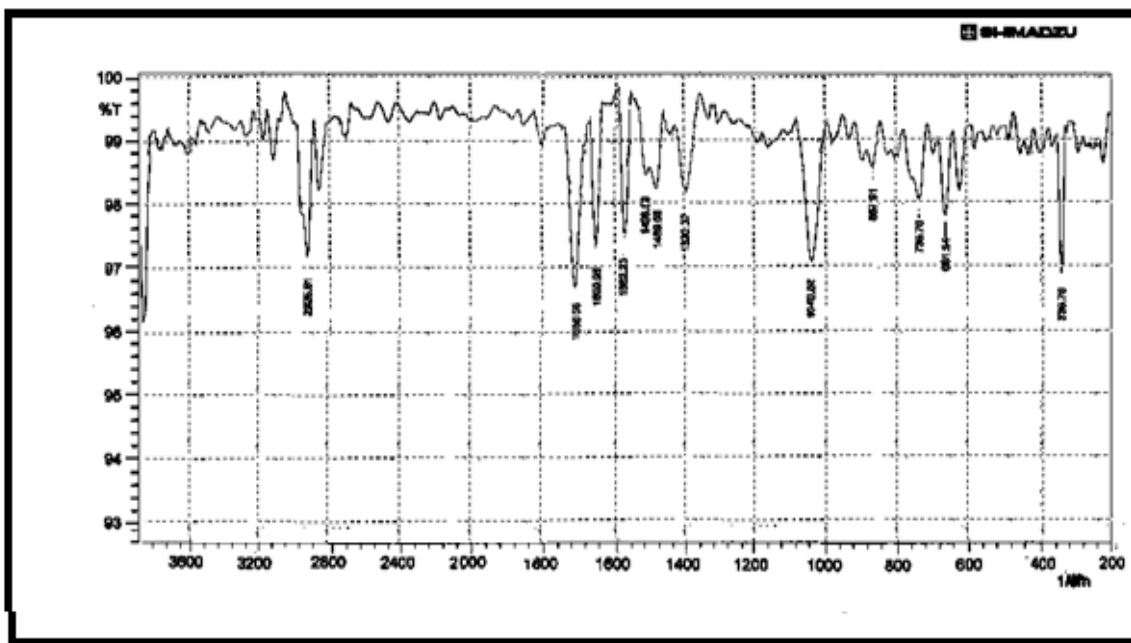


Figure 4: Infrared spectrum of [Pd(L)₂] complex

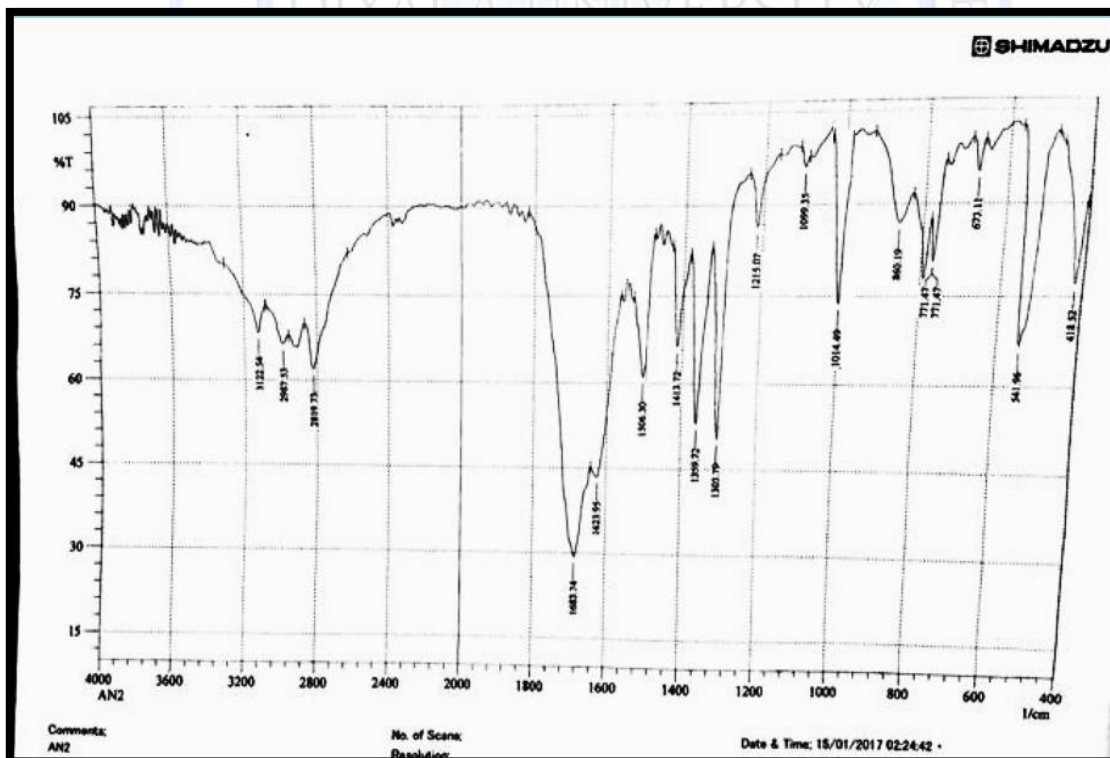
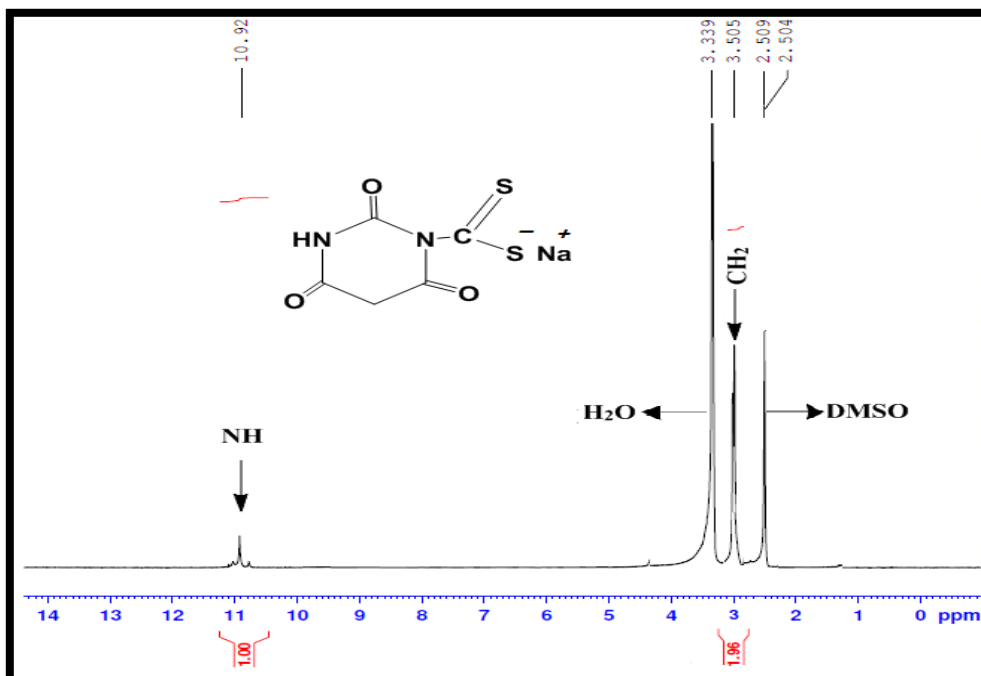
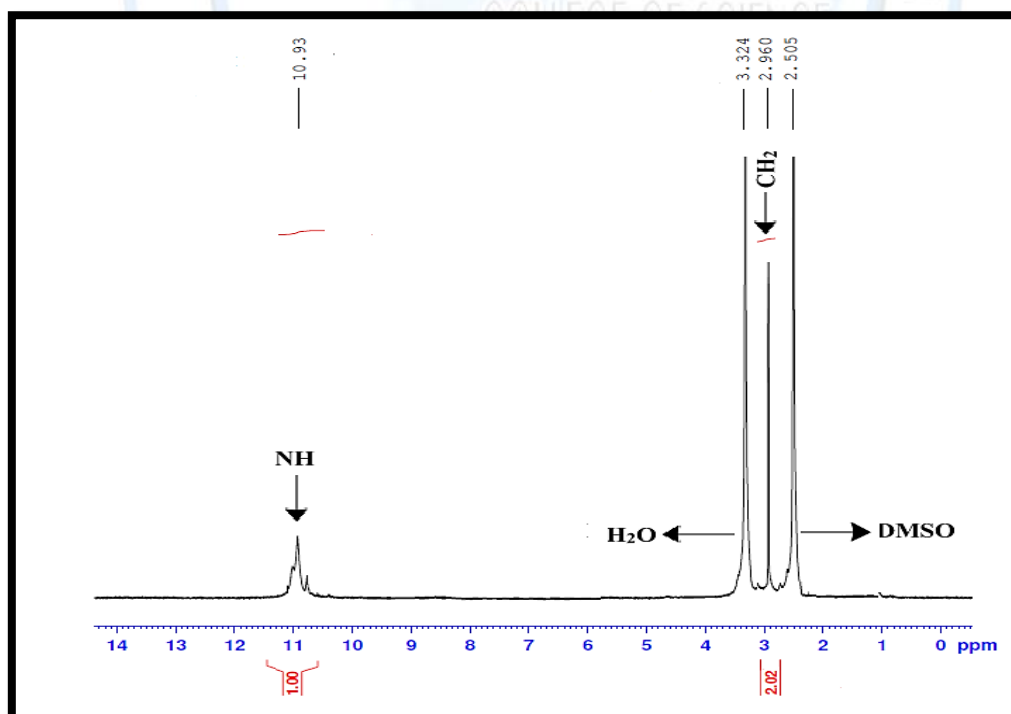


Figure 5: Infrared spectrum of [Ni(L)₂(H₂O)₂] complex

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Figure 6: ¹H- NMR Spectrum for NaBADTCFigure 7: ¹H- NMR Spectrum of [Pd(L)(H₂O)₂]Cl complex

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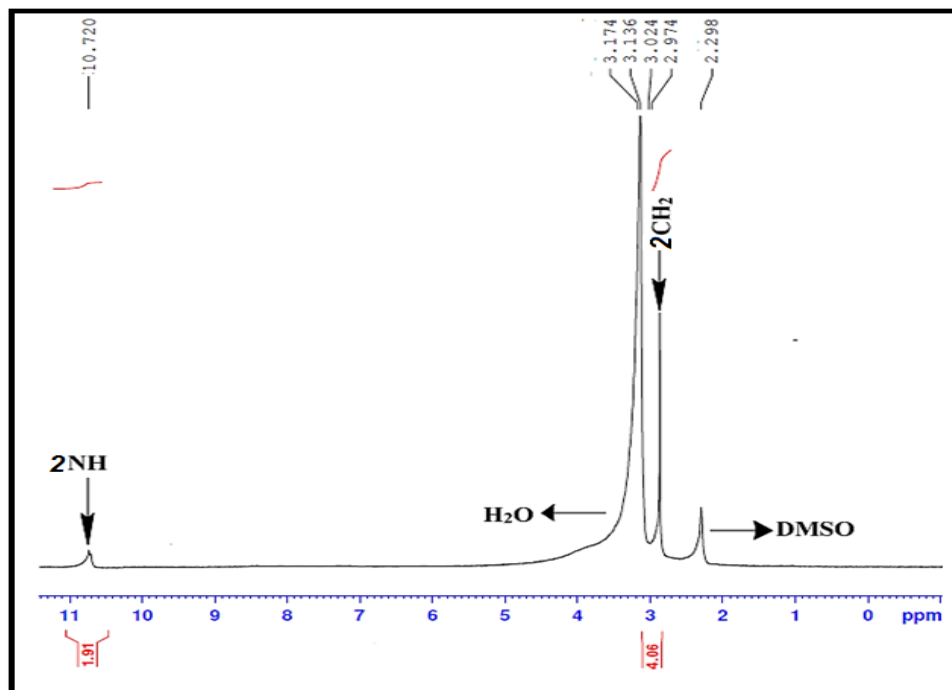


Figure 8: ^1H - NMR Spectrum of $[\text{Pd}(\text{L})_2]$ complex

Conclusion

From the previous considerations regarding the various physico-chemical, spectral and analytical studies we conclude that:

1. The ligand mono sodium 2,4,6-trioxotetrahydropyrimidine-1(2H)-carbodithioate, coordinate with metal ion, through two sulfur atoms (bidentate).
2. These complexes showed diversity in their geometry, which were octahedral geometry in the complexes of the type $[\text{M}(\text{L})(\text{H}_2\text{O})_3\text{Cl}]$, $[\text{M}(\text{L})_2(\text{H}_2\text{O})_2]$, and square planar in the palladium complexes. When $[\text{M}(\text{L})_2(\text{H}_2\text{O})_2]$ dried at 140 °C under vacuum give the complexes of the type $[\text{M}(\text{L})_2]$ which show square planar structure where $\{\text{M} = \text{Pd, Ni, Cu}\}$ whilst $[\text{Co}(\text{L})_2]$ showed a tetrahedral structure.

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Anwar Adil Hameed

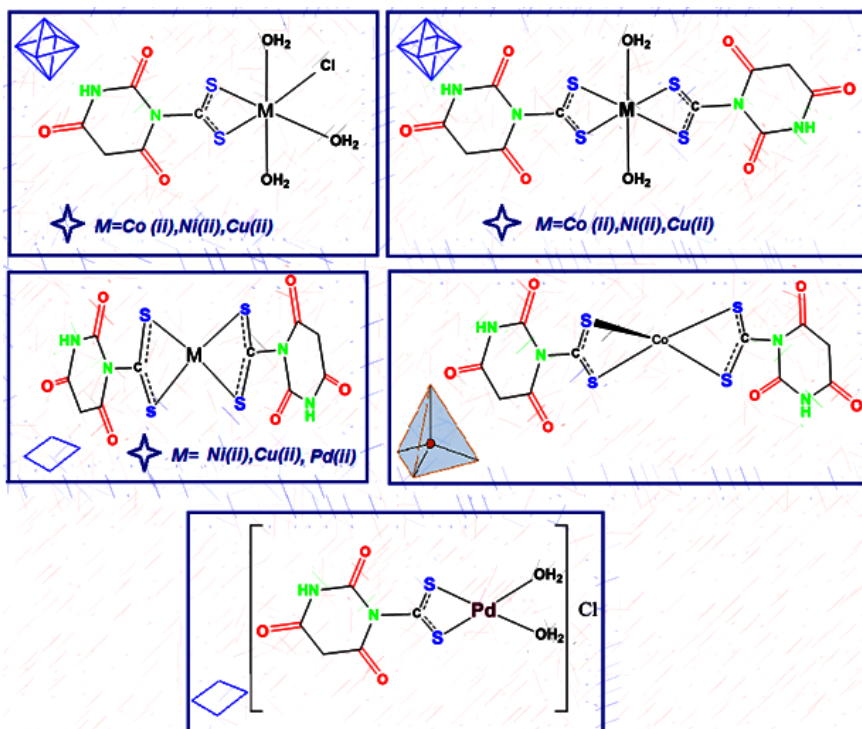


Figure 9: Configurations of the prepared complexes

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