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Synthesis and Spectroscopic Characterization of Some Transition Metal Complexes of 4-Nitro-N-(2-oxo-1,2-dihydropyrimidin-4-ylcarbamothioyl) benzamide (NPB)

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ABSTRACT

An isothiocyanate derivative was reacted with cytosine under appropriate reaction conditions to create 4-nitro-N-(2-oxo-1,2-dihydropyrimidin-4-ylcarbamothioyl)benzamide (NPB), a novel ligand. A number of analytical and spectroscopic methods, including as elemental analysis, ¹H NMR, ¹³C NMR, FT-IR, as well as UV–visible spectroscopy, were used to purify and characterize the synthesized ligand. These methods verified the ligand's functional groups and suggested molecular structure. The ligand was then reacted with various divalent metal ions to create a range of transition metal complexes. To ascertain the complexes' structural and electrical characteristics, conductivity experiments, FT-IR spectroscopy, UV-visible spectroscopy, along with magnetic susceptibility investigations were used. Through the use of suitable donor atoms, the ligand and metal ions were successfully coordinated, according to the analytical & spectral data. All complexes were isolated as stable crystalline solids exhibiting different colors depending on the metal ion involved. It was discovered that the metal-to-ligand molar ratio was 1:2, which translates to a (ligand: metal) ratio of 2:1. An octahedral geometry was suggested for the synthesized compounds based on the spectral and analytical data that were obtained. The complexes' overall molecular formula is $[M(NPB)_2Cl_2]$, where M^{2+} = Mn, Co, Ni, Cu, Zn, Cd, & Hg.

Keywords: NPB, Transition Metal Complexes, Spectroscopic Characterization, Octahedral Geometry, FT-IR/NMR/UV-Vis, Magnetic And Conductivity Studies.

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INTRODUCTION

One of the most well-known molecular structures in use today is the Watson-Crick structure of DNA. The alkali metal ion, which is connected to the N_3 & O_2 atoms of cytosine, is bidentate with every ligand in this structure. In order to enhance the chelation of the metal cation, hydrogen-bonding connections between the ligands are compromised [1]. The accumulation of somatic mutations in a variety of human malignancies, antibody diversification in adaptive immunity, & innate immunological responses to viruses and transposons are just a few of the significant biological processes that depend on the enzymatic deamination of cytosine in DNA [2-4]. Derived from pyrimidine, cytosine is a nitrogenous basic with the chemical formula $C_4H_2N_3O$ that is present in both ribonucleic acid (RNA) & deoxyribonucleic acid (DNA). An essential part of many physiologically active substances, pyrimidine is an aromatic heterocyclic molecule having nitrogen atoms at the first as well as third positions [1]. DNA base pairing in patterns other than the widely recognized Watson-Crick structure has been the subject of extensive investigation in recent years. Cytosine and guanine create three hydrogen bonds during Watson-Crick base pairing [7]. Although the cytosine tetrad has a sizable central cavity that is appropriate for cation recognition, these complexes frequently take on a non-planar form that is inappropriate

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for stacking, with the possible exception of ions with extremely large radii [2]. Furthermore, a variety of transition metal ions, such as Co(II), Ni(II), Cu(II), Zn(II), and Cd(II), have been produced and described in cytosine and thiocyanate mixed-ligand complexes [3]. Another work used spectroscopic, analytical, theoretical, and biological activity investigations to investigate new cytosine mixed-ligand complexes and related silver nanoparticles [4]. Schiff base compounds containing pyrimidines have also been reported [6]. IR & UV-visible spectroscopy, conductivity testing, chemical analysis, ^{1}H and ^{13}C NMR, and cyclic voltammetry were used to create and describe the ligand [5, 8]. The goal of this study is to create N-(2-oxo-1,2-dihydropyrimidin-4-ylcarbamothioyl)acetamide (NPB), a novel ligand, and its metal complexes with Mn^{2+}, CO^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, & Hg^{2+}.

MATRIAL AND METHODS

Experimental Chemicals

Fluka and Merck supplied metal salts such as $HgCl_2$, $MnCl_2{\cdot}4H_2O$, $CdCl_2{\cdot}H_2O$, $NiCl_2{\cdot}6H_2O$, $CoCl_2{\cdot}6H_2O$, $CuCl_2{\cdot}2H_2O$, as well as $ZnCl_2$. Cytosine, 4-nitrobenzoyl chloride, and ammonium thiocyanate were supplied by BHD, Merck, and Fluka. There was no need for further purification because all of the chemicals used were analytical grade.

Instruments

The ^{1}H and ^{13}C NMR spectra were obtained on a Varian Inova 500 MHz spectrometer (USA) at the Central Laboratory, College of Science, University of Tehran. Samples were dissolved in DMSO-d_6 as solvent, and tetramethyl silane (TMS) was used as the internal standard. An Electrothermal Stuart device (Model SMP30) at the College of Education for Pure Sciences, Ibn Al-Haitham, University of Baghdad, was used to test the melting points of the produced compounds. FT-IR spectra were captured using a Shimadzu FT-IR 3800 spectrophotometer using KBr disks. The spectra (4000–400 cm^{-1} for the ligand and 4000–200 cm^{-1} for the metal complexes) were obtained. Electronic spectra of DMSO solutions (10^{-3} M) were acquired using a Shimadzu UV-160 spectrophotometer. Utilizing a Philips PW digital conductivity meter, molar conductivity measurements were made. The synthesized ligand and several of its complexes underwent elemental analysis (C, H, N, and S) using a German Elementar Analyzer (EA1112) at the University of Tehran in Iran. A magnetic susceptibility balance (Model MSB-MK1) was used to test the magnetic susceptibility. Using a Shimadzu AA-680G equipment, atomic absorption spectroscopy was used to determine the metal contents of the complexes.

Preparation of the Ligand (NPB)

1. Preparation of 4-Nitrobenzoyl Isothiocyanate

A mixture of 4-nitrobenzoyl chloride (1.86 mL, 26 mmol) and ammonium thiocyanate (2 g, 26 mmol) diluted in 25 mL of acetone was combined. About three hours were spent stirring the reaction mixture.

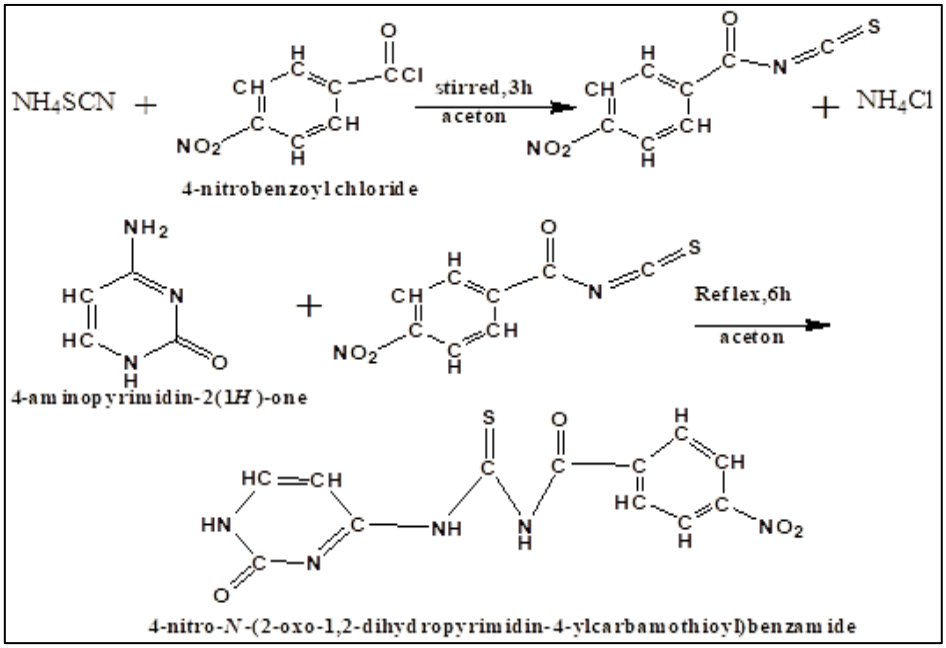
2. Preparation of 4-nitro-N-(2-oxo-1,2-dihydropyrimidin-4-ylcarbamothioyl)benzamide (NPB)

Acetone (25 mL) was used to dissolve cytosine (2.93 g, 26 mmol). Next, the cytosine solution was gradually supplemented with the previously made 4-nitrobenzoyl isothiocyanate solution. After refluxing the reaction mixture for six hours, it was allowed to dry. A 75% yield was achieved by recrystallizing the yellow product from ethanol. 276–278 °C was the melting point. Elemental analysis results were as follows:
C: found 44.47% (calcd. 45.14%), H: found 2.57% (calcd. 2.84%), N: found 21.11% (calcd. 21.93%), O: found 19.61% (calcd. 20.04%), S: found 9.73% (calcd. 10.04%). As shown in Scheme (1), the

ligand's suggested chemical formula is $C_{12}H_9N_5O_4S$.

Preparation of the Complexes

The ligand NPB (0.43 g, 2 mmol) was mixed with a solution containing 1 mmol of the appropriate metal salt ($MnCl_2·4H_2O$, $ZnCl_2$, $CoCl_2·6H_2O$, $NiCl_2·6H_2O$, $CuCl_2·2H_2O$, $CdCl_2·H_2O$, or $HgCl_2$) dissolved in 10 mL of acetone. The respective masses of the metal salts used were 0.20 g, 0.14 g, 0.24 g, 0.24 g, 0.17 g, 0.20 g, and 0.27 g. Six hours were spent stirring the reaction mixture at 25 °C. Filtration was used to gather the precipitate, which was then cleaned using a water–ethanol combination and dried.



Scheme (1): Ligand synthesis (NPB).

Table 1. Solubility of the ligand (NPB) in various solvents.

Ligand	Water	Acetone	MeOH	EtOH	DMF	DMSO
NPB	−	+	±	+	+	+

Symbols: *(+) soluble, (−) insoluble, (±) sparingly soluble.*

Table 2. The ligand (NPB) & its metal complexes' specific physicochemical characteristics and elemental analysis.

Compound	Color	Molecular Weight (g/mol)	M.P. (°C)	Yield (%)	C % Calc. (Found)	H % Calc. (Found)	N % Calc. (Found)	O % Calc. (Found)	S % Calc. (Found)	M % Calc. (Found)
$C_{12}H_9O_4N_5S$ (NPB)	Yellow	319	276–278	75	45.14 (44.47)	2.84 (2.57)	21.93 (21.11)	20.04 (19.61)	10.04 (9.73)	—
$[Mn(NPB)_2Cl_2]$	Deep pink	763.93	350 (d)	65	37.69 (37.61)	2.35 (2.03)	18.32 (18.07)	—	8.377 (8.48)	7.19 (7.13)
$[Co(NPB)_2Cl_2]$	Green	767.93	310 (d)	70	37.5	2.34	18.23	—	8.33	7.67 (7.33)
$[Ni(NPB)_2Cl_2]$	Deep yellow	767.69	365 (d)	75	37.5 (37.32)	2.34 (2.11)	18.23 (17.67)	—	8.336 (8.12)	7.64 (7.73)
$[Cu(NPB)_2Cl_2]$	Green	772.54	340 (d)	50	18.12 (18.02)	2.32 (2.11)	18.12 (17.79)	—	8.28 (7.78)	8.22 (8.02)

Compound	Color	Molecular Weight (g/mol)	M.P. (°C)	Yield (%)	C % Calc. (Found)	H % Calc. (Found)	N % Calc. (Found)	O % Calc. (Found)	S % Calc. (Found)	M % Calc. (Found)
$[Zn(NPB)_2Cl_2]$	White	774.37	385 (d)	65	37.19	2.32	18.07	—	8.264	8.44 (8.54)
$[Cd(NPB)_2Cl_2]$	White	821.40	380 (d)	80	35.06 (35.12)	2.19 (2.03)	17.04 (17.12)	—	7.791 (7.501)	13.68 (13.54)
$[Hg(NPB)_2Cl_2]$	White	909.6	395 (d)	50	31.6	1.9	15.3	—	7.036	22.05

RESULTS AND DISCUSSION

Ligand FT-IR Spectral Information (NPB)

The N–H stretching vibration is represented by a band at 3383 cm^{-1} in the FT-IR spectra of cytosine (Fig. 1), whereas the N–H_2 stretching vibration is represented by a band at 3174 cm^{-1} [7]. The ligand NPB exhibits a medium-intensity band at 3298 cm^{-1} in the FT-IR spectra (Fig. 2), which corresponds to ν(N–H). As shown in Table 3, the ν(C=S) stretching vibration is responsible for a medium-intensity band at 1215 cm^{-1} [6], whereas the amide Σ(C=O) stretching vibration is responsible for a strong band at 1666 cm^{-1}.

Table 3. Typical cytosine and ligand NPB infrared bands (cm^{-1}).

Compound	ν(N–H)	ν(N–H_2) Asym.	ν(C=O) Amide	ν(C=S)
Cytosine	3383 (m)	3174 (m)	—	—
NPB	3298 (m)	—	1666 (s)	1215 (m)

All produced metal complexes show notable changes in their FT-IR spectra when compared to the free ligand. Initially located at 1666 cm^{-1}, the ν(C=O) amide band moves by 11–134 cm^{-1} to lower frequencies in the range of 1532–1655 cm^{-1}. According to this alteration, the ligand is coordinated with the metal center via the carbonyl oxygen atom [11]. The free ligand's ν(C=S) stretching band, which was detected at 1215 cm^{-1}, moves to lower frequencies in the region of 1004–1138 cm^{-1} with shifts of 77–211 cm^{-1}, suggesting coordination through the thione group's sulfur atom. The ν(N–H) stretching bands exhibit only minor changes, appearing between 3090–3115 cm^{-1}, indicating that the amino groups do not participate in coordination [8, 12]. The formation of M–O, M–S, and M–Cl bonds in the complexes is confirmed by the appearance of new bands in the ranges 422–474 cm^{-1}, 326–391 cm^{-1}, and 232–297 cm^{-1}, respectively, as shown in Table 4 [13–16].

Table 4. Typical infrared bands (cm^{-1}) for the metal complexes of the free ligand (NPB).

Compound	ν(N–H)	ν(C=O) Amide	ν(C=S)	ν(M–O)	ν(M–S)	ν(M–Cl)
NPB	3109 (m)	1662 (s)	1215 (m)	—	—	—
$[Mn(NPB)_2Cl_2]$	3115 (m)	1624 (w)	1004 (w)	470 (w)	391 (w)	232 (w)
$[Co(NPB)_2Cl_2]$	3090 (m)	1650 (m)	1112 (w)	474 (w)	365 (m)	234 (w)
$[Ni(NPB)_2Cl_2]$	3108 (m)	1532 (m)	1110 (w)	425 (w)	370 (w)	281 (w)
$[Cu(NPB)_2Cl_2]$	3108 (m)	1574 (w)	1080 (w)	422 (w)	374 (w)	280 (w)
$[Zn(NPB)_2Cl_2]$	3109 (w)	1647 (s)	1106 (m)	426 (w)	385 (w)	287 (w)
$[Cd(NPB)_2Cl_2]$	3101 (m)	1655 (s)	1138 (m)	450 (w)	364 (w)	252 (w)
$[Hg(NPB)_2Cl_2]$	3101 (w)	1650 (s)	1102 (s)	425 (w)	326 (m)	297 (w)

Notes: *s = strong, m = medium, w = weak, "—" indicates no band observed.*

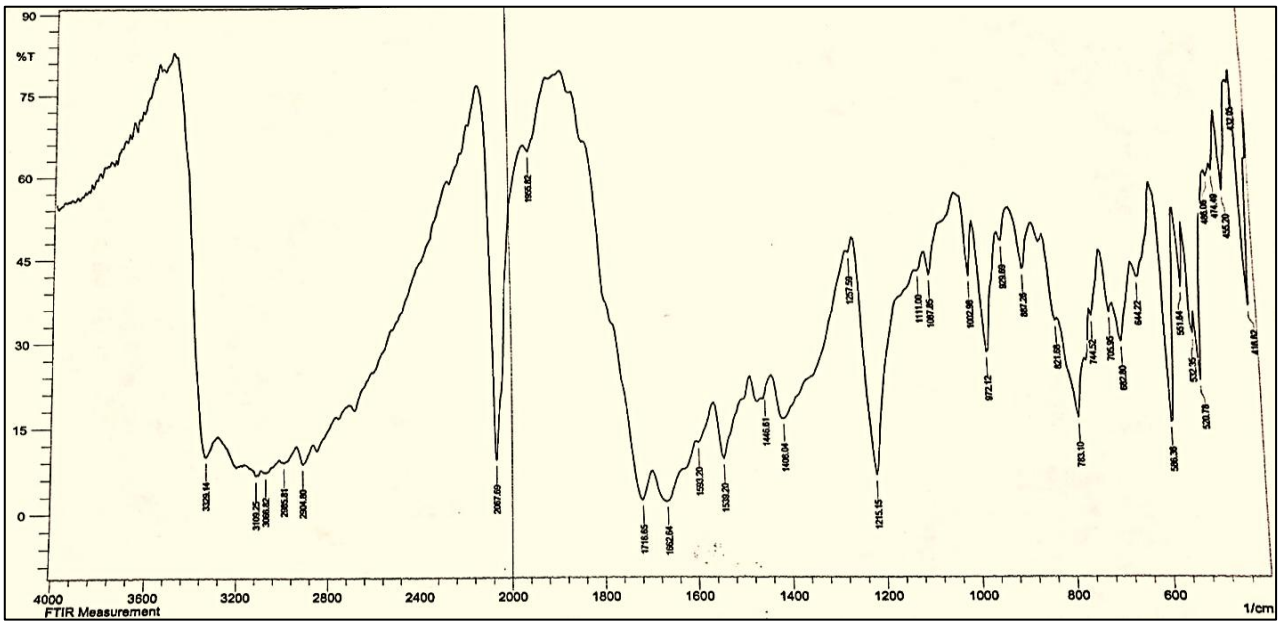


Fig. 1. FT-IR cytosine spectrum.

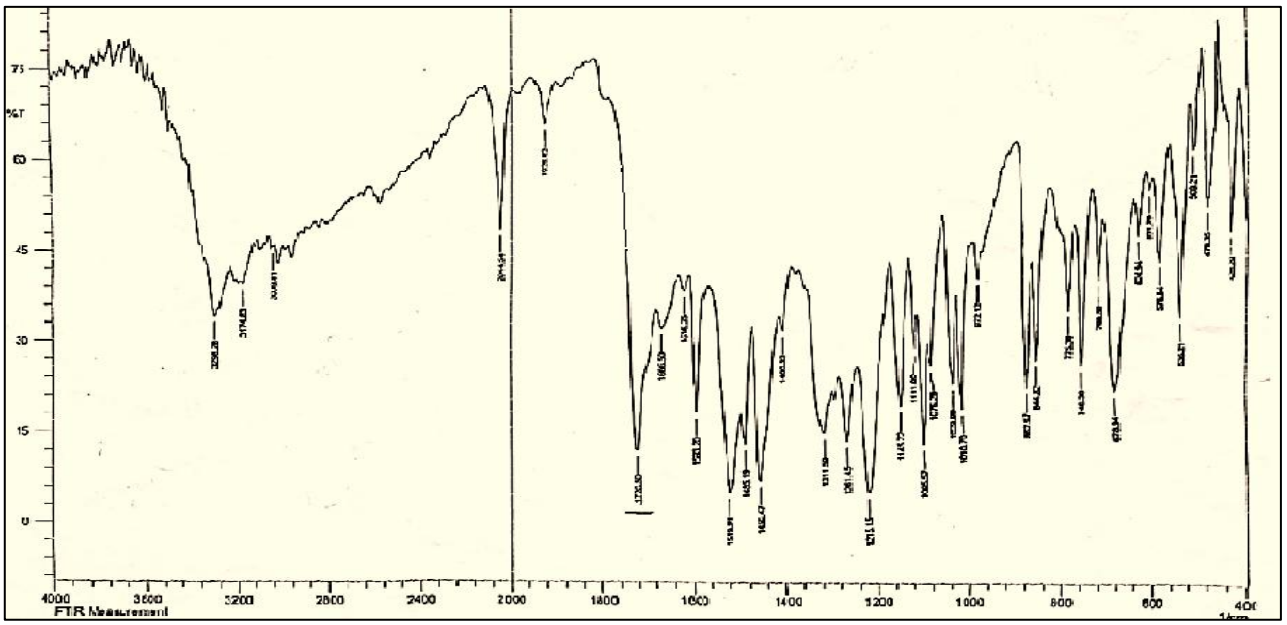


Fig. 2. The ligand NPB's FT-IR spectra.

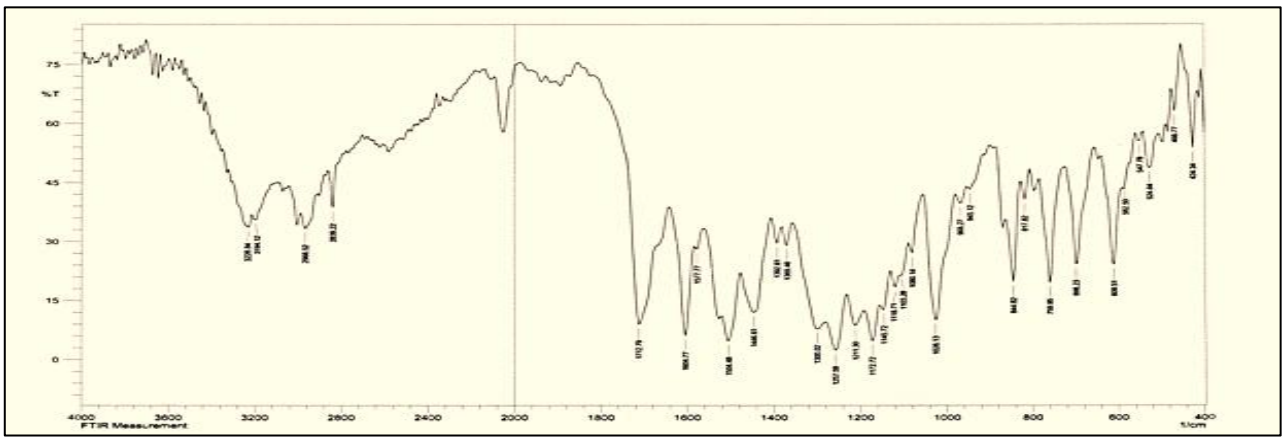


Fig. 3. The complex's FT-IR spectrum $[Mn(NPB)_2Cl_2]$.

^{1}H NMR Spectral Data of Ligand (NPB)

From the ^{1}H NMR spectrum of ligand NPB (Fig. 4), the following characteristic proton signals were observed (recorded in DMSO-d_6). The NH–CS protons were observed as a doublet at δ 4.03 ppm. The amine NH proton appeared as an upfield singlet at δ 11.24 ppm owing to its deshielded state due to extensive hydrogen bonding. The CH proton of the ethylene unit resonated as a singlet at δ 5.25 ppm. The NH protons of the secondary amide also appear as a multiple at δ 8.15–8.46 ppm. The δ 2.51 ppm signal of DMSO-d_6 residual solvent was also present, confirming the measurement solvent environment [13].

Table 5. **^{1}H NMR data in DMSO-d² for ligand NPB**.

δ (ppm)	Signal	No. of Protons	Assignment
4.03	Doublet	2H	NH–CS
11.24	Singlet	1H	NH, Amine
5.25	Singlet	1H	CH, Ethylene
12.14	Singlet	1H	NH, Secondary amide
8.15–8.46	Multiplet	1H	Aromatic CH protons

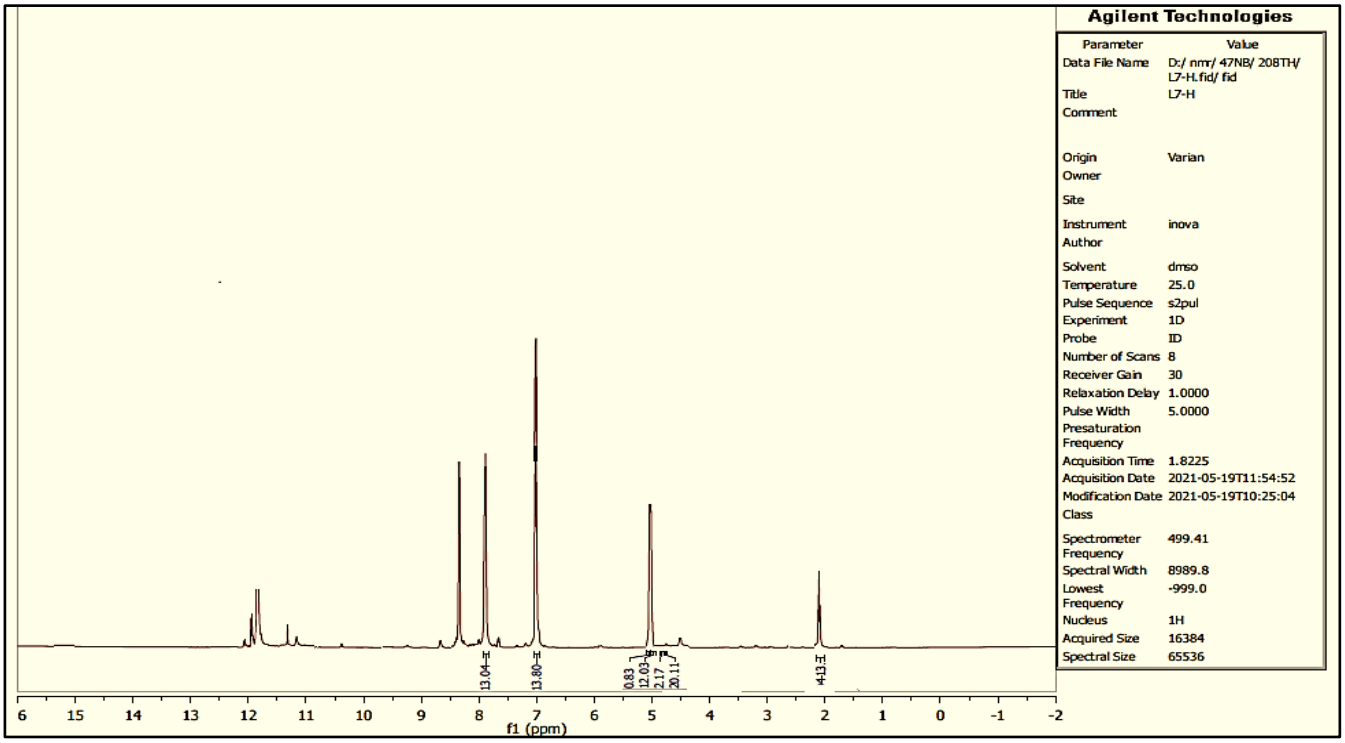


Fig. 4. The ligand NPB's ^{1}H NMR spectra was obtained in DMSO-d².

^{13}C NMR Spectral Data of Ligand (NPB)

The ligand NPB ^{13}C NMR spectrum (Fig. 5), measured in DMSO-d_6, displayed a number of diagnostic carbon resonances. The C=C carbon of the N-amide group was ascribed to a signal at δ 159 ppm. The observed peak around δ 150 ppm corresponds to the CH carbon of the ethylene group. The imine carbon (C=N) resonates around δ 161.20 ppm, confirming the development of Schiff base moiety.

The carbonyl carbon (C=O) of the amide group appeared at δ 146.71 ppm, whereas a deshielded signal corresponding to the thiocarbonyl carbon (C=S) was seen downfield at δ 175.01 ppm. For substituted aromatic systems, the carbons of the aromatic ring resonated in the δ range 118–141 ppm. As well, the DMSO-d_6 residual solvent signal was detected in the range δ 39.38–40.38 ppm confirming that it was used as a solvent during measurement.

Table 6. ^{13}C NMR data for ligand NPB in DMSO-d_6

δ (ppm)	Assignment
159	C=C (N-amide)
150	CH, Ethylene
161.20	C=N, Imine
146.71	C=O, Amide
175.01	C=S
118–141	Aromatic carbons
39.38–40.38	DMSO (residual solvent)

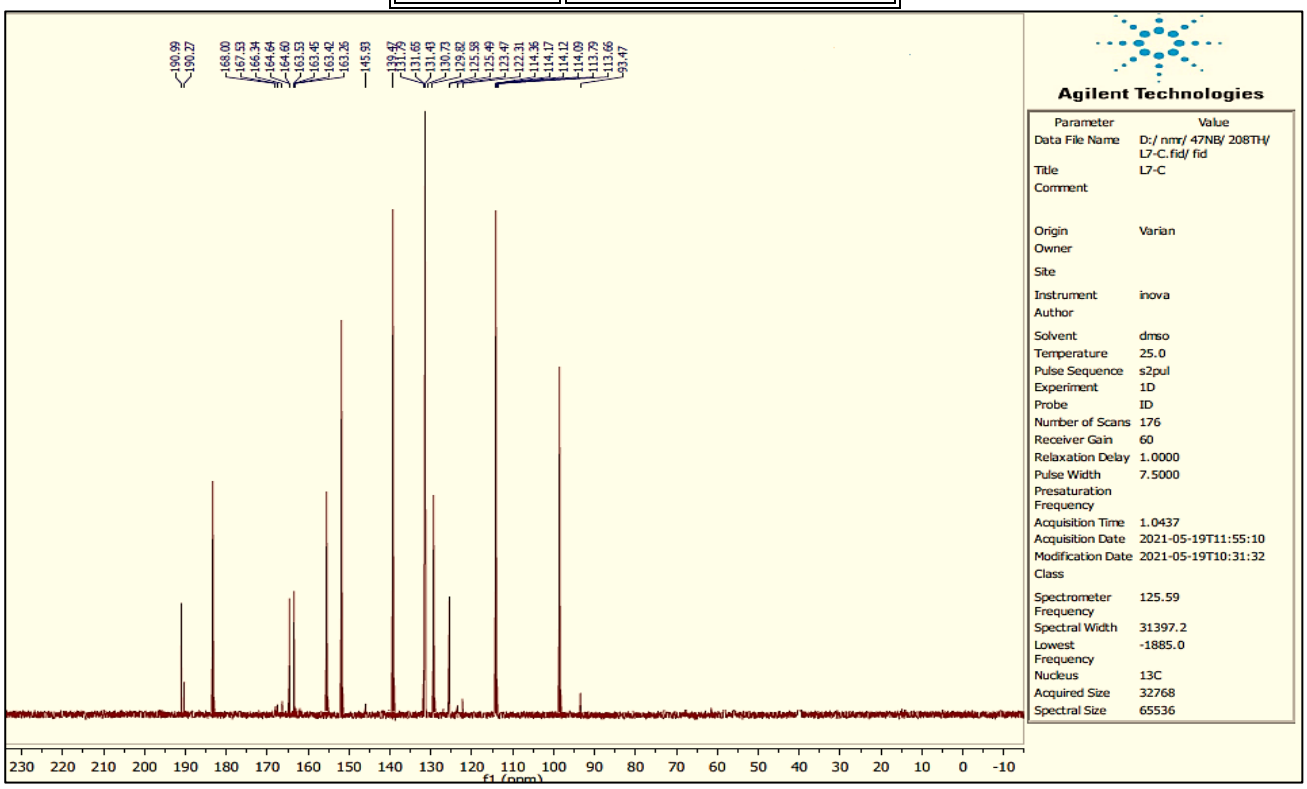


Fig. 5. The ligand NPB's ^{13}C NMR spectra was obtained in DMSO-d^2.

UV-Vis Spectral Information of Metal Complexes with Ligand (NPB)

The results of recording the UV-visible spectra of the ligand NPB and its metal complexes in DMSO (10^{-3} M) are shown in Table 6. The ligand NPB exhibits a band at 33,224 cm^{-1}, attributed to a π → π* electronic transition (Fig. 6) [10].

1. [Mn(NPB)$_2$Cl$_2$] – The deep pink Mn(II) complex shows absorption bands at 36,101 cm^{-1}, 27,855 cm^{-1}, and 11,261 cm^{-1}, which are assigned to intra-ligand transitions and ligand-field (LF) transitions: ^6A_1g → $^4T_2g(G)$ and ^6A_1g → $^4T_1g(G)$, respectively [18].

2. [Co(NPB)$_2$Cl$_2$] – The green Co(II) complex displays four bands at 32,679 cm^{-1}, 26,187

cm^{-1}, 17,543 cm^{-1}, and 10,373 cm^{-1}, corresponding to intra-ligand transitions and ligand-field transitions: $^4T_1g(F) \rightarrow {}^4T_1g(P)$, $^4T_1g(F) \rightarrow {}^4A_2g$, and $^4T_1g(F) \rightarrow {}^4T_2g(F)$. In accordance with an octahedral geometry for Co(II), the nephelauxetic ratio $\beta = B^- / B^0 = 0.865$ and the Racah electronic repulsion parameter (B^-) were determined to be 840 cm^{-1} [4].

3. $[Ni(NPB)_2Cl_2]$ – The deep yellow Ni(II) complex (Fig. 7) shows bands at 36,630 cm^{-1}, 32,786 cm^{-1}, 27,932 cm^{-1}, 18,518 cm^{-1}, and 11,627 cm^{-1}, corresponding to a combination of ligand-field and charge-transfer transitions: $^3A_2g \rightarrow {}^3T_1g(P)$, $^3A_2g \rightarrow {}^3T_1g(F)$, and $^3A_2g \rightarrow {}^3T_2g(F)$. The B^- value was determined as 771 cm^{-1}, giving a nephelauxetic ratio $\beta = 0.74$ [9].

4. $[Cu(NPB)_2Cl_2]$ – The green Cu(II) complex (Fig. 8) exhibits two bands at 33,670 cm^{-1} and 12,376 cm^{-1}, assigned to ligand-field transitions: $^2E\_g \rightarrow {}^2T_2g$, consistent with a distorted octahedral geometry for Cu(II) [6].

5. $[Zn(NPB)_2Cl_2]$, $[Cd(NPB)_2Cl_2]$, and $[Hg(NPB)_2Cl_2]$ – White complexes of Zn(II), Cd(II), and Hg(II) only show ligand-to-metal charge-transfer (LMCT) bands in the range of 32,258–32,786 cm^{-1} [17,3].

Table 6 provides a summary of all electronic transitions along with their assignments.

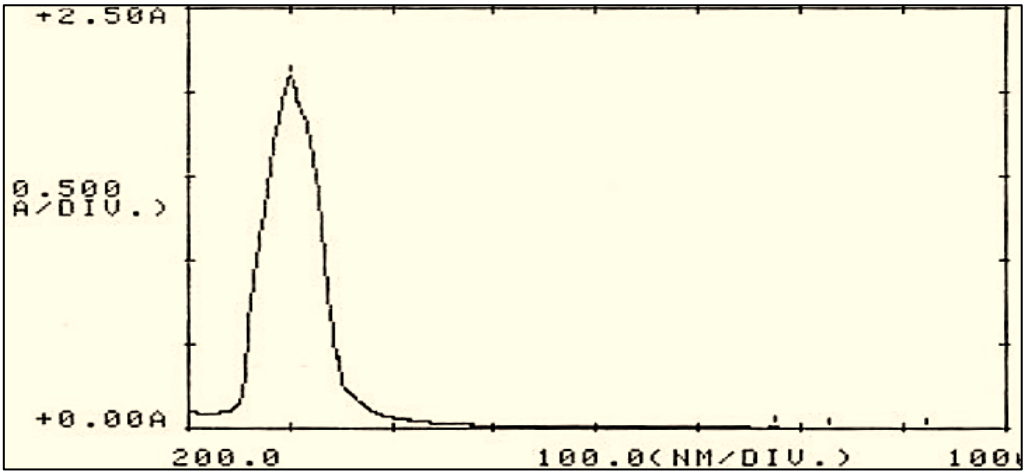


Fig. 6. The ligand NPB's electronic (UV-Vis) spectra was captured in DMSO (10^{-3} M).

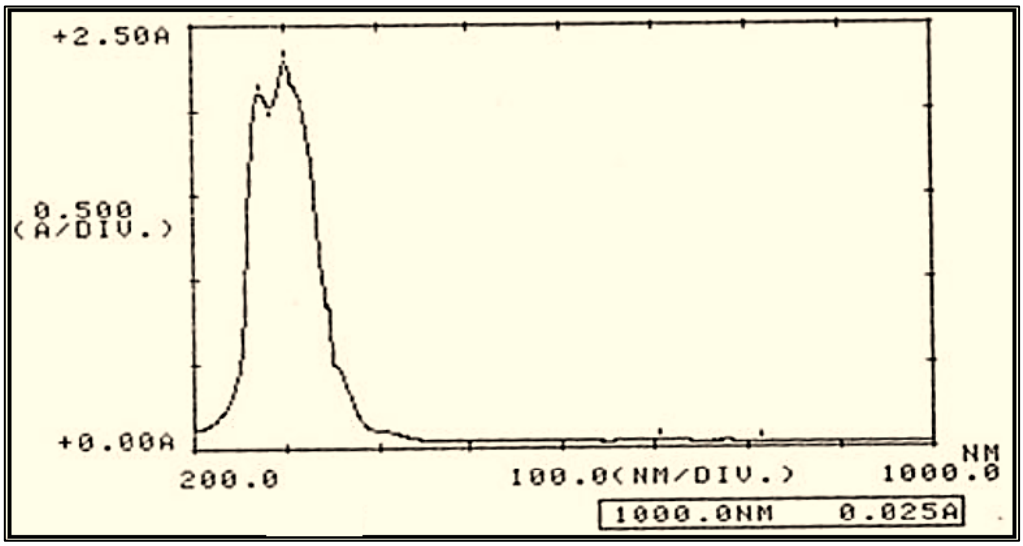


Fig. 7. The compound $[Ni(NPB)_2Cl_2]$'s electronic (UV-Vis) spectra was captured in DMSO (10^{-3} M).

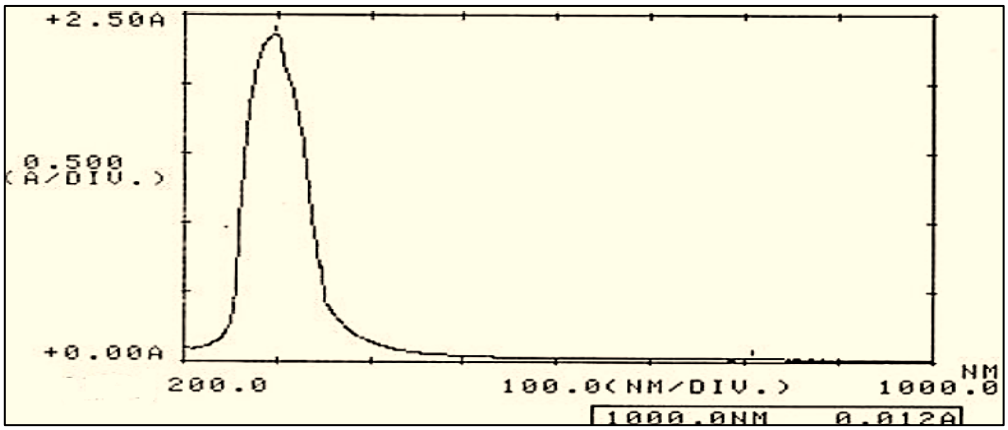


Fig. 8. The compound [Cu(NPB)₂Cl₂]'s electronic (UV-Vis) spectra was captured in DMSO (10^{-3} M).

Table 7. The ligand's (NPB) metal complexes' electronic spectra in DMSO.

Compound	λ (nm)	ν (cm^{-1})	ε_{max} (M^{-1} cm^{-1})	Type of Transition	Assignment
NPB	301	33,222	2104	π → π*	—
[Mn(NPB)₂Cl₂]	277, 359, 888	36,101, 27,855, 11,261	2230, 322, 30	Intra-ligand / LF	$^6A_1g \rightarrow {}^4T_2g(G)$, $^6A_1g \rightarrow {}^4T_1g(G)$
[Co(NPB)₂Cl₂]	306, 382, 570, 964	32,679, 26,187, 17,543, 10,373	1752, 42, 18, 15	Intra-ligand / LF	$^4T_1g(F) \rightarrow {}^4T_1g(P)$, $^4T_1g(F) \rightarrow {}^4A_2g$, $^4T_1g(F) \rightarrow {}^4T_2g(F)$
[Ni(NPB)₂Cl₂]	269, 330, 370, 530, 910	37,174, 30,303, 27,027, 18,867, 10,989	1352, 1819, 1800, 20	π → π*, C.T / LF	$^3A_2g \rightarrow {}^3T_1g(P)$, $^3A_2g \rightarrow {}^3T_1g(F)$, $^3A_2g \rightarrow {}^3T_2g(F)$
[Cu(NPB)₂Cl₂]	297, 360, 808	33,670, 27,777, 12,376	2364, 450, 30	Intra-ligand / LF / C.T	$^2E\_g \rightarrow {}^2T_2g$
[Zn(NPB)₂Cl₂]	310	32,250	1855	Intra-ligand	—
[Cd(NPB)₂Cl₂]	308	32,477	1322	Intra-ligand	—
[Hg(NPB)₂Cl₂]	305	32,756	2118	Intra-ligand	—

Notes: LF = Ligand Field; C.T = Charge Transfer.

Magnetic Properties

The magnetic moments (μ_{eff}) of the metal complexes were measured as follows:

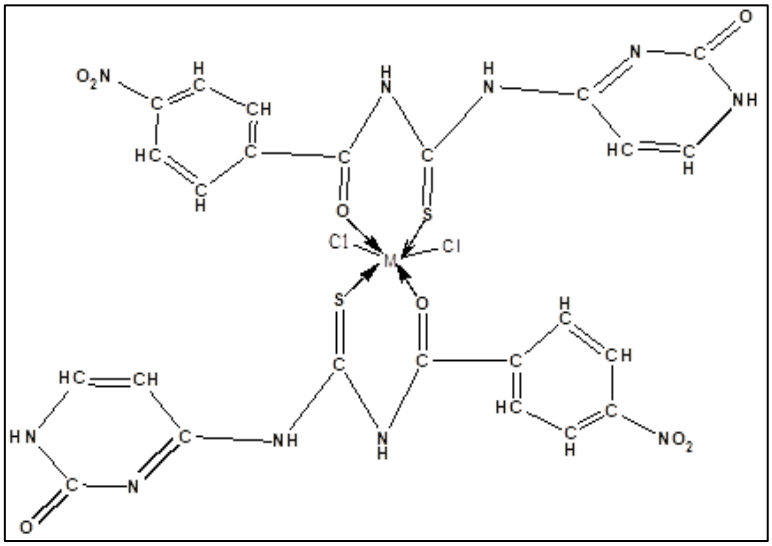
- [Mn(NPB)₂Cl₂] (Mn^{2+}, d^5): 5.81 B.M. (spin-only value expected).
- [Co(NPB)₂Cl₂] (Co^{2+}, d^7): 4.92 B.M. (consistent with octahedral geometry).
- [Ni(NPB)₂Cl₂] (Ni^{2+}, d^8): 2.85 B.M. (greater value as a result of orbital contribution).
- [Cu(NPB)₂Cl₂] (Cu^{2+}, d^9): 1.71 B.M. (consistent with one unpaired electron).
- [Zn(NPB)₂Cl₂], [Cd(NPB)₂Cl₂], [Hg(NPB)₂Cl₂]: Diamagnetic [15].

Table 8. The ligand NPB and its complexes' molar conductivity & magnetic moment in DMSO**.**

Compound	Molar Conductivity ($S \cdot cm^2 \cdot mol^{-1}$)	μ_{eff} (B.M.)
NPB	1.11	—
[Mn(NPB)₂Cl₂]	5.80	5.81
[Co(NPB)₂Cl₂]	5.44	4.92
[Ni(NPB)₂Cl₂]	3.17	2.85

Compound	Molar Conductivity ($S \cdot cm^2 \cdot mol^{-1}$)	μ_{eff} (B.M.)
$[Cu(NPB)_2Cl_2]$	3.78	1.71
$[Zn(NPB)_2Cl_2]$	11.99	Diamagnetic
$[Cd(NPB)_2Cl_2]$	10.22	Diamagnetic
$[Hg(NPB)_2Cl_2]$	12.50	Diamagnetic

Note: *All complexes act as non-electrolytes in DMSO, according to the low molar conductivity values [18].*



Scheme 2. The complexes $[M(NPB)_2Cl_2]$ have a proposed generic shape.

CONCLUSION

In this work, cytosine and 4-nitrobenzoyl chloride were reacted to create a novel ligand called NPB. A variety of spectroscopic and analytical methods, including as FT-IR, UV-Vis spectroscopy, elemental analysis for carbon, hydrogen, nitrogen, & sulfur (C, H, N, S)a, and proton and carbon-13 nuclear magnetic resonance (^1H and ^{13}C NMR) spectroscopy, were used to thoroughly describe the ligand. After the ligand characterization, a series of metal complexes of NPB were synthesized and systematically studied. Molar conductivity measurements to study the electrolytic behaviour, magnetic susceptibility studies to investigate their magnetic properties and FT-IR and UV-Vis spectroscopic analyses for coordination behavior and electronic transitions were used in the examination of these complexes. As a result, based on the combined data of spectroscopic studies and magnetic information these resulting metal complexes are proposed to have octahedral geometry around central metals ions.

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