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Synthesis, Characterization and Study of the Biological Activity of New Metal Complexes of Ligand (4-nitrobenzoyl) Carbamothioyl) Cysteine (NCC)

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ABSTRACT

Based on a reaction of 4-nitrobenzoyl isothiocyanate with the amino acid cysteine, we successfully synthesized a new ligand-(4-nitrobenzoyl)carbonothioyl cysteine (NCC) under further controlled experimental conditions. The synthesized ligand was characterized using various analytical and spectroscopic techniques including elemental analysis, proton nuclear magnetic resonance (¹H-NMR), Fourier transform Infrared spectroscopy (FTIR) and UV–Visible spectroscopy that confirmed formation of the desired compound as well as presence of functional groups and structural characteristics. As a result, transition metal(II) complexes with NCC ligand were prepared and characterized. To determine the mole ratio and coordination behaviour, prepared complexes were characterized using molar conductivity measurements, FTIR spectra, UV. Vis spectroscopy and magnetic susceptibility studies to examine structural geometry. The data suggested that the coordination occurred with a ligand-to-metal molar ratio of 2:1. All of the synthesized complexes were obtained in good yield as stable colored crystalline solids. While most complexes showed tetrahedral geometries as per spectral and magnetic data, the Cu(II) complex confirmed square planar geometry.

Keywords: 4-nitrobenzoyl isothicyanate , Cysteine , Meta complexes.

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INTRODUCTION

Cysteine is widely used in food, cosmetics, and pharmaceutical products. It is frequently used as a dough conditioner to break down gluten in baked goods, as a flavor enhancer and a precursor in some dietary supplements, as a radical scavenger in cosmetics, and as an expectorant in cough medicines. Cysteine deficiencies have been linked to skin lesions, drowsiness, hair loss, slow growth, liver damage, and patients with HIV [1,2]. Several compounds were synthesized from Cysteine and imported into medicine and industry. In 2009, Semiha Cakir et al. a new cysteine analogue, 2R)-2-(1,1-dioxo-1,2-dihydro-1λ6-benzo[d]isothiazol-3-ylideneamino)-3-mercaptopropionic acid L-cysteine hydrochloride, was obtained by electrochemically converting L-cysteine hydrochloride using electrophile sodium saccharin. The product was obtained from reactions at a 1:1 mole ratio Recently, Nebojsa D. Pantelic and co-workers [3] synthesized new Sn(IV) complexes mixing an equimolar amount of the ligands N-acetyl-S-(3,4-dihydro-3,4-dioxo-1-naphthyl)cysteine (1,2-NQC) or N-acetyl-S-(1,4-dihydro-1,4-dioxo-

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2-naphthyl)cysteine (1,4-NQC) with triphenyltin(IV) chloride (Ph_3SnCl). The complexes were characterized by mass spectrometry, IR, ^1H NMR, ^{13}C NMR and CHNS elemental analysis [4]. Using the fundamental structural formula for compounds as [M(NCC)2], where (M^{+2} = Mn, Co, Ni, Cu, Zn Cd & Hg), the study aims to create novel complexes with ligand (ATC).

MATERIAL AND METHODS

Experimental Chemicals

Metal salts ($HgCl_2$, $MnCl_2.4H_2O$, $CdCl_2.H_2O$, $NiCl_2.6H_2O$, $CoCl_2.6H_2O$, $CuCl_2.2H_2O$, and ZnCl2) where purchased from Fluka and Merck, Acetyal chloride, ammonium thiocyanate and cystein amino acid.

Instrumentations

The recordings of the ^1H NMR spectra were done using a Varian spectrometer 500 MHz made in the USA. Solvent (DMSO-d_6) and internal standard (Tetramethyl silane (TMS)) were both used for the zero chemical shift. The melting points of the synthesized compounds were measured using a Stuart melting point apparatus. The FTIR spectra were obtained on a Shimadzu 3800 spectrophotometer in the range of 4000–400 cm^{-1} using KBr discs for detecting functional groups present in ligands and complexes. The electronic absorption spectra were also registered in DMSO solutions ($C=10^{-3}M$) at 25°C using the Shimadzu UV-160 spectrophotometer. In addition, elemental analyses (C.H.N.S.) for synthesized ligands and some of their complexes were performed at University of Kashan using Elementary Germany 3000A analyzer. Electrical conductivity was measured using a Philips PW digital conductivity meter. Magnetic susceptibility measurements (via Balance magnetic susceptibility model MSB-MKI) and atomic absorption determinations of complex-associated metal contents (via Shimadzu [AA680G]) were both performed on the whole solid materials.

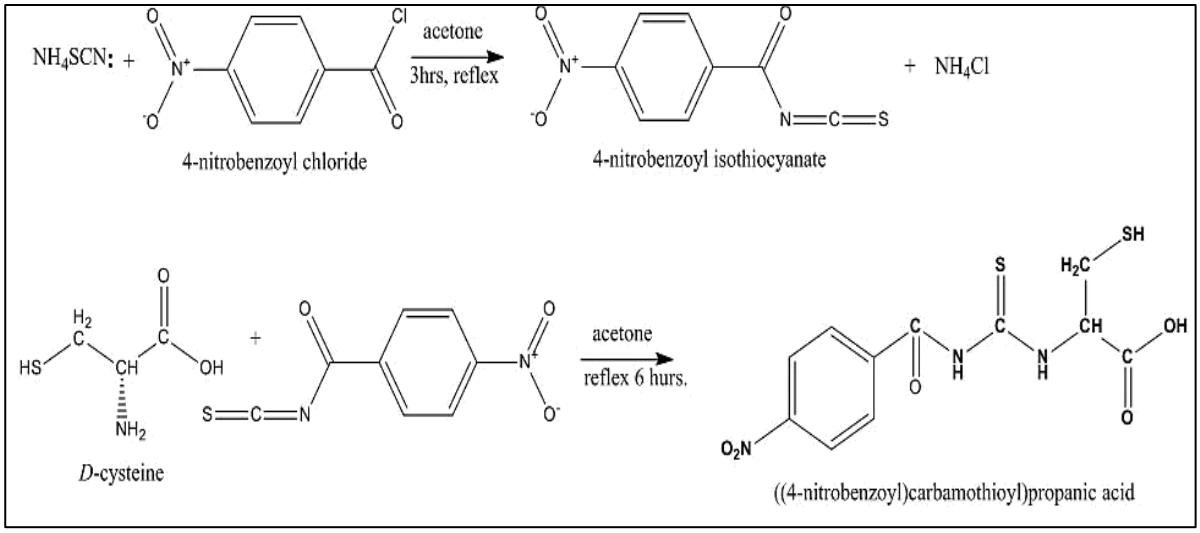
Preparation of the ligand (NCC)

1-preparation of the 4-Nitro isothiocyanate

This reaction mixture contained 4-nitrobenzoyl chloride (1.86 g, 26 mmol) in acetone (25 mL). Ammonium thiocyanate (2 g; 26 mmol) was dissolved in a separate flask containing 25 mL of acetone. These two solutions were then combined together and stirred for three hours at room temperature to allow complete reaction to occur, resulting in the formation of this intermediate compound. Upon completion of the reaction, the mixture was filtered to eliminate precipitated impurities and confirmed through spectroscopic methods that the resulting filtrate could be employed in further synthesis reactions.

2- Preparation of 3-sulfhydryl-2-(4-nitrobenzoylthioureido) propanoic acid

Cysteine amino acid (3.15 g, 26 mmol) was dissolved in 25 mL of acetone with the vigorous stirring to ensure complete dissolution. The batch was then added to the reaction mixture dropwise and refluxed under stirring for six hours until the complete formation of the desired product. Upon completion of the reaction, this solid precipitate was filtered and washed three times with acetone in order to remove any unreacted materials and/or impurities. The ligand was purified from the reaction mixture and recrystallized in ethanol to yield the pure ligand crystals. The melting point of the synthesized compound is in the range of 168–170 °C, which indicates its good purity and successful preparation. The yield was 75%. Elemental analysis showed: %C found (41.89) compared with calculated (40.64), %H found (4.15) compared with calculated (4.13), %N found (13.32) compared with calculated (13.45), %O found(20.29) compared with calculated (20.33) and %S found (20.33.) compared with calculated (20.36).



Scheme (1): The proposed ligand synthesis mechanism (NCC).

Preparation of the complexes

A solution of 0.444 g (2 mmol) of the ligand (NCC) in10 mL ethanol containing 0.112 g (2 mmol) KOH Prepared (10 mL) solutions of $MnCl_2·4H_2O$, $ZnCl_2$ $CoCl_2·6H_2O$, $NiCl_2·6H_2O$, $CuCl_2·2H_2O$ $CdCl_2·H_2O$ and HgCl 2; each solution contained the equivalent to 1 mmol of these metal salts from their individual masses: 0.200 g 0.136 g 0.237 g 0.237 g 0.170 g based on the above–for those which are solids previously prepared from appropriate mass amount listed below for others ranging between this quantity provided here and referenced add to where here though soluble respective hydrated ions prior to collection orientated dodecanoyl anions survived excess ethylenediamine coordinating initial active centers. The pH was adjusted to 7–8. After the dropwise addition of 10 mL ethanol to the mixture, a precipitate was produced instantly. For three hours, the reaction mixture was agitated at room temperature. The resulting precipitate was then filtered, cleaned with ethanol and water, and dried.

RESULTS AND DISCUSSIONS

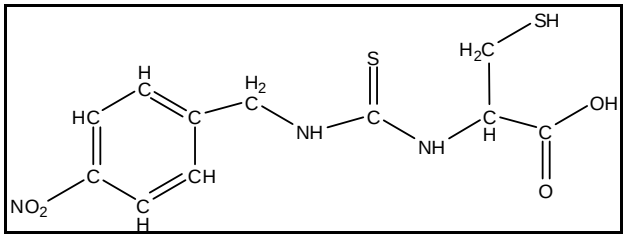
The molar conductivity values for the ligand (NCC) while others of its metal complexes in DMSO solvent are shown in Table (1). This indicates that these complexes are non-electrolytes.

Spectral studies

The ligand's NMR spectrum (NCC)

A-1H-NMR spectrum

The following signals were shown by the (1H-NMR) spectra associated with ligand (NCC) in Scheme (2):



Scheme (2): The ligand's structure (NCC).

In the ^{1}H NMR spectrum, the signal at δ 2.50 ppm was assigned to the residual solvent protons of dimethyl sulfoxide (DMSO-d_6). Methylene protons (2H, CH_2) were found at δ 2.8–3.1 ppm as doublet signals. The second signal observed as a triplet at δ 3.43 ppm was one which corresponds to proton of methine group (1H, CH). Moreover, downfield a singlet peak at δ 7.21 ppm was attributed to the amide proton (1H, NH). Additionally, new as two doublets of aromatic protons at δ 8.25–8.34 ppm attributed to four aromatic protons from synthesized compound.

In addition, the carboxylic proton (1H, COOH) assigned to peak δ 9.73 ppm appears as a singlet and the amide (1H, NH) appears at peak δ 11.6 ppm also as a singlet with very great change due to resonance in this group. The chemical shift for ligand (NCC) is displayed in Table 2 (values in ppm).

B- The (13C-NMR) spectrum of (NCC).

The following signals were displayed in the ligand (NCC) (13C, NMR) spectra in Figure (3).

The spectrum revealed signals between (CH2) and the signal at δ (25.67 ppm).

The solvent dimethyl sulfoxide (DMSO-d6) was identified as the source of the signals in the spectrum between δ 39.36 and 40.37 ppm. A signal at δ 61.50 ppm appeared as singlet assigned to (CH) one other signal was also founded in position δ 72.37 ppm which attributed to (CH). A signal (COOH) at δ 169.96 ppm was persisted.

The ligand has ^{13}C NMR spectrum showed a few singlet peaks in the δ range of 124.07150.39 ppm, which were given to aromatic carbon (4C, aromatic) on the benzol ring. Whereas a unique singlet peak at δ 166.2 ppm was assigned to the amide carbonyl carbon (C=O amide), confirming that an amide linkage formed in the ligand structure. Furthermore, the carboxylic acid carbonyl (COOH) emerges as a characteristic singlet signal at δ 172.4 ppm, confirming that the cysteine moiety is included in synthesized compound. Moreover, the thiocarbonyl group (C=S) was detected by a characteristic singlet peak found at δ 182 ppm for the carbon bonded to sulfur in thioamide. The values of these δ, are indicative of the synthesis and structural characterization of ligand (NCC). Full chemical shift values are provided in Table 3.

Infrared spectra

Table (4) shows the recorded vibrational characteristics of prepared compounds with KBr discs FTIR spectrum of NCC, shown in Fig.(4) had an absorption band of medium intensity at 3420 cm^{-1}, which was assigned to the stretching vibration of ν(N–H). A self-explanatory hydroxyl stretching vibration ν(O–H) band was attributed at 3178 cm−1 as another medium band. Moreover, a weak absorption band in the sulfur region at 2550 cm^{-1} was that assigned to stretching vibration of thiol group ν(S–H), which indicated the existence of sulfur functionality within the ligand.

The ν(C=S) stretching vibration resulted in a medium-intensity band which was appeared in the rhythm. The intense absorption band at 1630 cm^{-1} is regarded as the ν(C=O) stretching vibration. Two bands at 1720 cm^{-1} and 1345 cm^{-1} were respectively assigned to the asymmetric ν(COO)asym and symmetric ν(COO)sym stretching vibrations.

FTIR spectra of produced compounds at (1235cm^{-1}) [5,6] revealed υ(N-H) in the range (3460-3420cm^{-1}), indicating a shift to low frequencies in comparison to the indicated free ligand.

The band that corresponds to the stretching vibration of thiol, ν(S–H), was found in the range of 2558–2588 cm^{-1} and was also slightly displaced due to possible interaction with the nitrogen of the ligand's amine group with the metal ion [7]. Absorption band corresponding to ν(COO)sym also emerged in the 1410–1425 cm^{-1} region, shifted up by approximately 60–

65 cm^{-1}. On the other hand, the band that results from υ(COO)asym emerging at rang (1600-1645 cm^{-1}) has been moving to low frequencies through (120-75 cm^{-1}), referring to The metal ion was joined to the carboxylic group [8]. Additionally, the carbonyl group's stretching vibration bands of ν(C=S) and Σ(C=O) showed either no change or only a tiny shift in frequency (within ranges 1226–1242 cm^{-1} & 1600–1627 cm^{-1}, respectively), indicating that these groups are not coordinated with a metal ion [9]. Stronger stretching vibration bands were revealed for ν(M–N) and ν(M–O), detected within 428–462 cm^{-1} and 455–482 cm^{-1} respectively. FTIR spectrum of complex [Mn(NCC)2] is shown in Figure 5.

The metal complexes' magnetic characteristics

Using the appropriate approach, the effective magnetic moments (μeff in B.M.) for the Mn^{2+} (d^1) and Co^{2+}(d,7) complexes were found to be 5.92,4.88, with spin-only values expected [10]. In the case of Ni^{2+} (d^8), a high μeff value of 3.44 B.M., could originate from orbital contributions [11].

This finding supported the presence of a single unpaired electron in Cu^{2+} (d^9) complex [12], since the μ_{eff} value for 1 unpaired electron is expected to be close to 1.74 B.M. Table (1) shows all of the data.

The electronic spectra

Electronic spectra of the ligand (NCC) in Fig.6. The compound yellow colour and two characteristic absorption bands at4386 cm^{-1} and 26469 cm^{-1}. The π→π* and n→π* electronic transitions were assigned to these bands, respectively [13]. The observed high energy band at 36387 cm^{-1} is assigned to transitions of electrons in the aromatic system and conjugated bonds of the ligand, whilst the low energy band at 26469 cm^{-1} was attributed to excitation of nonbonding electrons related to heteroatoms (O, N, and S) that exist in its ligand structure. The spectral features support the proposed structure of ligands synthesized.

[Mn(NCC)2] complex

In its electronic spectrum, the deep yellow Mn^{2+} complex showed several characteristic absorption bands. A band with a high energy of 36480 cm^{-1} was assigned to ligand-field transitions, while the band at 28735 cm^{-1} was assigned to charge-transfer (CT) transitions between the ligand and metal ion. Moreover, a less energetic band located at 13298 cm^{-1} was attributed to the electronic transition $^6A_1 \rightarrow {}^4T_2(G)$ [14], which is commonly observed for Mn^{2+} complexes. These spectral transitions indicate the coordination of the ligand to manganese and confirm the geometrical structure of the complex.

[Co(NCC)2] complex

ESI-MS identification of the species produced The Co^{2+} complex was deep blackish-green colour and revealed four typical absorption bands in its electronic spectrum. Band one was at 36103 cm^{-1} and band two at 27029 cm^{-1}. The third band was observed at 14251 cm^{-1}, and an additional band at10910 cm^{-1}. These absorptions bands were assigned primarily to electronic transitions within the cobalt ion coordination sphere. The detected spectral characteristics correspond to Co^{2+} complexes, indicating the ligand coordination to metal sites. This also favours the geometry of the cobalt complex.

$^4A2(f) \xrightarrow{\nu_3} {}^4T1(p)$ with the mix (C.T) ,

so $^4A2 \xrightarrow{\nu_2} {}^4T2(f)$, and $^4A2 \xrightarrow{\nu_1} {}^4T2(F)$ transition

According to the relation (β=B-/BO), the interelectronic repulsion parameter B- has been (599cm^{-1}), and β has been equal to (0.617). For the Co^{+2} tetrahedral complex, these characteristics have been confirmed [15,16].

[Ni (NCC)$_2$] complex

Four bands corresponding to the following electronic transition: ligand-field were observed in the spectra of Ni^{+2} (at (36637 cm^{-1}), (28409 cm^{-1}), (13329 cm^{-1}) and 10757 cm^{-1}) - green-yellow complex, $^3T1 \rightarrow {}^3T1(Pmix)$ (C.T), $^3T1 \rightarrow {}^3A2(F)$, and $^3T1 \rightarrow {}^3T2(F)$; the B-value was (638 cm^{-1}), while β was (0.560) [17,18].

[Cu (NCC)$_2$] complex

The electronic spectra of the greenish-yellow Cu^{2+} complexes showed two characteristic bands with maxima at 36900 cm^{-1} and 12125 cm^{-1}. These bands were attributed to ligand-field transitions, more specifically the electronic transition $^2B_1g \rightarrow {}^2A_1g$ [19]. The spectral data we obtained provide sufficient evidence to justify coordination of the ligand with the copper ion and predict its configuration of the complex at these conditions.

[Zn(NCC)$_2$] complex

The complex with Zn^{2+} was colored orange and displayed two characteristic absorption bands in its electronic spectrum at 36230 cm^{-1} and 28911 cm^{-1}. The absorption bands were assigned to charge-transfer transitions, and the other L.F. electronic transitions. These spectral studies confirm our postulate of interaction between Zn ion and the coordinating ligand, resulting in metal complex formation with the suggested structural arrangement around the centre atom; geometric center being: Zn^{2+}.

[Cd(NCC)$_2$] complex

The electronic spectrum of the dark yellow Cd^{2+} complex showed an intense band centered at 36296 cm^{-1} attributed as a ligand-field (L.F.) transition. It confirms the presence of coordination complex as the appearance of this band represents interaction between cadmium ion and the donor atom(s) of ligand. This was also consistent with the spectral feature observed, providing additional evidence for the proposed arrangement and coordination environment around the Cd^{2+} metal center.

[Hg(NCC)$_2$] complex

The yellow Hg^{2+} complex in Fig. (7) showed a band at 36313 cm^{-1}, which was consistent with its absorption spectrum. This band may be due to the electronic transitions that involved in the coordination environment of mercury ion after complexation between metal ion and ligand. The emergence of this absorption band provides confirmatory evidence for the interaction of metal bond donor atoms coordinated with the Hg^{2+} ion, thus validating the predicted structure [20]. Table (5) summarize the associated electronic transition and spectral data.

Table (1): The metal complexes of ligand NCC & their physicochemical properties.

Compounds	M.W. (g·mol^{-1})	Color	M.P. (°C)	Molar Conductance (Ω^{-1} cm^2 mol^{-1}) in DMSO	μeff (B.M.)
Ligand (NCC) $C_{11}H_{13}N_3O_4S_2$	315	Yellow	145–150	—	—
[Mn(NCC)$_2$]	710.9	Deep yellow	146	—	—
[Co(NCC)$_2$]	714.9	Black green	290 (dec.)	—	—
[Ni(NCC)$_2$]	714.7	Green yellow	110	—	—
[Cu(NCC)$_2$]	719.5	Green yellow	170	—	—
[Zn(NCC)$_2$]	721.4	Orange	109	—	0
[Cd(NCC)$_2$]	768.4	Deep yellow	240 (des.)	—	0

Compounds	M.W. (g·mol⁻¹)	Color	M.P. (°C)	Molar Conductance (Ω⁻¹ cm² mol⁻¹) in DMSO	μeff (B.M.)
[Hg(NCC)₂]	856.6	Yellow	215 (des.)	—	0

Abbreviations:

dec. = decomposition, des. = decomposition.

Table (2): 1H-NMR ligand spectral data (NCC).

Compound	Functional Groups	δ (ppm)
	s (1H, SH)	1.4
	d (2H, CH_2)	2.8–3.1
NCC	t (1H, CH)	3.43
	s (1H, NH amine)	7.21
	(d-d) (4H, aromatic proton)	8.25–8.34
	s (1H, COOH)	9.73
	s (1H, NH sec amide)	1

Table (3): Display the (NCC) 13C-NMR spectrum data.

Compound	Functional Group	δ (ppm)
	(CH_2)	25.67
	(CH)	61.50
NCC	(C, aromatic)	124.07–150.39
	(C=O amide)	160.2
	(COOH)	172.4
	(C=S)	182.2

Table (4): FT-IR The data of Electronic spectral for metal.

Compound	ν(COO) Asym (cm⁻¹)	ν(COO) Sym (cm⁻¹)	Δν	ν (NH) (cm⁻¹)	ν(SH) (cm⁻¹)	ν(OH) (cm⁻¹)	ν(C=S) (cm⁻¹)	N (C=O) (cm⁻¹)	N (M–N) (cm⁻¹)	ν(M–O) (cm⁻¹)
NCC	1720 (s)	1345 (m)	—	3420 (m)	2550 (w)	3178 (m)	1256 (m)	1630 (s)	—	—
[Mn(NCC)₂]	1630	1411 (s)	219	3433 (b)	2560 (w)	—	1285 (w)	1660 (m)	464 (w)	429 (w)
[Co(NCC)₂]	1610	1411 (m)	199	3419 (b)	2562 (w)	—	1270 (m)	1668 (m)	470 (w)	474 (w)
[Ni(NCC)₂]	1615	1419 (s)	196	3418 (m)	2559(w)	—	1272 (m)	1626 (m)	443 (w)	482 (w)
[Cu(NCC)₂]	1609	1410 (m)	199	3459 (m)	2565 (w)	—	1264 (m)	1674 (m)	447 (w)	455 (w)
[Zn(NCC)₂]	1605	1400 (s)	205	3419 (m)	2558 (w)	—	1272 (m)	1664 (m)	432 (w)	480 (w)
[Cd(NCC)₂]	1620	1400 (m)	220	3421 (m)	2558 (w)	—	1266 (m)	1631 (m)	455 (w)	474 (w)
[Hg(NCC)₂]	1608	1415 (m)	193	3455 (m)	2567 (w)	—	1276 (m)	1678 (w)	482 (w)	520 (w)

Abbreviations:

s = strong, m = medium, w = weak, b = broad.

Table (5): Electronic spectrum data for metal complexes in DMSO solvent with the ligand (NCC)

Compound	λ (nm)	ν̄ (cm^{-1})	A	εmax ($L \cdot mol^{-1} \cdot cm^{-1}$)	Transitions
NCC	281	36387	2.343	2343	π⟶π*
	355	26469	0.701	750	n⟶π*
[Mn(NCC)$_2$]	277	36480	1.277	1277	L.F
	348	28735	0.624	624	C.T
	971	13298	0.025	25	$^6A_1 \rightarrow {}^4T_1(G)$
[Co(NCC)$_2$]	275	36103	1.931	1931	L.F
	346	27029	1.459	1459	$^4A_2(F) \rightarrow {}^4T_1(P)$ mix (C.T)
	799	14251	0.035	35	$^4A_2 \rightarrow {}^4T_1(F)$
	925	10910	0.021	21	$^4A_2 \rightarrow {}^4T_2(F)$
[Ni(NCC)$_2$]	270	36637	—	691	L.F
	352	28409	—	168	$^3T_1 \rightarrow {}^3T_1(P)$ mix (C.T)
	505	13329	—	30	$^3T_1 \rightarrow {}^3A_2(F)$
	760	10757	—	22	$^3T_1 \rightarrow {}^4T_2(F)$
[Cu(NCC)$_2$]	275	36900	2.029	2029	L.F
	389	12125	1.005	1005	$^2B_1g \rightarrow {}^2A_1g$
[Zn(NCC)$_2$]	274	26230	2.029	2029	L.F
	—	28911	—	—	C.T
[Cd(NCC)$_2$]	274	3629	1.021	1021	L.F
[Hg(NCC)$_2$]	268	36313	1.244	1244	L.F

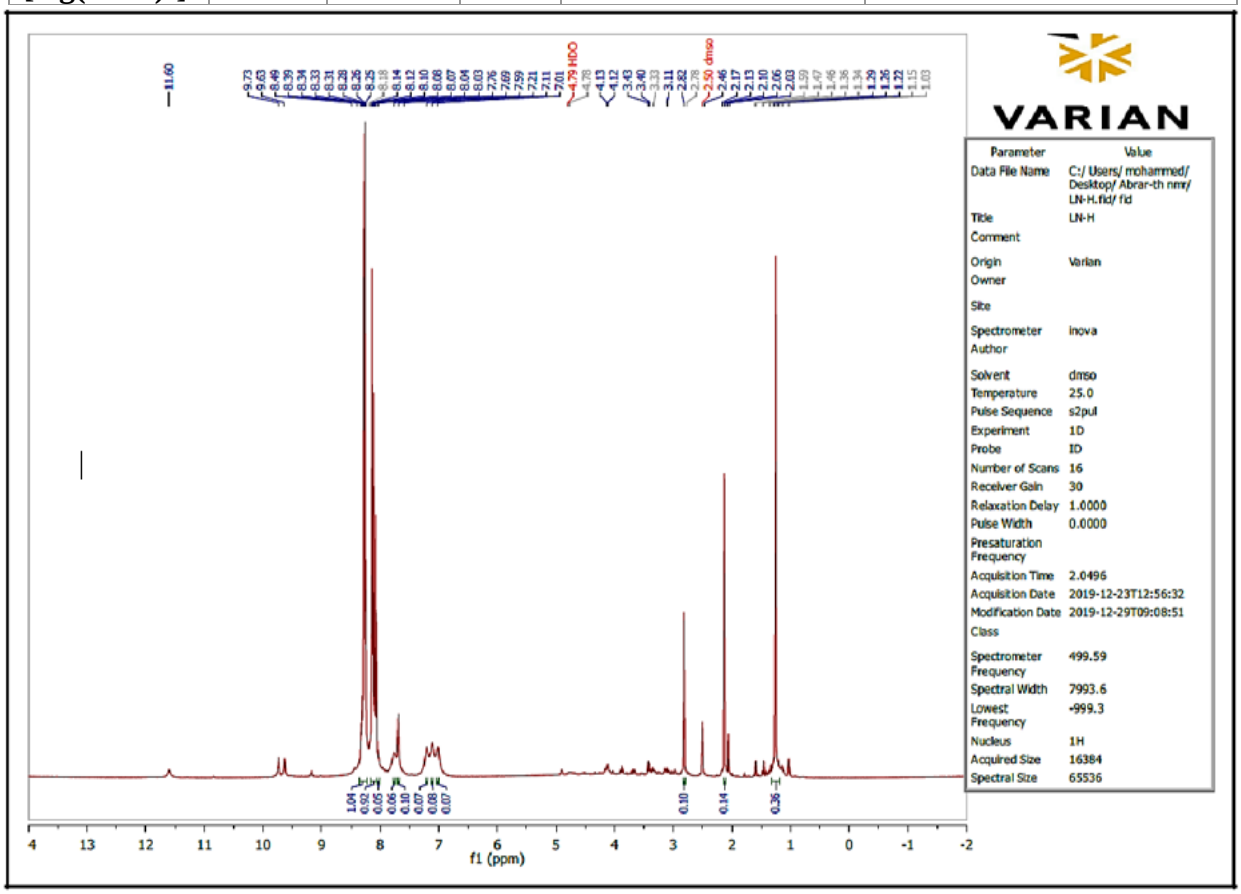


Figure (2): 1HNMR Spectrum of the ligand (NCC).

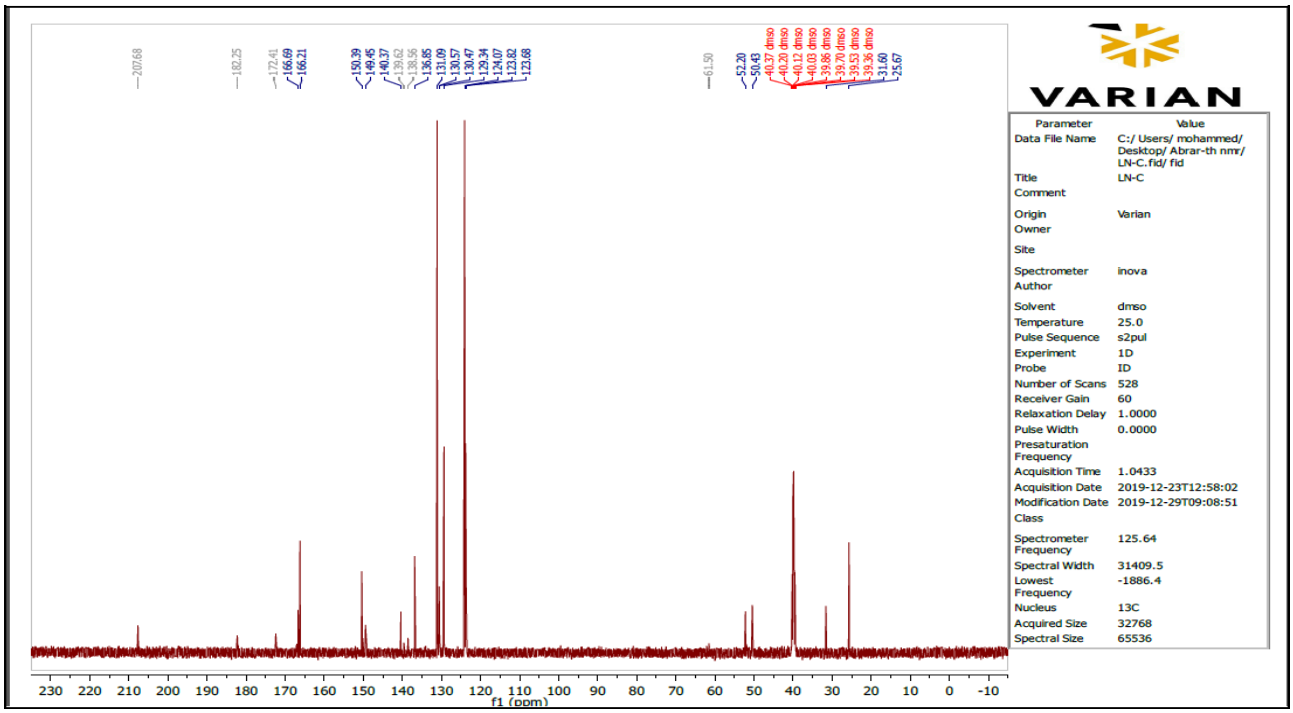


Figure (3): (NCC) 13C-NMR spectrum.

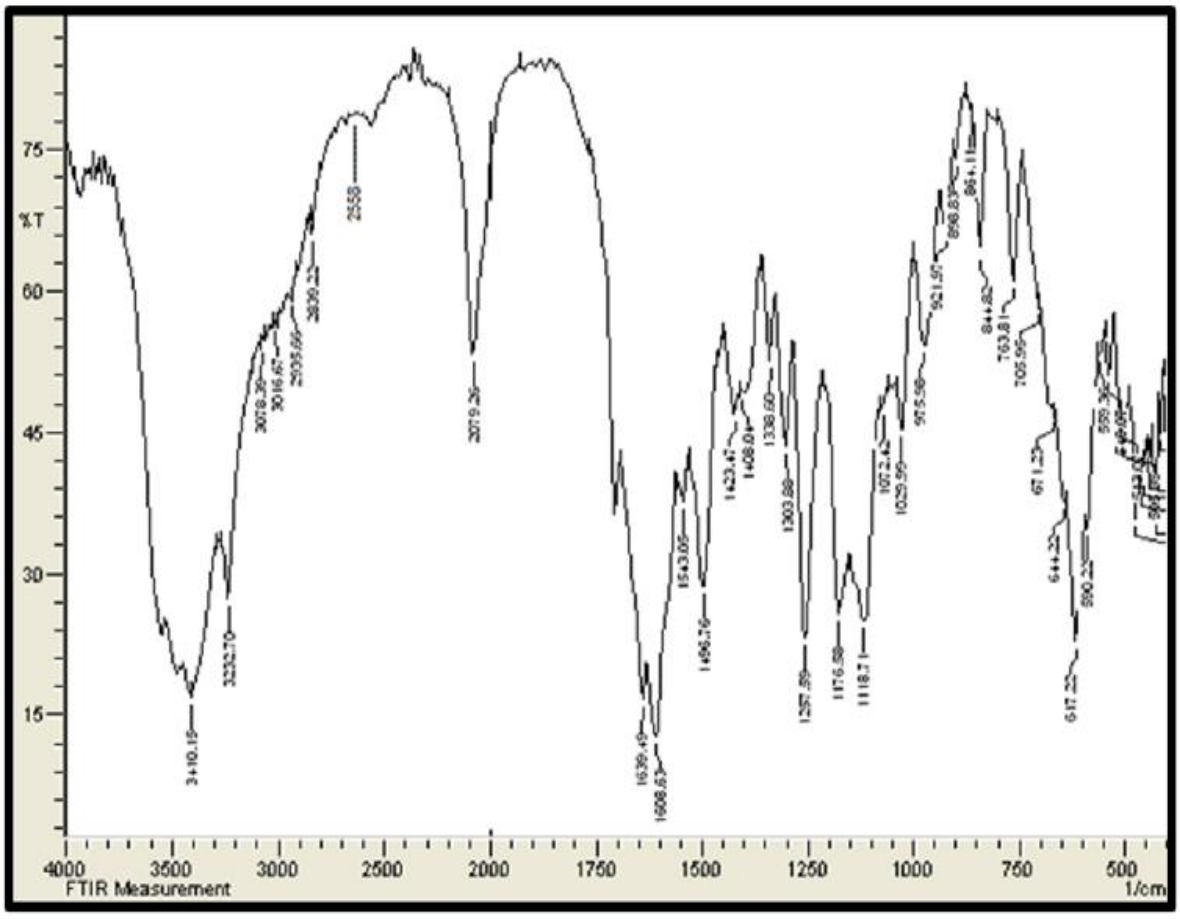


Figure (4): The ligand's FT-IR spectra (NCC).

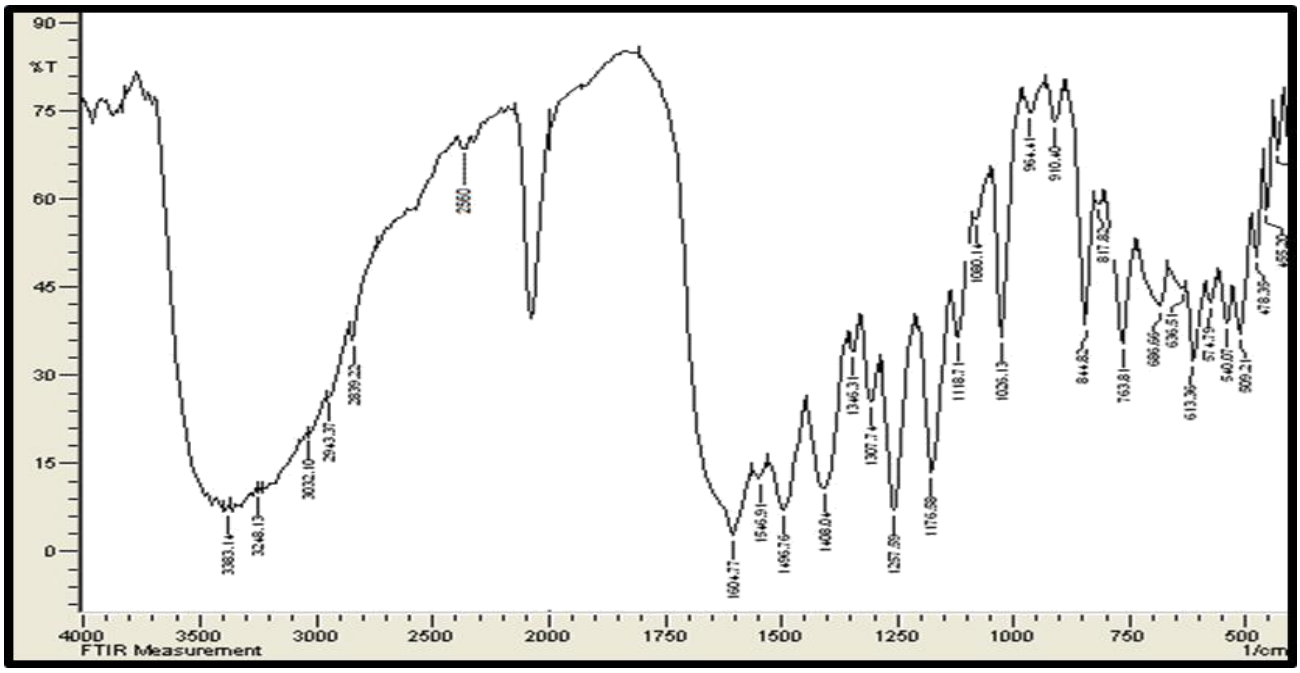


Figure (5): FT-IR spectrum of [Mn(NCC)2] complex.

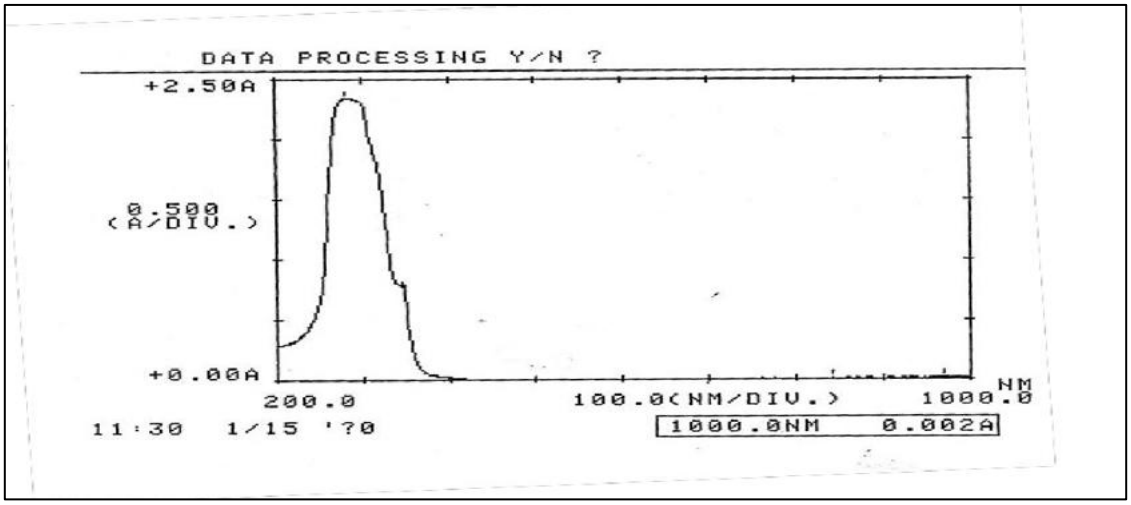


Figure (6): UV-Vis. Ligand spectrum (NCC).

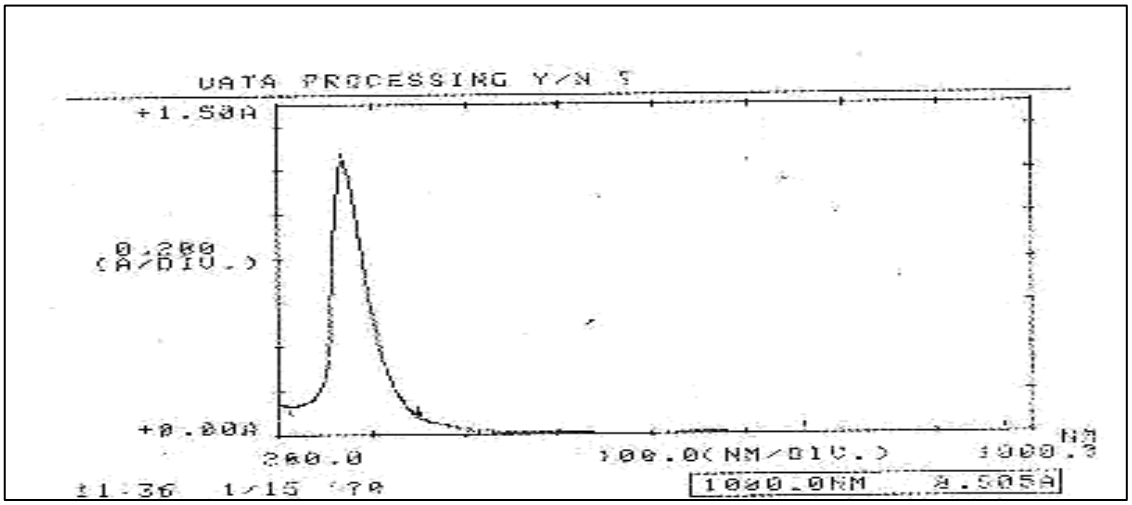
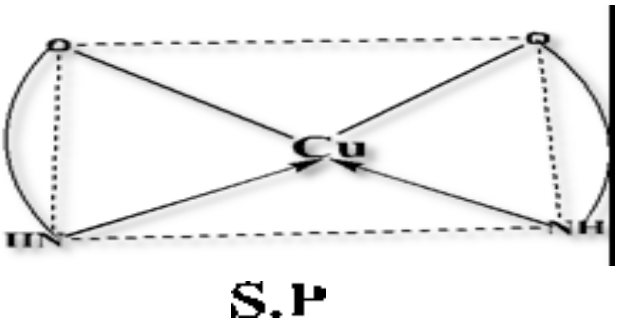


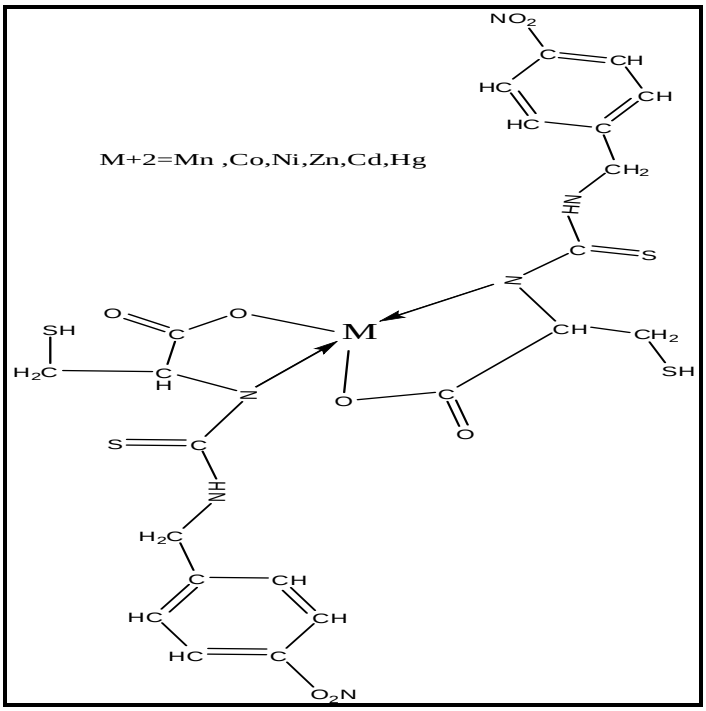
Figure (7): UV-Vis. [Hg(NCC)2)] spectrum.

CONCLUSION

In this work, a new ligand was synthesized via the reaction of 4-nitrobenzoyl isothiocyanate and cysteine amino acid. The ligand was fully characterized by elemental microanalysis (C, H, N and S), FTIR spectroscopy, UV–Visible spectroscopy as well as ^{1}H and ^{13}C NMR spectroscopy confirming the proposed structure. In addition to this, a series of metal complexes were generated from the ligand and then characterized with FTIR and UV–Visible spectroscopy along with magnetic susceptibility and conductivity measurements. The spectral and the magnetic data clearly indicated that either metal complex have tetrahedral geometries Scheme (4) except copper has square planar geometry Scheme (3) but this has a weak evidence to prove it was one of the non-innocent metals which reagent used in to synthesis.



Scheme (3): The proposed geometry of [Cu (NCC)$_2$].



Scheme (4): General suggested geometry of the complexes [M (NCC)$_2$].

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