



RESEARCH ARTICLE

Preparation and Diagnosis of a New Derivative of the Amine Acide (4- nitro benzoyl) Carbamothioyl Glycine) and some of its Complexes

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ARTICLE INFO	ABSTRACT
Received: May 21, 2024 Accepted: Jul 1, 2024	4-nitrobenzoylcarbamothioyl Glycine, New derivative NCG Prepare from interation Glycine 1:1 and 4-nitrobenzoyl isothiocyanate , its distinguished by Careful analysis of elements "C.H.N.S" , Infrared, Ultraviolet/Visible rays, "1HNMR" "13CNMR" spectrum Some of complexes for this NCG were prepared and diagnosed by UV- Visible rays "FT-IR" ,atomic absorption , "conductivity" and "magnetic susceptibility" measurements, .Through these results ,The proposed molecular formulas and structures for the complexes M(NCG) ₂ Where M represents bivalent" Magnesium, cobalt, nickel, zinc, mercury, cadmium and copper , has a tetrahedral geometry for all these complexes ,except for the copper complex which has a planer square geometry.
<p>Keywords</p> <p>4- nitro benzoyl iso thiocyanate Glycine Complexes</p>	

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INTRODUCTION

Complexes of the amino acids "glycine" with transition metals are of great importance due to their biological activity, such as antibacterial activities or antimicrobial activities or antifungal activities ¹⁻⁴ ,Glycine derivatives and their complexes that include various transition metals have antibacterial activity ^{5,6} , These complexes are used in a variety of biological processes ⁶⁻⁸ , complexes consisting of mixed ligand with glycine also have biological activities ^{10, 11} , Such as electron transfers, oxidation processes, and oxygen conversion, it forms the enzymatic active site for dimetallic ion complexes. ⁹⁻¹⁴

There is concern about " organic materials" involved in manufacturing iron in the body, peptides and Some food compounds" amino acids" like

"Gly,, Glu,, Asp and His" which are It is important in the digestion of food proteolysis', It can enhance iron absorption while iron chelating complexes improve solubility and improve bioavailable iron as well.. simple non-essential amino acid such as glycine, It, s a chelated ring for a group of different metal ions (Magnesium, Cobalt and Zinc ...ext) are divalent .In addition to this,there are stable

complexes chelates like "iron, zinc and glycine" It has vital biological activity¹⁵. In general through thermal reaction, organic iron chelating complexes can be formed, As a new method has been developed such as Pulsed electric field method "PEFS" that synthesizes iron complexes with glycine by in this study physiochemical properties were evaluated for Fe-glycine complexes, The results showed that the highest percentage of productivity reached 81.2 % by treating these PEFS at '4 kV/cm' at 25 degrees Celsius in 15 minutes, and it was higher than the percentage of heat treatment of 79.5 % at 60 degrees Celsius in 30 minutes, and the highest productivity appeared. The chelated iron capacity is 107.1 Mg/l. After the same treatment for PEFS Infrared transmission, diffraction, and thermo gravimetric analysis were measured to obtain the form of ferrous iron the preparation and heat treatment of the iron-glycine complexes were identical. This mean the possibility of using anew and effective method "the PEF treatment" can be used in Preparation of metal ion complexes of amino acids 25 C° and "pH 6.0". in general, these processes take place at high temperatures Higher than 80 degrees and the longest possible processing time (1 – 8) h¹⁶. Therefore, Other important and advanced methods must be discovered that are necessary to increase efficiency and save energy in the manufacturing process. There is a non-thermal method¹⁷, for preparing and sterilizing food, which is pulsed electrical fields which are used to extract expensive things inside cells containing plant biological materials¹⁸. In recent years, the The geometry of the polymer composites has been changed using PEFS treatments to promote chemical reaction and decomposition. "the Maillard reaction" It has become a new research method¹⁹⁻²², enhancement of ethanolic acid esterification at 25 C°, degradation of chitosan, and changing the structure of proteins, these studies showed that PEFS treatments could polarize the compounds, that is used to promote the reaction of glycine chelating accordingly promote the relative chemical reactions.

The aim of this work is to prepare some new complexes of metal ions "NCG "(4- nitro benzoyl) carbamothioyl - glycine

MATERIALS USED AND METHOD OF WORK

4-nitrobenzoyl chloride glycine "Fluka" Manganese chloride tetrahydrate " $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ " Cobalt chloride hexahydrate " $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ " " $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ " " $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ " " ZnCl_2 " " $\text{CdCl}_2 \cdot \text{H}_2\text{O}$ " and " HgCl_2 ". All reagents were chemical pure grade by "BDH", Merck and "Fluka".

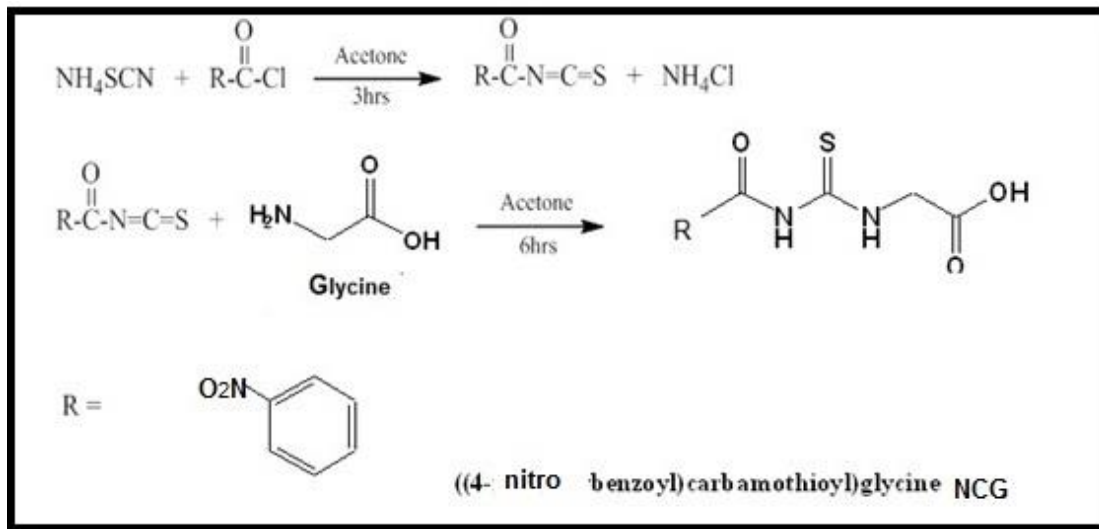
Used equipments

(¹H, ¹³C) NMR was recorded by Ultra Shield "300 MHz", Switzerland at sharif university of techonlg and Melting point by using "Stuart- melting point apparatus" and FT-IR spectra were recorded as "KBr" disc using '3800' Shimadzu in the range of 4000-400 cm^{-1} . The electronic spectra have been recorded by "UV-160" Shimadzu spectra, photometer at 25 C° in "1x10⁻³ Mollary" DMSO solvent. 'Philips Pw' To measure conductivity through digital micro-analysis for elements "C.H.N.S" use the "acrlo Erba "'1106' analyzer to measure Magnetic susceptibility. The components of the complexes were determined using the atomic absorption method "AA680G" by (model MSB-MKI)

How to prepare NCG

_ the material has been Prepared "4- nitro benzoyl isothiocyanate "²³
by mixing (4-nitrobenzoylchloride, 4.82g) with (NH_4SCN , 2g) in "25 ml" of $\text{C}_3\text{H}_6\text{O}$ solvent by stirring and refluxing for "3 hours" and by the filtration process, the filter was taken to the reactions.
_The ligand has been Prepared" (4-nitrobenzoyl) carbamothioyl) glycine"
by mixing ($\text{C}_2\text{H}_5\text{NO}_2$, 1.95g) with "21 ml" $\text{C}_3\text{H}_6\text{O}$ solvent was After refluxing for 6 hours, the solid product was taken and washed with acetone as a solvent recrystallization from $\text{C}_2\text{H}_6\text{O}$ solvent "Scheme 1" It's percentage "80%" , it's melting point "185 -187 C°" , carbon% found "42.40" ,

calculate "42.60" Hydrogen% found "3.21", calculate "3.45" nitrogen % "14.80", calculate "15.66", Sulfur % found "11.00" while calculate "11.90"



“Scheme 1 Preparation of Ligand NCG “

Preparation of metal complexes

"0.566 g, 2mmole" of 'Ligand NCG' was dissolved in "20 ml" of ethanol solvent containing "0.112 g, 2mmole" of 'KOH', then the solution of following metal salts $MnCl_2 \cdot 4H_2O$ "0.200 g, 1mmole", $CoCl_2 \cdot 6H_2O$ "0.237 g, 1mmole", $NiCl_2 \cdot H_2O$ "0.237 g, 1mmole", $CuCl_2 \cdot 2H_2O$ "0.170 g, 1mmole", $ZnCl_2$ "0.136 g, 1mmole", $CdCl_2 \cdot H_2O$ "0.201 g, 1mmole", $HgCl_2$ "0.272 g, 1mmole" in ethanol, were added dropwise to the solution of (NCG- K^+), The precipitate formed immediately after stirring the mixture at "25 C°" for "3 hours", The precipitate was collected by filtration, washed with distilled water and ethanol solvent and dried under vacuum, Physical properties were given in the "Table 1"

RESULTS WORK AND DISCUSSION

The complexes were solid and relatively thermally stable and soluble in some common solvents e.g "dimethyl sulphoxide, dimethyl formamide,,. The molar conductivity of all complexes in "DMSO" were found to be 'non-electrolyte' as in "Table 1 includes the physical properties for (NCG) and its metal complexes".

Table 1 Physical properties of "ligand NCG and its metal complexes"

Ligand and Its complexes	M.wt 'g/mol'	ColorLigand andIts complexes	M.p °C or dec.	% M "Calculation Found'	Molar condu. 'Ohm ⁻¹ cm ² mol ⁻¹ '	μ_{eff} 'B.M'
(NCG)	283	Yellow	185-187	-	3.21	-
[Mn(NCG) ₂]	619	Yellow	182- 184	8.90 (8.21)	6.33	5.83
[Co(NCG) ₂]	623	Deep Blue	288-290	9.50 (9.22)	10.11	4.63

[Ni(NCG) ₂]	622	Deep Yellow	184	9.00 (8.88)	3.14	2.96
[Cu(NCG) ₂]	627	Yellow	198	10.00 (9.87)	6.70	1.74
[Zn(NCG) ₂]	629	Yellow	200-202	10.00 (9.52)	5.82	0
[Cd(NCG) ₂]	676.4	yellow	223-250	16.60 (16.50)	18.42	0
[Hg(NCG) ₂]	765	White	178-180	26.30 (26.07)	6.58	0

Spectral

(¹H and ¹³C) NMR spectra

¹HNMR 'spectrum for' NCG in 'DMSO as solvent' Fig1 "showed the following signals" singlet at "δ4.01 ppm" for (2H, CH₂) multiplet at "δ 2.48-2.49 ppm" for DMSO solvent ,, singlet at "δ 7.14 ppm" for (1H, NH) sec.amine,, multiplet at "δ 8.01-8.27 ppm" for aromatic protons, singlet at "δ 13.01 ppm" for (1H, NH) sec.amide ,, singlet at "δ 12.88 ppm" for(1H, COOH) ²⁴.

Table 2 "¹HNMR Spectral data for NCG"

Compound	Functional grop	ppmδ
Ligand NCG	S ' 2H,CH ₂ '	4.01
	S 1H,NH aromatic	7.14
	2d ' 4H,aromatic '	8.01-8.27
	S ' 1H,COOH'	12.88
	S '1H,NHsec,amide'	13.01

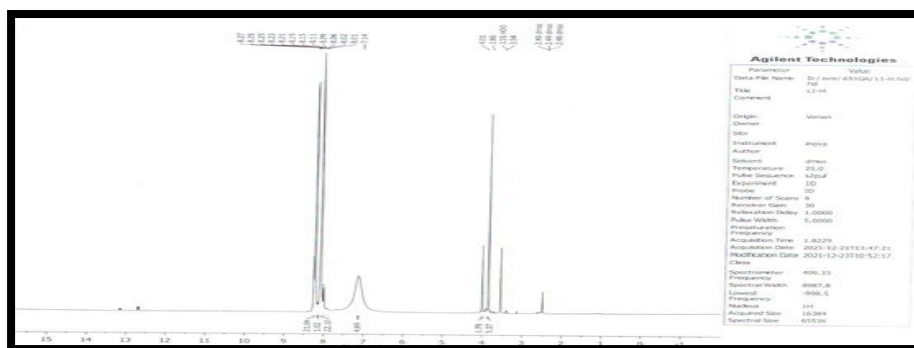


Fig 1 (¹HNMR spectrum of NCG)

The ^{13}C NMR spectrum for NCG in DMSO solvent, Fig2 showed the following signals, signals at δ 53.21 ppm for (CH_2 , multiplet) at "39.24-40.57" ppm for DMSO, signals at δ 123.77-150.54 ppm for aromatic carbons, signal at δ 165.14 ppm for ($\text{C}=\text{O}$ sec.amide), signal at δ 166.31 ppm for COOH, signal at δ 185.01 ppm for $\text{C}=\text{S}$.

Table 3 ^{13}C NMR Spectral values for NCG

Ligand	Protons'	δ 'ppm'
NCG	s ' CH_2 '	53.21
	m C -aromatic	123.77-150.54
	s $\text{C}=\text{O}_{\text{sec.amido}}$	165.14
	COOH s	166.31
	s $\text{C}=\text{S}$	185.01

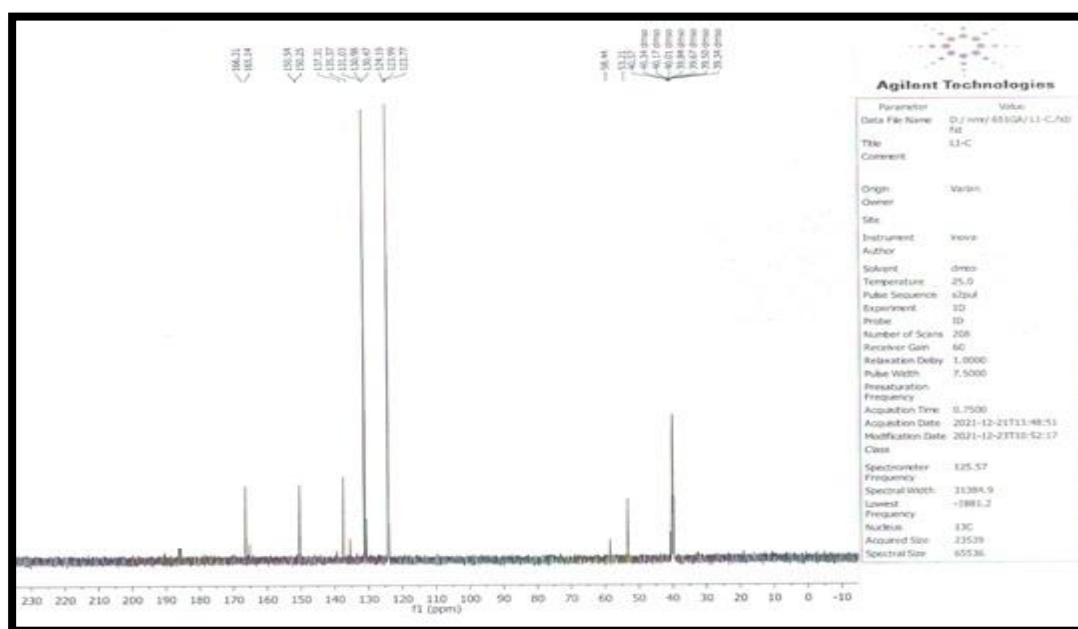


Fig 2 ^{13}C NMR spectrum of NCG 'Infrared spectra'

"Infrared spectrum of the free NCG Fig3 showed bands" due to amido ν (NH , $\text{C}=\text{O}$, $\text{C}=\text{S}$) which absorbed at ($3414, 1608, 1280$) cm^{-1} respectively: while another absorption band appeared at (1716 cm^{-1}) could be explained as ' $\nu\text{COO}'_{\text{asym}}$ '^{25,26}. where the ' $\nu\text{COO}'_{\text{sym}}$ ' was noticed at

(1350 cm^{-1}): The Infrared spectra of complexes exhibited marked difference between bands belonging to the stretching vibration of ' νNH ' of the "amine group" in the range between ($3255 - 3395$) cm^{-1} shifted lower frequency ($159 - 19$) cm^{-1} suggesting of the possibility of the 'coordination' of NCG, through the nitrogen atom at the amine group²⁷. absorption assigned for ' $\nu\text{COO}'_{\text{asym}}$ ' was noticed at the range ($1573 - 1620$) cm^{-1} shifted to "higher frequencies" ($143 - 96$) cm^{-1} while the band caused by ' $\nu\text{COO}'_{\text{sym}}$ ' appeared between ($1362 - 1495$) cm^{-1} shifted to lower frequency by ($145-12$) cm^{-1} which indicates to the "coordination of the carboxylic group" to the central ion²⁸.

The stretching vibration band $\nu(\text{C}=\text{O})$, $\nu(\text{C}=\text{S})$ carbonyl group either shows no change or very "little" in their frequencies ($1610 - 1627$) cm^{-1} , ($1276 - 1288$) cm^{-1} respectively there for indicating do" The asymmetry of the metal ions²⁹ with the bonds of both nitrogen and oxygen is confirmed by the

presence of the stretching vibration Frequencies (M-O, M-N) around them (466 - 505 , 420 – 493) $1/ \text{cm}$.

table 4 ' it showed the exact spectral frequencies of the ligand free NCG and its complexes. FT-IR spectrum of "Ni complex "showed in Fig 4

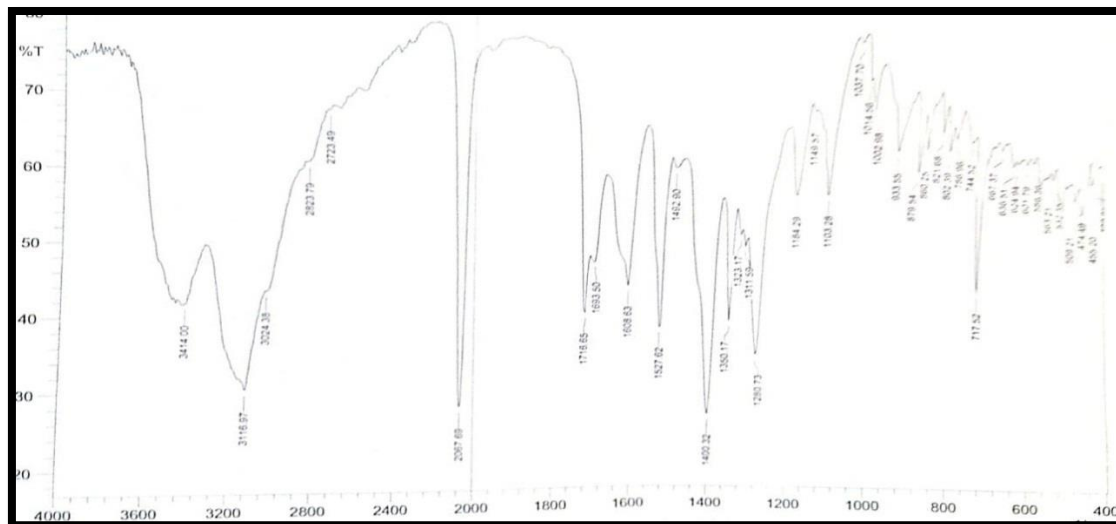


Fig 3 "Infrared spectrum of Ligand NCG"

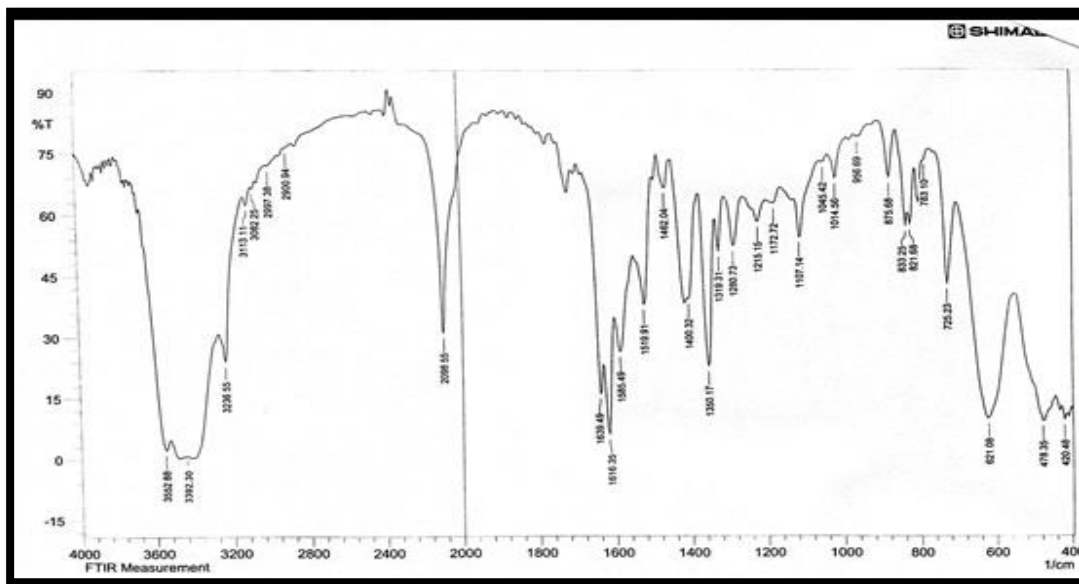


Fig 4 "Infrared spectrum of Ni(NCG)₂ Complex"

Table 4 Some FTIR frequency values for 'ligand NCG' and its complexes in (cm^{-1})

Ligand, Complex	ν 'N-H'	ν 'COO' _{sym}	ν 'COO' _{asym}	ν 'C=O'	ν 'C=S'	ν 'M-O'	ν 'M-N'
NCG	3414 (s)	1350 (s)	1716(s)	1608(s)	1280(s)	-	-
[Mn(NCG) ₂]	3348(m)	1492(w)	1612(m)	1612 (w)	1280(m)	470(w)	420(w)
[Co(NCG) ₂]	3390(m)	1492(w)	1616(m)	1616(m)	1280(m)	470(w)	424(w)
[Ni(NCG) ₂]	3392(m)	1462(w)	1616(s)	1616(s)	1280(m)	478(w)	420(w)
[Cu(NCG) ₂]	3395(m)	1470(w)	1585(s)	1610(s)	1276(m)	505(w)	493(w)
[Cd(NCG) ₂]	3391(m)	1473(w)	1585(s)	1627(s)	1276(s)	470(w)	455(w)
[Zn(NCG) ₂]	3255(w)	1465(w)	1573(m)	1624(m)	1288(m)	466(w)	420(w)
[Hg(NCG) ₂]	3394(m)	1495(m)	1620(m)	1620(m)	1276(s)	466(w)	447(w)

b "browed" w "weak" s "strong" m "medium"

Metal Complexes and their Magnetic properties

Magnetic moments μ_{eff} of complexes related to "Mn⁺⁺ d⁵", "Co⁺⁺d⁷" have been indicated '5.83 B.M' ,, "4.63 B.M" Higher spin values are expected μ_{eff} "Ni⁺⁺d⁸" complexes "2.96 B.M" μ_{eff} "Cu⁺⁺d⁹" complexes "1.74 B.M" One electron is an expected value ^{31,39}. All value are indicated in 'Table 1'

Spectra of ligand and its Complexes

UV-Vis. spectra of NCG and it's complexes "Table 5" the solution of NCG in 10⁻³ Mollary DMSO solvent showed (2peaks) " Fig 5" (37313 - 27027) 1/cm which are Referred ($\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$) ³².

[Magnesium (NCG)₂] d⁵: complex Mn⁺² spectrum "brown" shows a link at (37878 ,26738) cm⁻¹ with ligand felid and the electronic transfer "⁶A₁→⁴T₂" at (17793) 1 /cm⁻¹, also link at (12376) cm⁻¹ which is caused "⁶A₁→⁴T_{1G}" transfer ³³

[cobalt (NCG)₂] d⁷: complex Co⁺² spectrum "deep green" gave(4) bands at (25706, 19047, 12269 , 37878)cm¹ attributed to "ligand felid" , " C.T" (⁴A₂→⁴T_{1(P)}),(⁴A₂→⁴T_{1(F)}) and (⁴A₂→⁴T_{2(F)}) ,,the rach interelectronic repulsion parameter '(B= 529.7)cm⁻¹, from " $\beta = B / B_0$ " found = "0.545", Structure of divalent tetrahedral cobalt complexes ³⁴.

[Nickel (NCG)₂] d⁸: complex Ni⁺² spectrum " Brown " has revealed the

electronic transfers "ligand felid", "C.T" mix(³T_{1(F)}→³T_{1(P)}),(³T_{1(F)}→³A_{2(F)}) and (³T₁→³T_{2(F)}) at (37453, 25904 , 20242) 1/cm and (11976) 1/cm, the (B-) = 681.2 cm⁻¹, (β) = "0.654" Structure of divalent tetrahedral nickel complexes ³⁵.

[copper (NCG)₂] d⁹: complex Cu⁺² spectrum "green" in Fig 6 ,, show '3 bands' at (37735, 26455, 16920) 1/cm and (12738) 1/cm to "ligand felid" , "C.T" (²B_{1g}→²A_{1g})and(²B_{1g}→²B_{2g}) ³⁶

zinc (NCG)₂, Cadmium (NCG)₂ and Mercury (NCG)₂ complexes give "ligand field" and give "charge transfer" (37594, 26525, 37735) 1/cm., (27173 , 37735, 27548) 1/cm³⁷., All information and transitions are in Table 5.

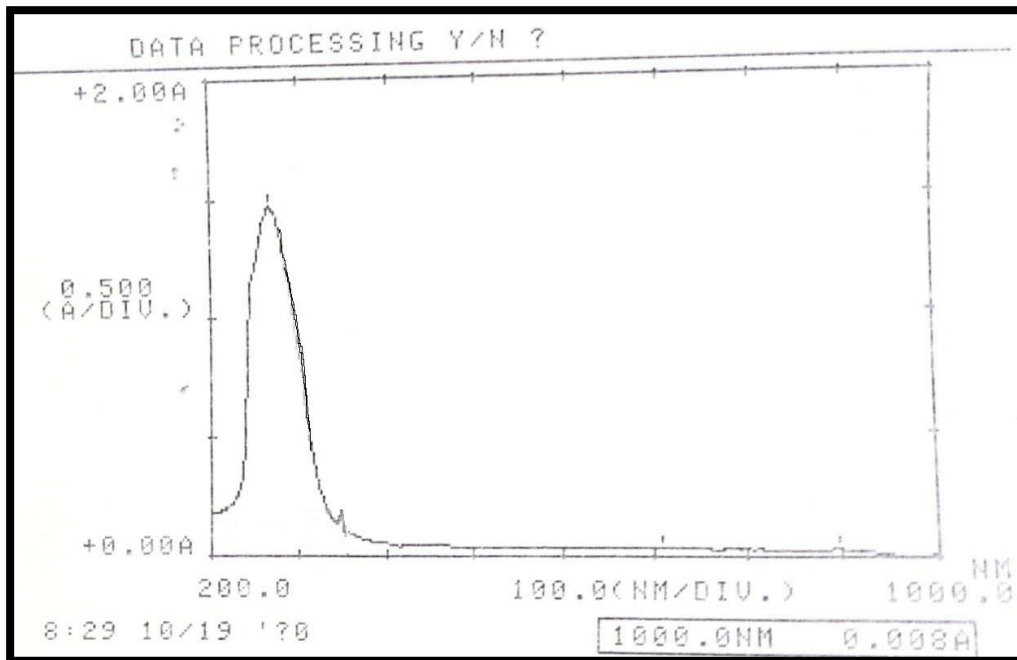


Fig5 " transition NCG"

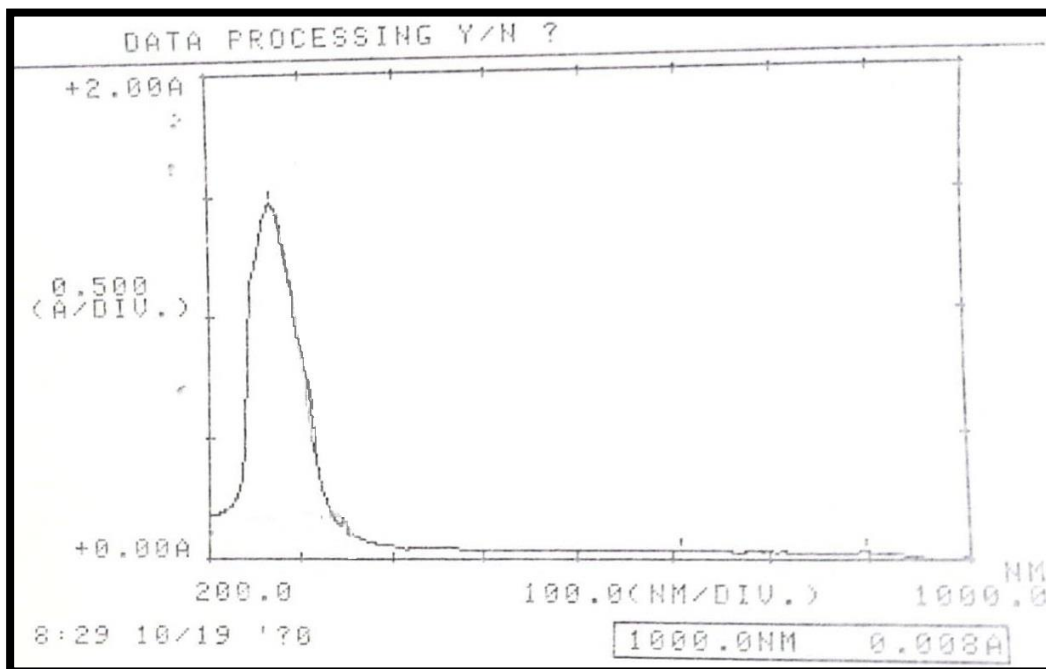


Fig 6 " transition Cu (NCG)₂ Complex"

Table 5 peaks of electronic' transitions and chemical the Geometric shapein " NCG and some of their metal complexes"

Transitions	max" ϵ " L.mol ⁻¹ .cm ⁻¹ (Wave length number λ cm ⁻¹	(ABC)	Wave length λ 'nm'	Ligand and Complexs
$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	1471 210	37313 27027	1.471 0.210	268 370	NCG
L.F C.T ${}^6A_1 \rightarrow {}^4T_1(G)$ (${}^6A_1 \rightarrow {}^4T_2(G)$ (1787 175 45 37	37878 26738 17793 12376	1.787 0.175 0.045 0.037	264 374 562 808]Mn(NCG) ₂ [
L.F (C.T mix ${}^4A_2 \rightarrow {}^4T_1(p)$) ${}^4A_2 \rightarrow {}^4T_1(f)$ (${}^4A_2 \rightarrow {}^4T_2$) (1810 110 65 35	37878 25706 19047 12269	1.810 0.110 0.065 0.035	264 389 525 815]Co(NCG) ₂ [
L.F)(${}^3T_1 f \rightarrow {}^3T_1 p$) C.T mix (${}^3T_1 f \rightarrow {}^3A_2$)(${}^3T_1 f \rightarrow {}^3T_2 f$)(1537 145 58 35	37453 25904 20242 11976	1.537 0.145 0.058 0.035	267 386 494 835]Ni(NCG) ₂ [
L.F C.T ${}^2B_{1G} \rightarrow {}^2A_{1G}$) ((${}^2B_{1G} \rightarrow {}^2B_{2G}$)	1865 210 75 60	37735 26455 16920 12738	1.865 0.210 0.075 0.060	265 378 591 785]Cu(NCG) ₂ [
L.F C.T	1656 195	37594 26525	1.656 0.195	266 377]Zn(NCG) ₂ [
L.F C.T	1691 225	37735 27173	1.691 0.225	265 368]Cd(NCG) ₂ [
L.F C.T	1700 148	37735 27548	1.700 0.148	265 363]Hg(NCG) ₂ [

" Charge transfer" C.T

"Ligand felid" L.F

Proposed chemical engineering of complexes relying on data or spectral values obtained from elemental analyses,, "tetrahedral" for M(NCG)₂ complexes " M= Magnesium ,Cobalt , Zinc , nickel , Cadmium , Mercury,and Cu , M (dual equivalent) Fig7 while Cu⁺² complex has "square planer".

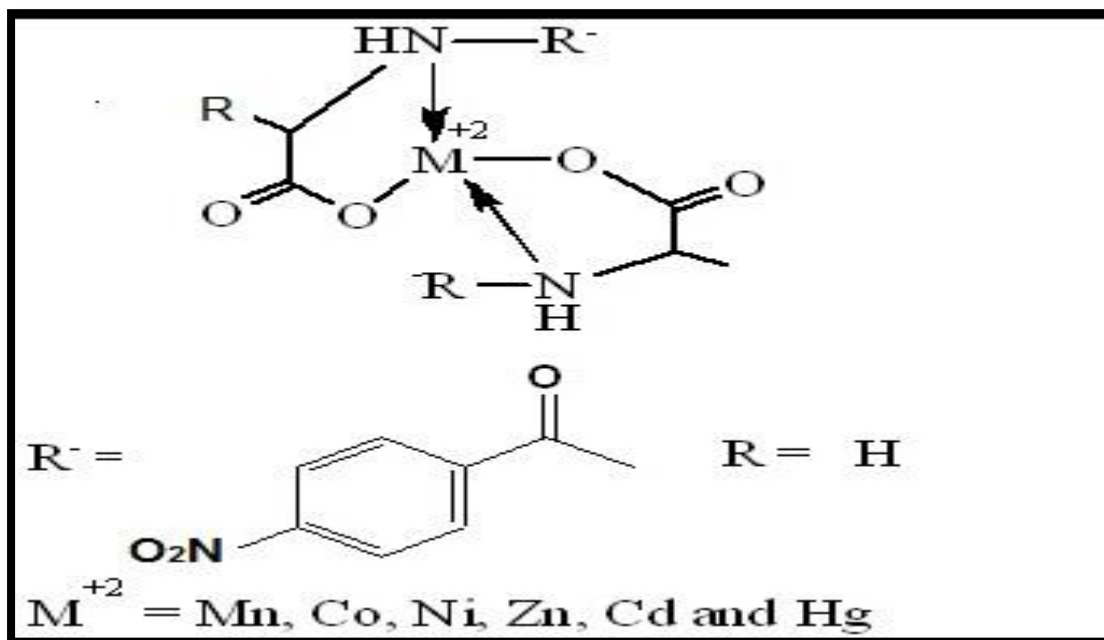


Fig7 " proposed chemical structure of $M(\text{NCG})_2$ complexes"

CONCLUSION

Through the presented study, a new ligand " NCG" prepared from the simplest amino acid, which is glycine, was prepared and characterized through its interaction with the chemical substance 4-Nitro benzoyl isothiocyanate, it was diagnosed by micro analysis elements "C.H.N.S" Infrared rays, UV rays -Visible rays ; ^1H - ^{13}C NMR . Some new metal complexes I attended and then I was diagnosed from this ligand were measured (FTIR rays, UV rays - Vis) also both magnetic measure and conductivity measure, Proposed chemical engineering with copper complex that has " square planer geometry " while with all complexes have been " tetrahedral geometry " .

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