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New Metal Complexes of 2-(3-(4-Nitrobenzoyl)Thioureido)Benzoic Acid, Synthesis and Characterization (NTB)

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Abstract

The presence of a drive site within ligand structures plays a crucial role in enhancing their interaction with metal ions, resulting in highly efficient complex formation research. A new ligand, 2-(3-(4-nitrobenzoyl) thioureido) benzoic acid (NTB), was successfully synthesized via the reaction of 4-nitrobenzoyl isothiocyanate with anthranilic acid in a 1:1 molar ratio. The structure of the ligand was confirmed using elemental analysis, FT-IR, UV-Vis., and ^{1}H and ^{13}C NMR spectroscopy. Then a series of metal complexes was synthesized by reacting NTB with divalent metal ions, including Hg^{2+}, Cd^{2+}, Zn^{2+}, Cu^{2+}, Ni^{2+}, Co^{2+}, and Mn^{2+}. The analytical results suggest that all metal complexes adopt a tetrahedral geometry, except for the copper complex, which exhibits a square planar structure. The metal-to-ligand ratio in all complexes was determined to be 1:2, supporting the proposed $[M(NTB)_2]$ formulation.

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1. Introduction

Anthranilic acid, which is known as 2-aminobenzoic acid, is an aromatic compound of significant biochemical and industrial relevance due to its structural versatility and bioactivity[1-3]. It serves as a key precursor in the synthesis of various biologically active molecules, including alkaloids, dyes, and pharmaceutical agents[4,5]. Several studies have highlighted its pharmacological properties, particularly in anticancer, antimicrobial, and anti-inflammatory applications[6-8]. Additionally, derivatives of anthranilic acid have demonstrated potent inhibitory effects against metal corrosion, especially for copper and its alloys, in aggressive environments[9]. Transition metal complexes formed with anthranilic acid and its derivatives have garnered considerable interest for their diverse geometrical arrangements, rich electronic structures, and potential therapeutic benefits[10,11]. Their coordination behavior often leads to complexes with unique optical and catalytic properties, making them valuable in various scientific and industrial applications[10-12]. In recent years, thiourea-based ligands incorporating anthranilic acid moieties have emerged as promising candidates in coordination chemistry due to their bidentate nature and electron-donating functionalities[13,14]. In particular, the incorporation of nitrobenzoyl isothiocyanate into such ligands can modulate their electronic characteristics and enhance metal-binding affinities[15-17]. The present study introduces a ligand, 2-(3-(4-nitrobenzoyl) thioureido) benzoic acid (NTB), synthesized via the reaction of 4-nitrobenzoyl isothiocyanate with anthranilic acid. The novelty of this ligand lies in its dual donor centers (NH and C=O/S), enabling the formation of stable complexes with a large range of divalent transition metals, including Hg(II), Cd(II), Zn(II), Cu(II), Ni(II), Co(II), and Mn(II). A comprehensive characterization of these complexes has been conducted using analysis of elemental, FT-IR, ^{1}H with ^{13}C-NMR, UV-Vis spectroscopy, magnetic susceptibility, molar conductivity, and atomic absorption techniques. This research contributes to the field of coordination chemistry by:

- Proposing a new ligand framework with tunable coordination sites.
- Demonstrating the formation of metal complexes with diverse geometries (e.g., square planar for Cu(II) and the tetrahedral for others).

- Providing spectral and structural insight into metal-ligand interactions that may inspire further studies in bioinorganic and materials chemistry.

Such findings not only deepen the understanding of ligand design but also expand the potential for developing new functional materials with tailored properties.

2. Experimental

2.1 Chemicals and Reagents

In the present study, all chemicals were of analytical grade and procured from reputable suppliers, including BDH, Merck, and Fluka. The primary materials included: Anthranilic acid, 4-nitrobenzoyl chloride, ammonium thiocyanate, acetone, ethanol, and a series of metal salts: $HgCl_2$, $CdCl_2 \cdot H_2O$, $ZnCl_2$, $CuCl_2 \cdot 2H_2O$, $NiCl_2 \cdot CoCl_2 \cdot 6H_2O$, and $MnCl_2 \cdot 4H_2O$.

2.2 Instrumentation

The instrumentation deployed for the characterization includes:

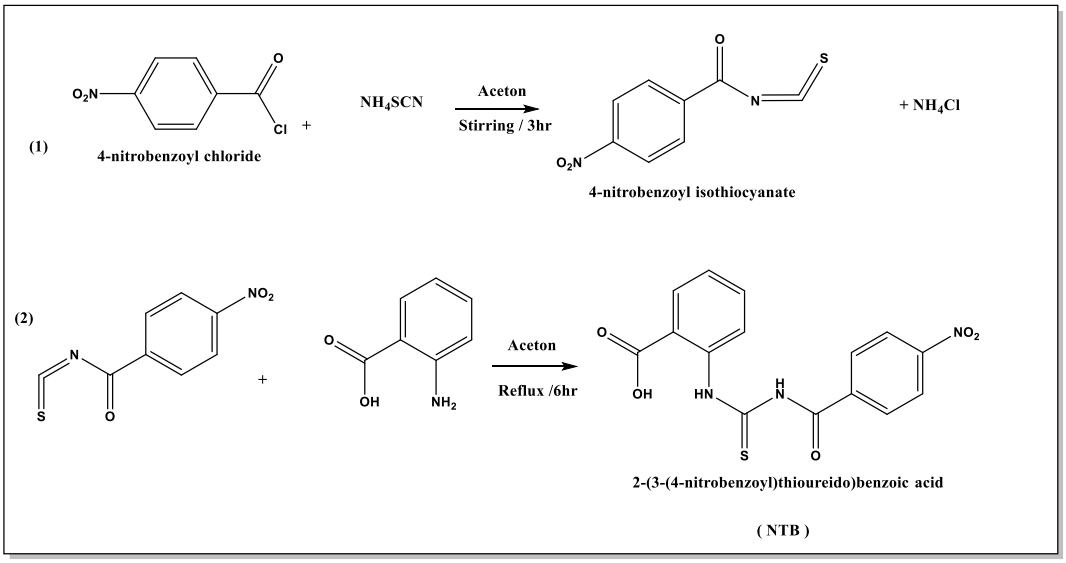
- FT-IR Spectroscopy: Conducted using a Shimadzu 3800 instrument in the range of 400–4000 cm^{-1} with samples prepared as KBr discs.
- UV-Vis Spectroscopy: Measured using a Shimadzu UV-160 spectrophotometer in DMSO at 25°C.
- NMR Spectroscopy: 1H and 13C-NMR spectra were recorded using a Bruker UltraShield 300 MHz spectrometer.
- Elemental Analysis: Implemented by an Elemental Vario EL III analyzer (C, H, N, S).
- Magnetic Susceptibility: Determined with an MSB-MK1 balance.
- Conductivity Measurements: Carried out using a Philips PW Digital Conductometer.
- Atomic Absorption Spectroscopy (AAS): Employed a Shimadzu AA-680G spectrometer for metal content analysis.

2.3 Synthesis of Ligand (NTB)

Step 1: Synthesis of 4-nitrobenzoyl thiocyanate. A solution of 4.87 g of 4-nitrobenzoyl chloride and 1 mmol of ammonium thiocyanate in 25 ml of acetone was refluxed for 3 hours, helping obtain the benzoyl isothiocyanate intermediate [18]. After filtration, the product is used in the next step.

Step 2: Synthesis of NTB, To the filtrate, 3.60 g of anthranilic acid in 20 ml of acetone was added under continuous reflux for 6 hours, as in green synthesis protocols using elemental sulfur for thiourea formation[19]. The precipitated yellow solid was filtered, washed with acetone, and then recrystallized from ethanol.

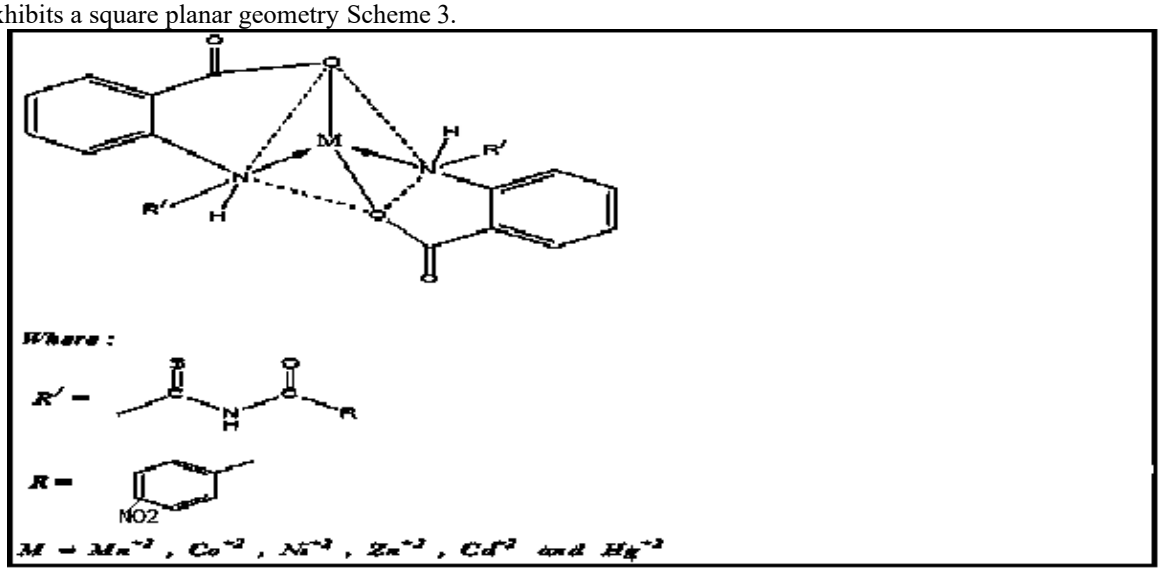
Yield: 79%, Melting point: 240–242°C, Elemental Analysis: Calcd. C (52,17%), H (3.21%), N (12.17%), O (23,16%), S (9,28%). Elemental analysis found: C (51.99%), H (3.16%), N (12.11%), O (23,84%) ,S (8.90%), as shown in the following Scheme 1



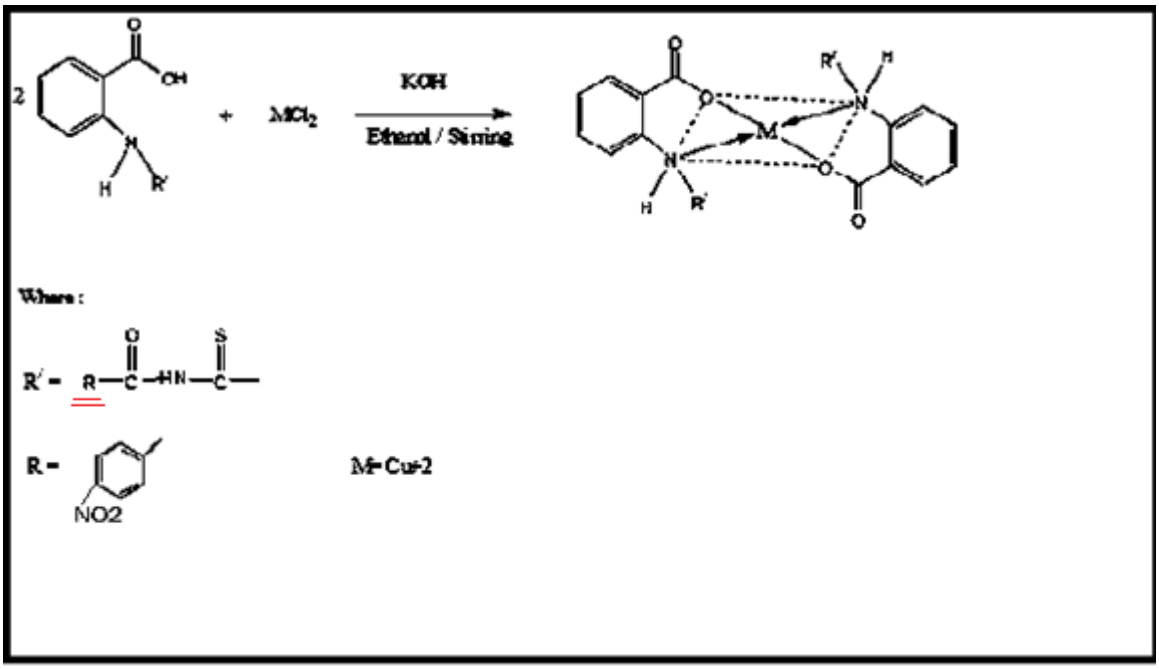
Scheme(1) preparation of ligand (NTB)

2.4 Synthesis of Metal Complexes

The complexes were prepared by slowly adding a metal salt solution (1 mmol) dropwise to a potassium salt of NTB solution. The mixture was stirred for 3 hours at room temperature. The resulting precipitate was filtered, washed with ethanol and water, and dried under vacuum. Scheme 2 presents the general tetrahedral structure of the complexes $[M(NTB)_2]$, while Cu(II) exhibits a square planar geometry Scheme 3.



Scheme 2**.** The proposed chemical structure formula of the complexes



Scheme 3**.** The proposed chemical structure formula of the complex with Cu

2.5 Spectral and Physicochemical Characterization

a. NMR Spectra of NTB

The 1H-NMR spectrum, as shown in Figure 1, reveals characteristic peaks: DMSO solvent at 2.51 ppm, NH (amine) at 7.35–7.74 ppm, NH (amide) at 11.64 ppm, and COOH at 13.14 ppm. The 13C-NMR spectrum (Figure 2) shows peaks at: C=S (180 ppm), COOH (168.11 ppm), aromatic carbons (125–139 ppm), and amide C=O (155.12 ppm).

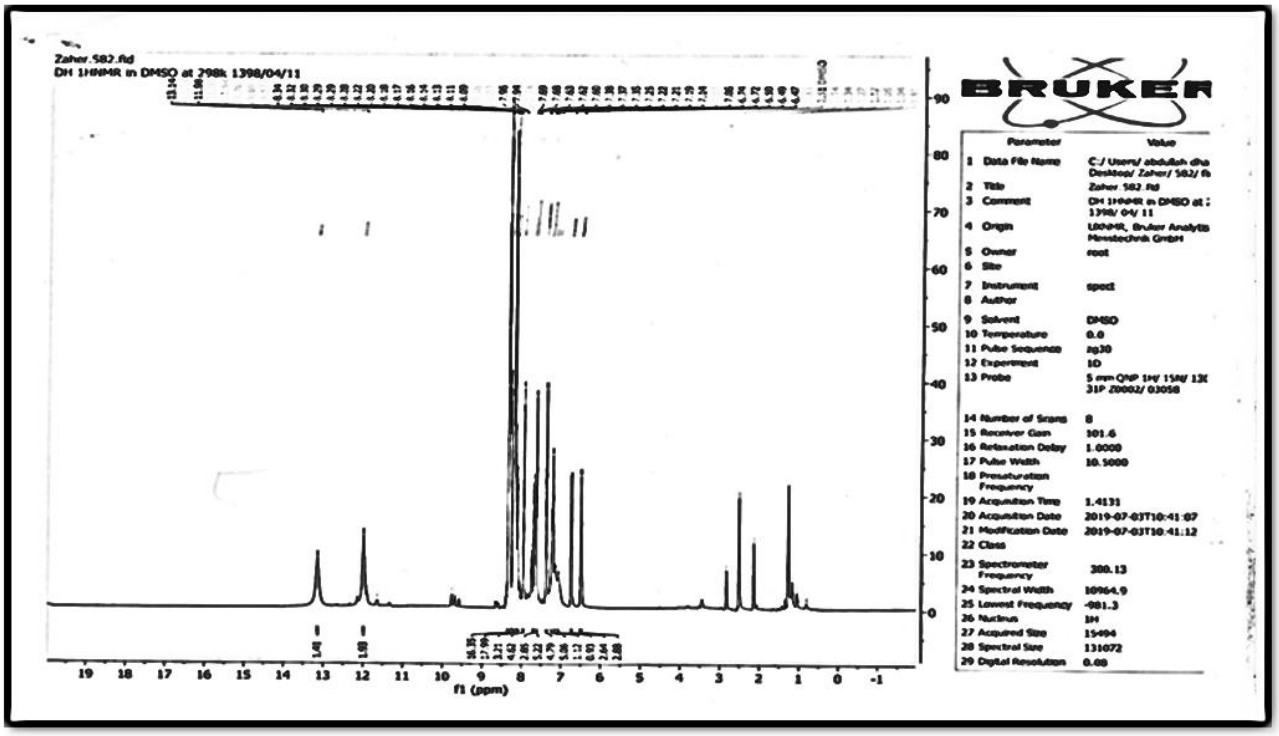


Figure 1. the ^{1}H-NMR spectrum of Ligand (NTB)

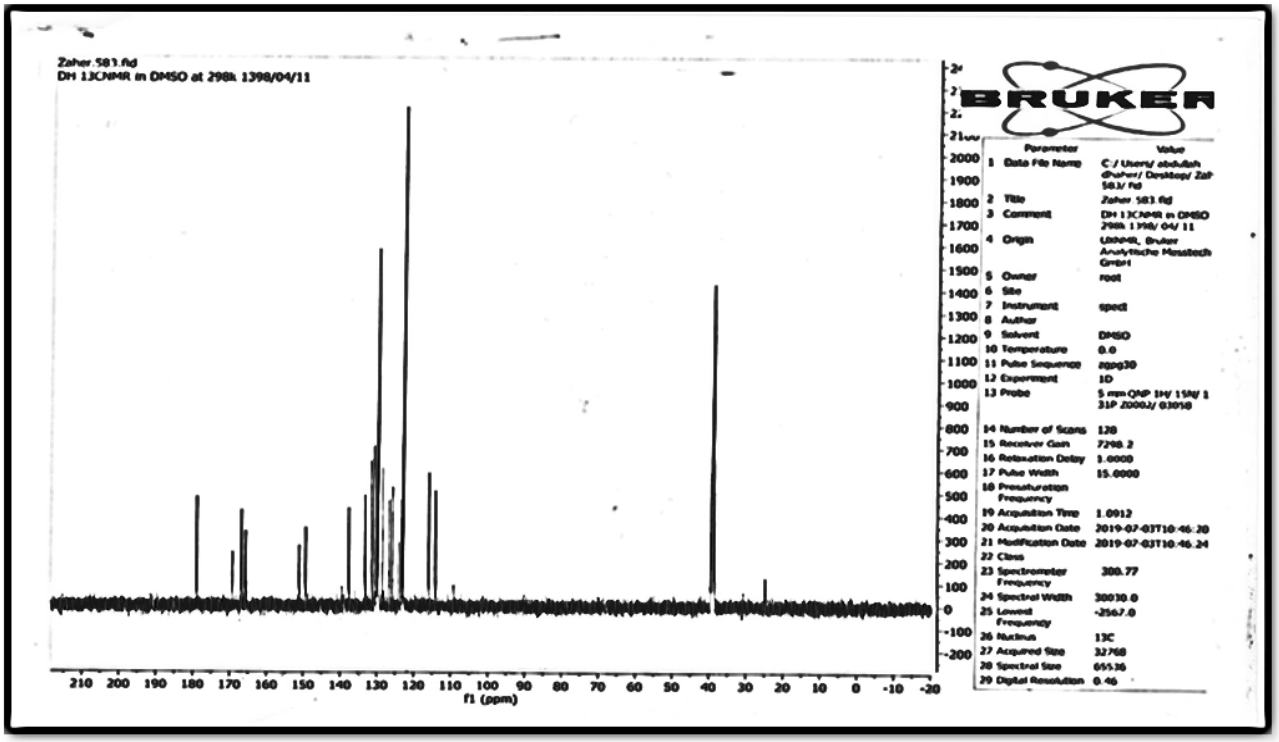


Figure 2. The C^{13}-NMR spectrum of ligand(NTB)

The ^{1}H-NMR and ^{13}C-NMR spectra of the free ligand (Figures 1 and 2) confirm the structure of NTB. The presence of peaks at δ ≈ 13.14 ppm (COOH), δ ≈ 11.64 ppm (amide NH), and aromatic signals between δ ≈ 7.35-8.20 ppm are

consistent with the expected proton environments. The ^{13}C-NMR further supports the structure with signals for carbonyl (C=O), thiocarbonyl (C=S), and aromatic carbons[22].

b. FT-IR Analysis

The FT-IR spectrum of the free ligand (NTB) is presented in Figure 3, displaying characteristic absorption bands at: ν(N–H and O–H): 3251–3460 cm^{-1}, ν(C=O): 1597 cm^{-1}, and ν(C=S): 1242 cm^{-1}.

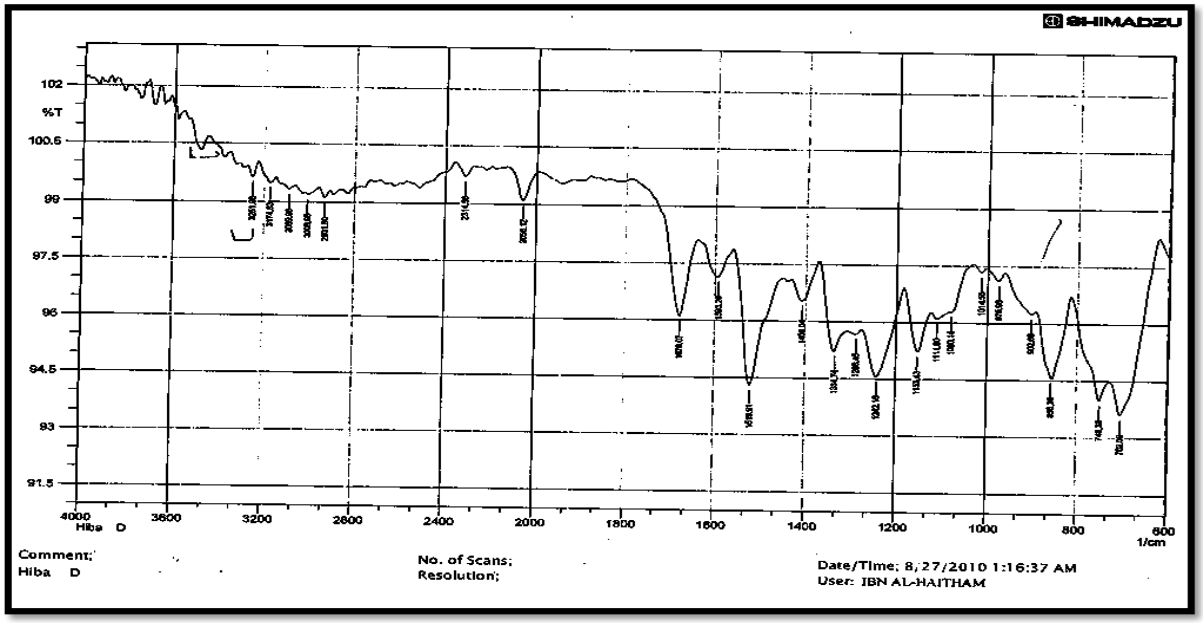


Figure 3. The FT-IR spectrum of Ligand (NTB)

Upon complexation, noticeable shifts and new bands appear in the metal complexes ' spectra. For example, the FT-IR spectrum of the cobalt(II) complex (Figure 4) reveals shifts in the ν(COO$^-$) symmetric and asymmetric stretching vibrations, confirming coordination through the carboxylate group[20]. Additionally, new bands observed in the 400–500 cm^{-1} region are attributed to M–O and M–N bond vibrations, providing further evidence of metal-ligand interaction.

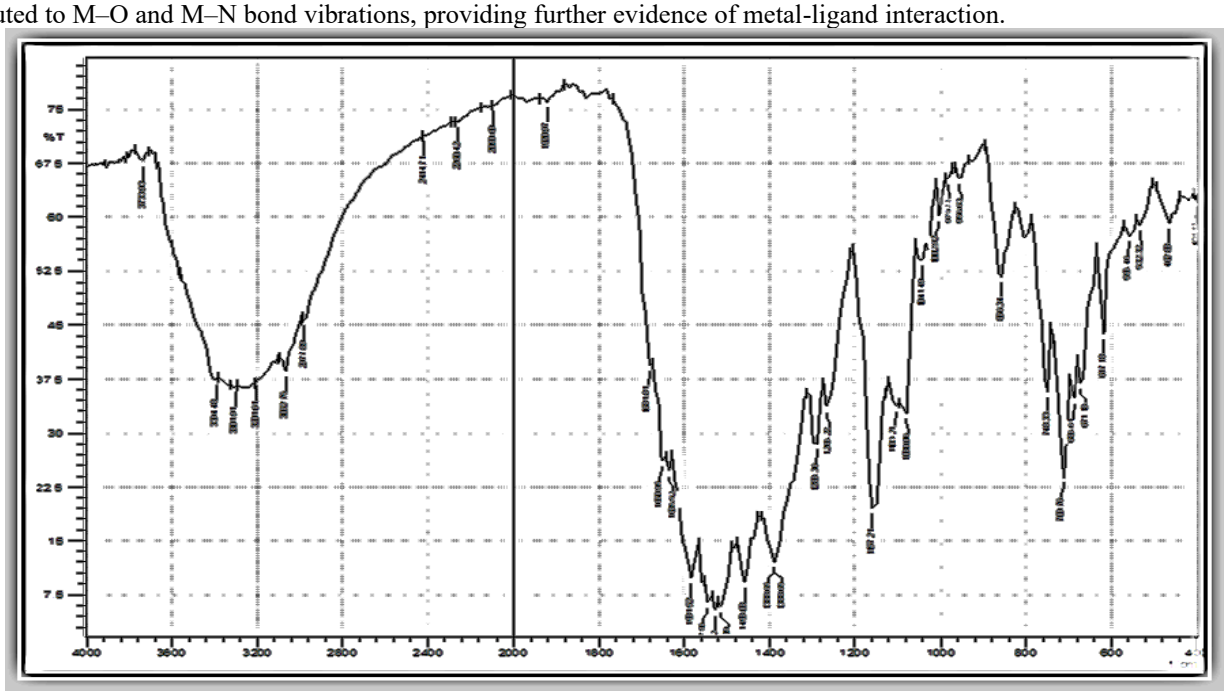


Figure 4. The FT-IR spectrum for the cobalt complex

The key FT-IR frequencies for all complexes are summarized in Table 1, supporting the coordination through amide, carboxylate, and thioamide functionalities.

Table 1. Some FT-IR frequencies in (cm-1) for (NTB) and its metal complexes

Compound	υ(N-H)	υ(COO)sym	υ(COO)asym	υ(C=O)	υ (C=S)	υ(M-O)	υ (M─N)
(NTB)	3251 (m)	1334(s)	1678 (m)	1597 (s)	1242 (s)	-	-
[Mn(NTB)2]	3375 (m)	1474 (s)	1623 (m)	1581 (w)	1249 (s)	424 (m)	443 (w)
[Co(NTB)2]	3358 (m)	1467 (m)	1627 (s)	1588 (m)	1265 (s)	438 (m)	439 (w)
[Ni(NTB)2]	3344 (b)	1498 (m)	1587 (m)	1585 (m)	1256(s)	432 (w)	462 (w)
[Cu(NTB)2]	3300 (b)	1488 (m)	1605 (m)	1587 (m)	1257 (s)	478 (m)	532 (w)
[Zn(NTB)2]	3362 (m)	1480 (m)	1589 (s)	1586 (m)	1256 (s)	428 (w)	443 (m)
[Cd(NTB)2]	3348 (b)	1493 (s)	1608 (m)	1589 (w)	1250 (m)	416 (m)	445 (m)
[Hg(NTB)2]	3340 (b)	1446 (m)	1543 (s)	1584 (m)	1253 (s)	435 (m)	493 (m)

b = browed, w = weak, s = strong, m medium

The FT-IR spectrum of NTB exhibits characteristic absorption bands for NH/OH stretching (~3251 cm^{-1}), C=O (1597 cm^{-1}), and C=S (1242 cm^{-1}) functionalities. Upon complexation, shifts in the NH and COO– stretching bands and the appearance of new bands assigned to M–O and M–N (in the 416–493 cm^{-1} region) confirm ligand coordination through the oxygen of the carboxylate and nitrogen of the amide groups. These shifts are summarized in Table 2 and support a bidentate coordination mode.

c. UV–Vis Spectroscopy

Figure 5 shows the UV-Vis spectrum of NTB, exhibiting π→π* at 34013 cm^{-1} and n→π* at 28985 cm^{-1}, which are characteristic of conjugated organic systems and agree with standard interpretations of such transitions[21]. The metal complexes display d–d transitions and ligand field (L.F.) bands. Notably: [Mn(NTB)$_2$] shows a band at 10493 cm^{-1} ($^6A_1 \rightarrow {}^4T_2$), [Co(NTB)$_2$] has multiple transitions including 20366 cm^{-1} ($^4A_2 \rightarrow {}^4T_1P$), [Cu(NTB)$_2$] shows $2B_1g \rightarrow 2A_1g$ transition at 18450 cm^{-1} (Figure 6), and [Zn, Cd, Hg] complexes primarily show charge transfer (C.T.) bands. Full spectral data and assignments are given in Table 2.

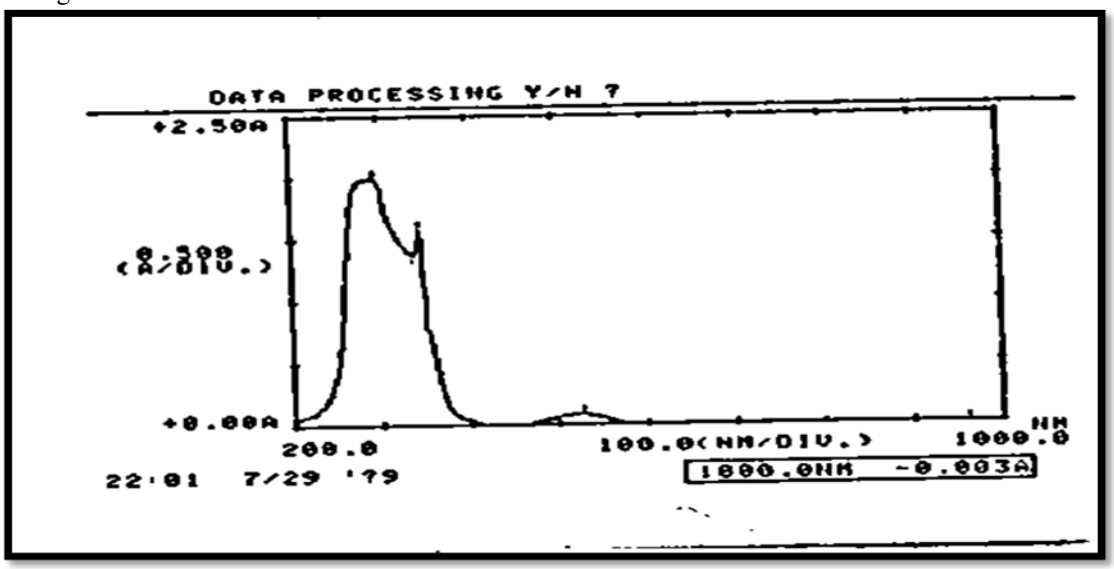


Figure 5. UV-VIS spectrum of the Ligand (NTB)

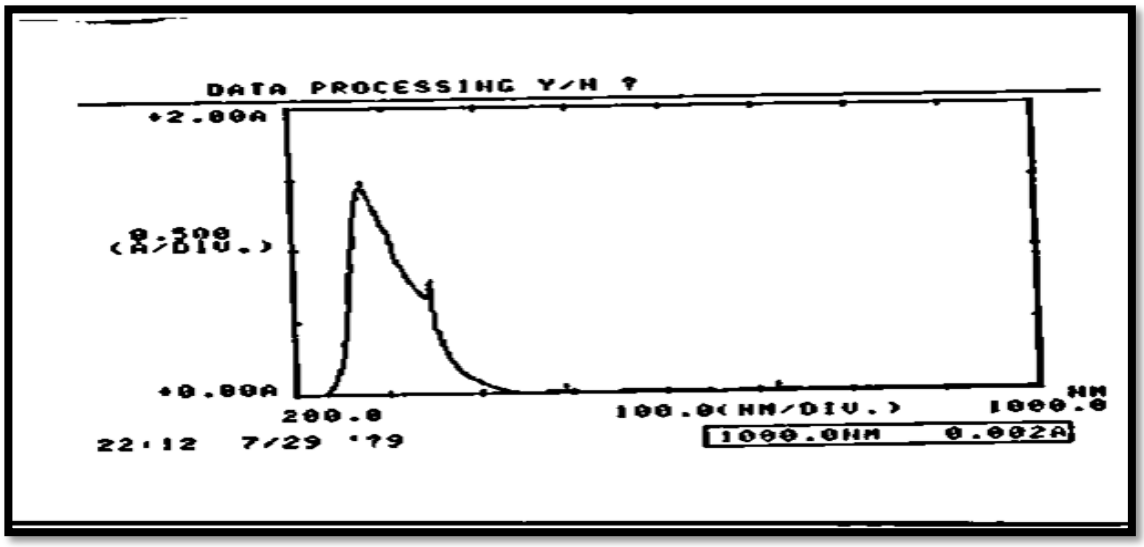


Figure 6. The UV-VIS spectrum of the Co complex

Table 2. The electronic peaks, transitions, and structure geometries of (NTB) and its complexes

Compound	λ (nm)	ύ (cm^{-1})	ABC	ε_{max}	Transition
(NTB)	294 345	34013 28985	2.000 1.582	2000 1582	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$
$[Mn(NTB)_2]$	272 953	36764 10493	0.936 0.025	936 25	L.F $^6A_1 \rightarrow {}^4T_{2(G)}$
$[Co(NTB)_2]$	275 491 710	36363 20366 14084	1.440 0.025 0.018	1440 25 18	L.F $^4A_{2(F)} \rightarrow {}^4T_{1(P)}$ $^4A_{2(F)} \rightarrow {}^4T_{1(F)}$
$[Ni(NTB)_2]$	297 350 504 571	33670 28571 19841 17513	2.226 1.500 0.045 0.025	2226 1500 45 25	L.F $^3T_{1(F)} \rightarrow {}^3T_{1(P)}$ $^3T_{1(F)} \rightarrow {}^3A_{2(F)}$ $^3T_{1(F)} \rightarrow {}^3T_{2(F)}$
$[Cu(NTB)_2]$	297 542 853	33670 18450 11723	2.184 0.020 0.018	2184 20 18	L.F $^2B_{1g} \rightarrow {}^2A_{1g}$ $^2B_{1g} \rightarrow {}^2B_{2g}$
$[Zn(NTB)_2]$	290	34482	2.248	2248	C.T
$[Cd(NTB)_2]$	275	36363	1.091	1091	C.T
$[Hg(NTB)_2]$	298	33557	2.301	2301	C.T

C.T = Charge transfer , L.F=ligand field

Electronic spectra (**Table 2**, Figures 5 and 6) of the ligand show π→π* and n→π* transitions. The metal complexes exhibit additional d–d transitions characteristic of their coordination geometries. For instance:

- **$[Mn(NTB)_2]$** shows a band at 10,493 cm^{-1} corresponding to $^6A_1 \rightarrow {}^4T_1(G)$, indicating a high-spin tetrahedral geometry.
- **$[Co(NTB)_2]$** displays multiple bands, suggesting a tetrahedral structure, while
- **$[Ni(NTB)_2]$** exhibits bands assigned to $^3T_1(F) \rightarrow {}^3A_2(F)$, consistent with tetrahedral coordination.
- **$[Cu(NTB)_2]$** exhibits bands supporting a square planar geometry, distinguishing it from the rest.
- Complexes of **Zn^{2+}, Cd^{2+}, and Hg^{2+}** show only charge transfer transitions due to their d^{10} configurations, confirming a non-degenerate ground state.

d. Physical Properties and Conductivity

As summarized in Table 3, the resulting metal complexes exhibit distinct colors and melting points, indicating successful complexation. All complexes are thermally stable and soluble in polar organic solvents such as DMSO and DMF. Molar conductivity measurements revealed that all metal complexes behave as non-electrolytes in DMSO, suggesting the absence of ionic dissociation in solution, thus supporting the proposed neutral complex formulas $[M(NTB)_2]$.

Table 3. Physical properties of (NTB) and its metal complexes

Compound	M.wt (gm/mol)	Color	M.p °C or dec.	M% Calculation (Found)	Molar condu. $Ohm^{-1}Cm^2mol^{-1}$	μeff (B.M)
(NTB)	345	Yellow	240-242	-	-	-
$[Mn(NTB)_2]$	742.94	Pale yellow	300-298	7.39 (744)	11	5.94
$[Co(NTB)_2]$	746.93	Green	320-322	7.89 (7.56)	5.12	4.88
$[Ni(NTB_2]$	746.71	deep green	336-338	7.86 (7.45)	16.13	2.89
$[Cu(NTB)_2]$	751.55	green	338-340	8.46 (8.56)	18.8	1.73
$[Zn(NTB)_2]$	753.41	Yellow	288-290	8.68 (8.59)	15.19	0
$[Cd(NTB)_2]$	800.4	Pale yellow	350dec	14.04 (13.85)	19.2	0
$[Hg(NTB)_2]$	888.6	Pale yellow	350 dec.	22.57 (22.26)	14.22	0

3. Results and Discussion

The synthesized ligand (NTB) AND its metal complexes with divalent transition metals (Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, and Hg^{2+}) were successfully characterized using various analytical and spectroscopic techniques. The obtained results provide comprehensive insight into the structural, spectroscopic, and electronic behavior of these complexes.

3.1. Complex Formation in Solution

Moler retio studies using UV-Visible spectroscopy confirm a 1:2 (metal: ligand) stoichiometry in solution, consistent with the solid-state findings[23]. These results affirm the reproducibility and stability of the complexes in both states.

3.4. Proposed Structures

Based on spectral data, magnetic measurements, and molar conductance values, tetrahedral geometries are proposed for all complexes except for the copper complex, which exhibits a square planar structure (**Figures 7 and 8**). These structural assignments are supported by ligand field theory and observed transitions[24,25].

4. Conclusion

In this study, a new ligand,2-(3-(4-nitrobenzoyl)thioureido)benzoic acid (NTB), was successfully synthesized, and its complexes with divalent metal ions (Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, and Hg^{2+}) were prepared and characterized. Various spectroscopic techniques, including FT-IR, UV-Vis, ^1H and ^{13}C-NMR, alongside molar conductivity, magnetic susceptibility, and elemental analysis, confirmed the proposed structures. The results revealed that the ligand acts as a bidentate donor, coordinating through both nitrogen and oxygen atoms. All synthesized complexes exhibited a tetrahedral geometry, except the copper complex, which adopted a square planar configuration.

The findings suggest strong metal-ligand interactions and structural stability of the complexes. This opens promising directions for exploring their biological activities or catalytic properties in further studies. The consistent coordination behavior across different metal ions highlights the ligand's versatility and potential for designing new coordination compounds.

5. **Acknowledgments**

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6. Declarations

6.1 Ethics approval and consent to participate

Not applicable.

6.2 Consent for publication
Not applicable.

6.3 Availability of Data and Materials
Data will be provided upon receiving a valid request.

6.4 Conflicts of interest
The author declares that there is no conflict of interest

6.5 Funding
The author declares that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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تحضير وتشخيص بعض المعقدات الفلزية الجديدة مع الليكاند (2-(3-(4-nitrobenzoyl)thioureido)benzoic acid (NTB)

دينا حميد زيدان

قسم الكيمياء، كلية التربية للبنات، جامعة الانبار، الانبار، العراق

المستخلص

يشكل وجود المجاميع الوظيفية النشطة في تركيبة الليكاندات عاملًا رئيسيًا في تحسين تفاعلها مع الأيونات الفلزية التي تساعد على تكوين معقدات ذات فعالية متعددة. تم تحضير ليكاند جديد يُعرف باسم حمض 2- (3- (4- نتروبنزويل) ثيوريدو) بنزويك (NTB) من خلال تفاعل 4-نتروبنزويل أيزوثايوسيانات مع حمض الأنثرانيليك بنسبة مولية 1:1. تم تأكيد بناء الليكاند باستخدام تقنيات التحليل العنصري، والأطياف تحت الحمراء(FT-IR) ، والأشعة فوق البنفسجية المرئية (UV-Vis)، وطيفي الرنين النووي المغناطيسي للبروتون والكربون (^{1}H-NMR) و(^{13}C-NMR) بعد ذلك، تم تحضير عدد من المعقدات الفلزية عن طريق تفاعل NTBمع أيونات معادن ثنائية التكافؤ تشمل: الزئبق، الكادميوم، الخارصين، النحاس، النيكل، الكوبالت، والمنغنيز. جرى تشخيص هذه المعقدات باستخدام تقنيات التوصيلية المولارية، الحساسية المغناطيسية، الأطياف تحت الحمراء، الأشعة فوق البنفسجية، والامتصاص الذري. أظهرت النتائج أن جميع المعقدات تمتلك بنية رباعية السطوح حول أيون الفلز، باستثناء معقد النحاس الذي اتخذ هيكلًا مستويًا مربعًا. وقد تم تحديد النسبة بين الفلز والليكاند في جميع المعقدات لتكون 1:2، مما يدعم الصيغة العامة المقترحة[$M(NTB)_2$] .