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Original Article

Synthesis of the Complex Compounds of Zn (II) and Co (II) with Isoniazid (Pyridine-4-Carbohydrazide)

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Abstract *- In this paper, the optimal conditions for the complexation of isoniazid (pyridine-4-carbohydrazide) with some 3d-metal Zn $(NO_3)_2$ and Co $(NO_3)_2$ salts were studied. In this case, complex compounds $[Zn(L)_2]$ and $[Co(L)_2]$ were synthesized in a 2:1 ratio of Ligand and Metal and a weakly acidic pH=5 environment. The composition and structure of the synthesized complex were studied using modern physico-chemical methods. In particular, the structure of the obtained compound was studied using IR-Fourier spectroscopy, mass spectrometry and elemental analysis methods, and the chemical structure of complex compounds was determined. According to the results of TGA and DTA analysis, it was confirmed that the synthesized complex compounds containing $[Me(L)_2]$ are stable and specific to the ligand.*

Keywords *- Isoniazid, Zinc nitrate, Cobalt nitrate, Ethanol, Chromatomass spectrum, Scanning electron microscope, IR spectrum.*

1. Introduction

The significance of N-heterocyclic carbene (NHC) ligands in organic and organometallic chemistry is steadily growing [1]. It has been highlighted that the notable impact of NHC ligands on transition metal catalysis arises from their strong sigma donor properties, a feature recognized even in earlier literature reviews. It is posited that in cross-coupling reactions, in particular, the robust M-NHC bond serves to inhibit the degradation of molecular catalysts, thereby stabilizing the higher oxidation states crucial for various catalytic processes. This research involves synthesizing bis(NHC)PdX_2 complexes substituted with 2-methyl-1,4-benzodioxan and evaluating their catalytic performance in direct arylation reactions. The preparation of these complexes entails the transformation of 2-methyl-1,4-benzodioxan-substituted Ag(I)NHC complexes through a transmetallation approach [2]. Nickel (II) and cobalt (II) complexes containing optically active diaminodioxime (H_2L), a derivative of 3-carene, were synthesized. These complexes have the compositions $[Ni(H_2L)\ NO_3]NO_3$ (I), $Ni(H_2L)\ Cl_2$ (II), [Ni (HL)] $ClO_4 \cdot H_2O$ (III), and $Co(H_2L)\ Cl_2$ (IV). X-ray diffraction analysis revealed that the structures of paramagnetic compound I and diamagnetic complex III are ionic in nature [3]. Two acetyl hydrazones, specifically benzoyl acetone nicotinoyl hydrazone (H2L1) and acetoacetanilide benzoyl hydrazone (H2L2), were synthesized. Additionally, the $MoO_2L1 \cdot MeOH$ solvate complex was prepared. The structures of these compounds were analyzed using single-crystal X-ray diffraction and IR spectroscopy techniques [4]. The dissociation constants of 2-furancarboxylic and 2-furylacrylic acids, as well as the stability constants of their complexes with Pr^{3+}, Nd^{3+}, Sm^{3+}, Eu^{3+}, Gd^{3+}, Tb^{3+}, Ho^{3+}, Er^{3+}, and Yb^{3+} ions, were determined in an aqueous ethanol solution (with a volume ratio of 5:1). This determination was carried out through pH-potentiometric titration. It was observed that complex formation occurred within the pH range of 3.0–6.5. Moreover, the stability constants of the complex compounds exhibited an increasing trend with higher values of the protonation constant of the ligands [5]. Two solvate complexes, denoted as $MoO_2L1 \cdot MeOH$ (where H_2L1 is isonicotinoyl hydrazone acetylacetone) (I) and $MoO_2L_2 \cdot Me_2SO$ (where H_2L_2 is benzoyl hydrazone benzoyl acetone) (II), were synthesized. Their structures were elucidated through X-ray diffraction analysis [6]. New copper (II), cobalt (II), and nickel (II) complexes were synthesized using 1-[4-(pheny diazenyl) phenyl diazenyl]-naphthalen-2-ol and 1-[4-methyl-2-(4-thyl phenyl diazenyl) phenyldiazenyl]naphthalen-2-ol.

These complexes were prepared through both chemical and electrochemical methods [7]. New Co (II), Ni (II), and Cu (II) complexes with 4-(3-hydroxyphenyl)-1,2,4-triazole (denoted as L) have been synthesized. The compositions of these complexes are as follows:

$Co_3L_6(H_2O)_5(C_2H_5OH)_6 \cdot 2H_2O \cdot C_2H_5OH$, $Ni_3L_6(H_2O)_{66} \cdot 2H_2O$, $Co_3L_6(H_2O)_{66} \cdot 2H_2O$ (M = Co^{2+}, n = 2), $Ni_3L_6(H_2O)_{66} \cdot 2H_2O$ (M = Ni^{2+}, n = 2), $Cu_3L_6(H_2O)_{66}$ (M = Cu^{2+}, n=0) [8].

2-Oxopropanoic acid reacts with N-(prop-2-en-1-yl) hydrazine carbothioamide in ethanol in a 1:1 mole ratio to produce the thiosemicarbazone compound, denoted as H_2L[9]. This paper investigates the optimal conditions for synthesizing complex compounds of zinc (II) and cobalt (II) metals with aciclovir, a drug commonly used against viruses. The synthesis process was conducted at a temperature range of 50-60°C, with a mixing duration of two hours.

Additionally, all reactions were performed under a pH of 5[10]. Four new coordination compounds of Cu(II) were synthesized using ethyl-5-amino-1-methyl-1H-pyrazole-4-carboxylate (L) and different co-ligands. In the reaction, warm methanolic solutions of $CuX_2 \cdot nH_2O$ (where X = Cl with n =2, X = Br with n = 0, X = NO_3 with n = 3) were combined with the ligand in a mole ratio of 1:2. This resulted in the formation of bis(ligand) complexes: $Cu(L)_2Cl_2$ (1), $Cu(L)_2Br_2$ (2), and $Cu(L)_2(NO_3)_2$ (3) [11,12].

1.1. The Aim of the Work

Synthesis of new complex compounds based on Zn^{+2} and Co^{+2} with isoniazid, which is one of the organic ligands whose molecule contains nitrogen and oxygen, consists in studying the physico-chemical analysis, composition and structure of synthesized complex compounds.

2. Experimental Part

2.1. Materials

In this study, analytically pure isoniazid and nitrate salts of Zn(II) and Co(II) were used for the synthesis of complex compounds. Organic reagents and solvents used in the experiment were cleaned and dried by certain methods.

2.2. Methods

Chromatomass-spectrum analysis of the synthesized complex compound 6420 Triple Quard LC/MS (Agilent Technologies, USA). It was carried out using a mass spectrometer. Physico-chemical analysis, composition and structure of the synthesized complex were studied using an IR-spectrum device (IK-Fourier, SHIMADZU, Japan) in the range of 4000-600 cm^{-1}. In addition, the synthesized compounds were studied using a MIRA 2 LMU scanning electron microscope equipped with an INCA Energy 350 energy dispersive microanalysis system. The analysis capability of the microscope is 1 nm, and the sensitivity of the INCA Energy detector is equal to 133 ev/10mm^2, which allows the analysis of elements from beryllium to plutonium.

2.3. Synthesis of Complex Compound of Zn(II) with Isoniazid

Synthesis of the complex combination of Zn^{+2} ion with isoniazid was carried out as follows: $Zn(NO_3)_2$ was prepared in a volume sufficient for synthesis from a 0.01mol/l aqueous solution and brought to pH=5 using HNO_3 to prevent hydrolysis. The medium pH=5 was determined visually using a universal indicator. To obtain the complex compound intended to be synthesized, solutions of isoniazid $C_6H_7N_3O$ (Table 1) were prepared in 96% ethyl alcohol and stirred in a magnetic stirrer (t=400C) for 60 minutes. The color of the solution is white under the influence of $Zn(NO_3)_2$ and isoniazid. The resulting mixture was evaporated in an electric furnace at 80-90 ^{0}C until it became a wet salt. In cases of increased pH values and increased acidity, it was regularly controlled so that it did not exceed pH=5 using 0.01M NH_4OH. The reason is that they can be removed during the heating process. The residue at the bottom of the vessel was dried in a desiccator for 12 days for appropriate analyses.

2.4. Synthesis of Complex Formation of Co(II) with Isoniazid

The synthesis of the complex compound of Co^{+2} ion with isoniazid was carried out as follows: from a 0.01mol/l aqueous solution of $Co(NO_3)_2$, sufficient volume for synthesis was prepared and brought to pH=5 using HNO_3 to prevent hydrolysis. The medium pH=5 was determined visually using a universal indicator. To obtain the complex compound intended to be synthesized, solutions of isoniazid $C_6H_7N_3O$ (Table 2) were prepared in 96% ethyl alcohol and stirred in a magnetic stirrer (t=400C) for 60 minutes. Under the influence of Co $(NO_3)_2$ and isoniazid, the color of the solution is pinkish-red.

The resulting mixture was evaporated in an electric furnace at 80-90 ^{0}C until it became a wet salt. In cases of increased pH values and increased acidity, it was regularly controlled so that it did not exceed pH=5 using 0.01M NH_4OH. The reason is that they can be removed during the heating process. The residue at the bottom of the vessel was dried in a desiccator for 12 days for appropriate analyses [13,14].

3. Results and Discussion

3.1. IR-Analyses

Physicochemical analysis, composition and structure of the synthesized complex compounds containing $[Zn(L)_2]$ and $[Co(L)_2]$ were studied using an IR-spectrum device (IK-Fourier, SHIMADZU, Japan). According to the results of the analysis, absorption frequencies caused by the valence vibrations of the OH- group were observed in the 3300.20 cm^{-1} area of the spectrum, while the valence vibrations of the NH group at 3107.32 cm^{-1}, in the 1662.64 cm^{-1} area > The valence vibration related to the C=O group and the vibrational frequencies characteristic of the $–NH_2$ bond at 1633.71 cm^{-1} were also observed. According to the results of the analysis, the absorption frequencies caused by the valence vibrations of the OH- group were observed in the 3454.51 cm^{-1} area of the spectrum, while the valence vibrations of the NH group were observed at 3261.63 cm^{-1}, in the 1664.57 cm^{-1} area > The valence vibration related to the C=O group, the scissor characteristic of the $–NH_2$ bond at 1604.77 cm^{-1}, and the vibrational frequencies characteristic of the Zn-N bond at 705.95 cm^{-1} were also observed[15-17].

Table 1. Zn^{+2} and isoniazid solutions (in volume and concentration)

	Zn^{+2} 0,01 mol/l	Isoniazid ($C_6H_7N_3O$)	∑
1	V=50ml	0,02mol/l, V=50ml	100ml
2	V=50ml	0,04mol/l, V=50ml	100ml
3	V=50ml	0,06mol/l, V=50ml	100ml

Table 2. Co^{+2} and isoniazid solutions (in volume and concentration)

	Co^{+2} 0,01 mol/l	Izoniazid ($C_6H_7N_3O$)	∑
1	V=50ml	0,02mol/l, V=50ml	100ml
2	V=50ml	0,04mol/l, V=50ml	100ml
3	V=50ml	0,06mol/l, V=50ml	100ml

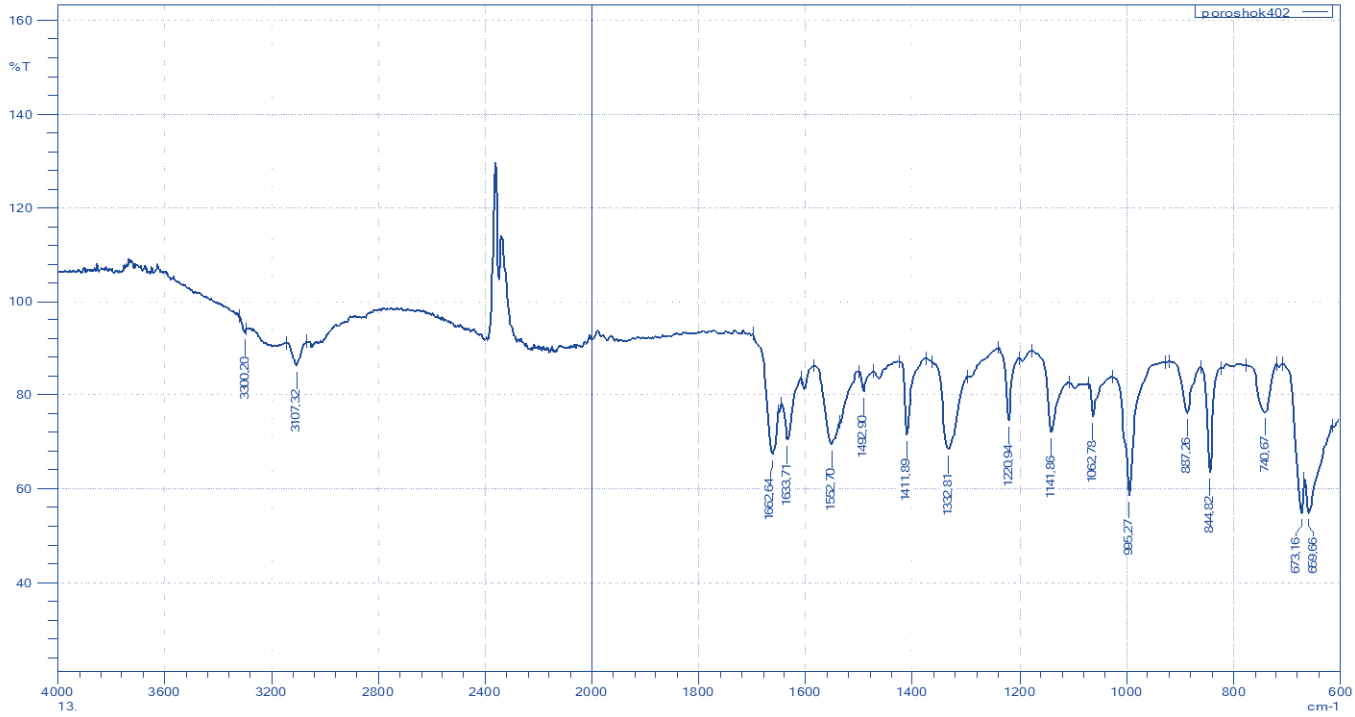

Fig. 1 IR spectrum analysis of the ligand

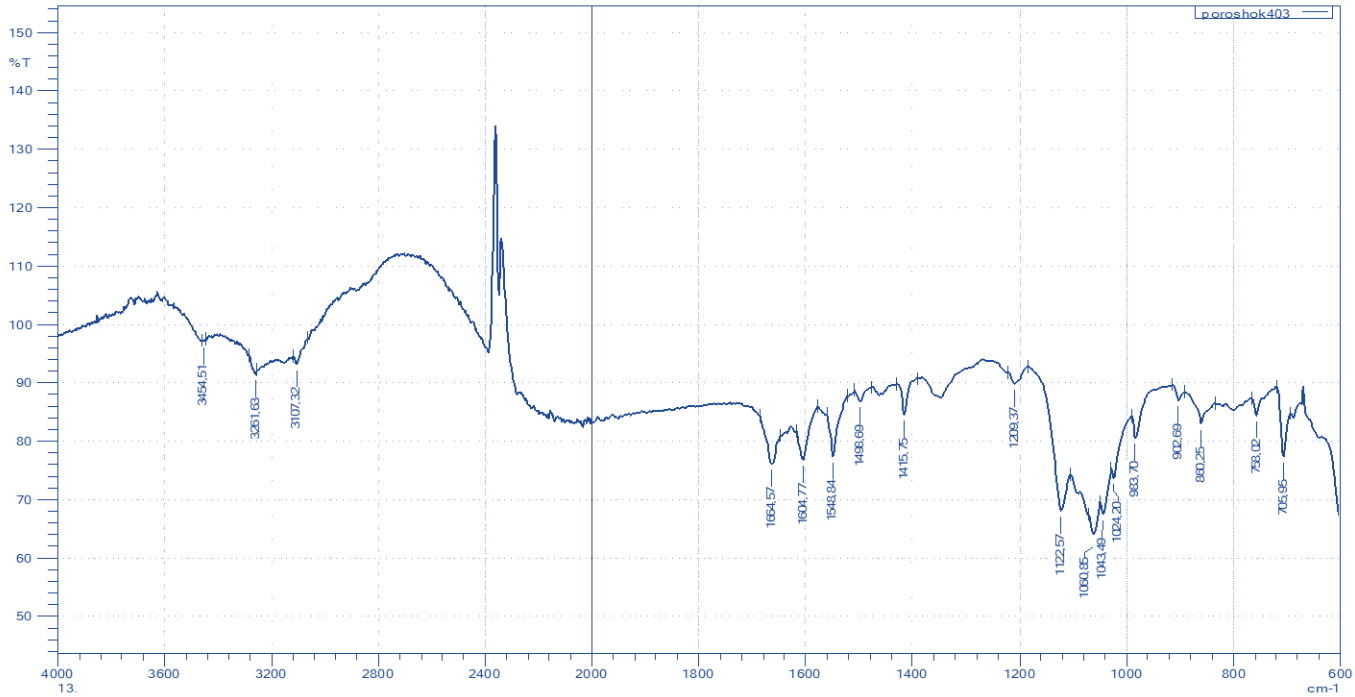

Fig. 2 IR spectrum analysis of complex compound $[Zn(L)_2]$

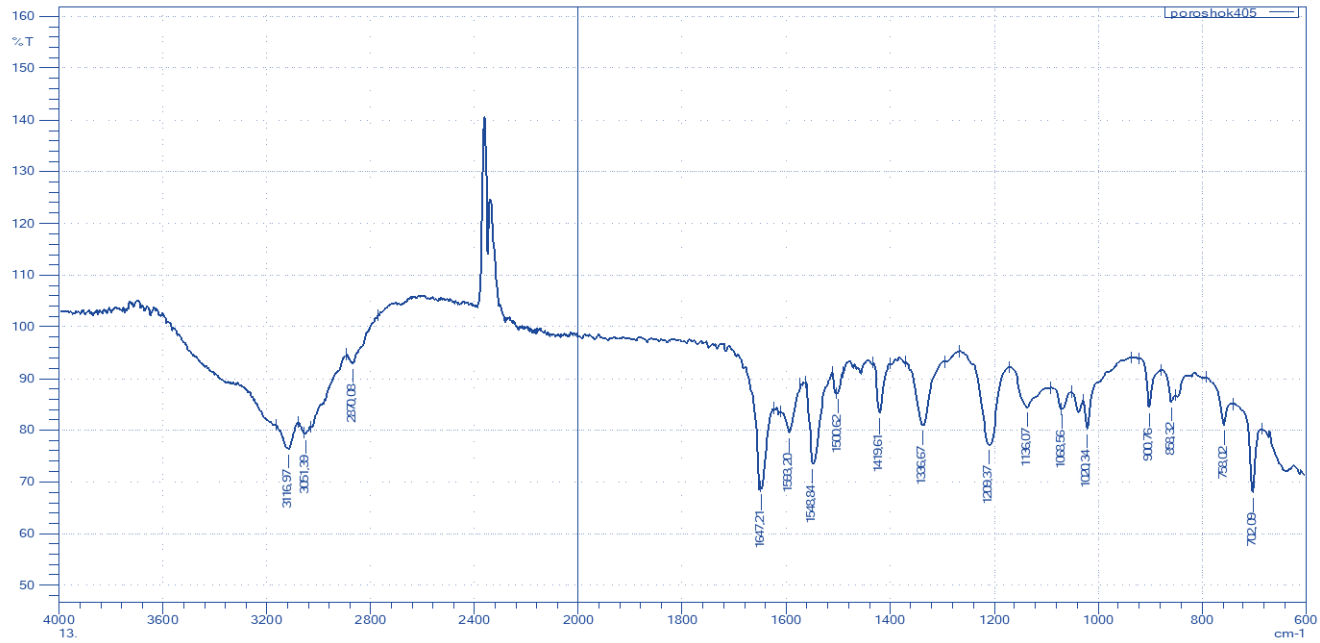

Fig. 3 IR spectrum analysis of $[Co(L)_2]$ complex compound

Table 3. IR spectrum analysis of ligand(isoniazid) and $[Zn(L)_2]$ complex

Vibrational frequencies in IR spectra, cm-1		Bonds
Ligand	[Zn $(L)_2$]	
3300,20	3454,51	OH
3107,32	3261,63	NH
1662,64	1664,57	>C=O
1633,71	1604,77	NH_2
-	705,95	Zn-N

Table 4. IR spectrum analysis of ligand(isoniazid) and $[Co(L)_2]$ complex

Vibrational frequencies in IR spectra, cm-1		Bonds
Ligand	[Co $(L)_2$]	
3300,20	3116,97	OH
3107,32	3051,39	NH
1662,64	1647,21	>C=O
1633,71	1593,20	NH_2
-	702,09	Co-N

According to the results of the analysis, absorption frequencies caused by the valence vibrations of the OH-group were observed in the 3116.97 cm^{-1} area of the spectrum, while the valence vibrations of the NH group at 3051.39 cm^{-1}, in the 1647.21 cm^{-1} area > The valence vibration of the C=O group, scissor vibration characteristic of the $–NH_2$ bond at 1593.20 cm^{-1}, and vibrational frequencies characteristic of the Co-N bond at 702.09 cm^{-1} were also observed.

3.2. Chromatomass-Spectrum Analysis

Chromatomass-spectrum analysis of the synthesized $Zn(L)_2$-containing complex was performed by 6420 Triple Quad LC/MS (Agilent Technologies, USA). It was carried out using a mass spectrometer [18-22]. The obtained chromato-mass spectrum analysis revealed that the molecular mass (m/z) of the synthesized complex is 337.6. This is indeed consistent with the calculated (m/z) 337.66 for $[(C_6H_6N_3O)_2Zn]$. Chromatomass-spectrum analysis of the synthesized complex compound containing $Co(L)_2$ was performed by 6420 Triple Quad LC/MS (Agilent Technologies, USA). It was carried out using a mass spectrometer [23]. The obtained chromato-mass spectrum analysis revealed that the molecular mass (m/z) of the synthesized complex is 331.2. This indeed matches the calculated (m/z) 331.21 for $[(C_6H_6N_3O)_2Co]$.

4. SEM-EDT-Analysis

The composition of the synthesized complex compound $[Zn(L)_2]$ was studied using the SEM-EDT method. It showed that the mass ratio of the elements in the synthesized complex is C- 42.6%, N- 24.8%, O- 9.4% and Zn- 19.3% Figure 6 shows that the synthesized complex compound has the gross formula $[(C_6H_6N_3O)_2Zn]$ [24,25]. The composition of the synthesized complex compound $[Co(L)_2]$ was studied using the SEM-EDT method. The mass ratio of the elements in the synthesized complex showed that C- 43.4%, N- 25.3%, O- 9.6% and Co- 17.7% in percent. Figure 8 shows that the synthesized complex compound has the gross formula $[(C_6H_6N_3O)_2Co]$ [26].

5. Thermogravimetric (TGA) and Differential Thermal Analysis

The thermal stability of the synthesized complex compound containing $[Zn(L)_2]$ was analyzed by differential-thermal and thermogravimetric methods using the device of the Japanese company SHIMADZU-DTG 60. It was studied by automatic recording of the derivatogram at the speed of 10 degrees/min, T-900, TG-200, DTA-1/10, and DTG-1/10 galvanometer sensitivity in the derivativeograph. Thermal analysis of $[Zn(L)_2]$ complex compound, 17.4 mg was taken for thermogravimetric analysis of complex compound, and the process was studied

at a temperature of 20-800 ^{0}C. The obtained analysis showed that the complex compound synthesized on the basis of Zn+2 and isoniazid takes place between 2 intensively decomposing temperatures. The first decomposition interval takes place in the temperature range of 97.61-203.45 0C, in which the mass change is 1.926 mg 11.068%; the second decomposition interval occurs in the temperature range of 324.26-470.84 0C, in which 21.959% of the mass that is, 3,821mg of mass is lost. No change is observed after 470,84 0C.

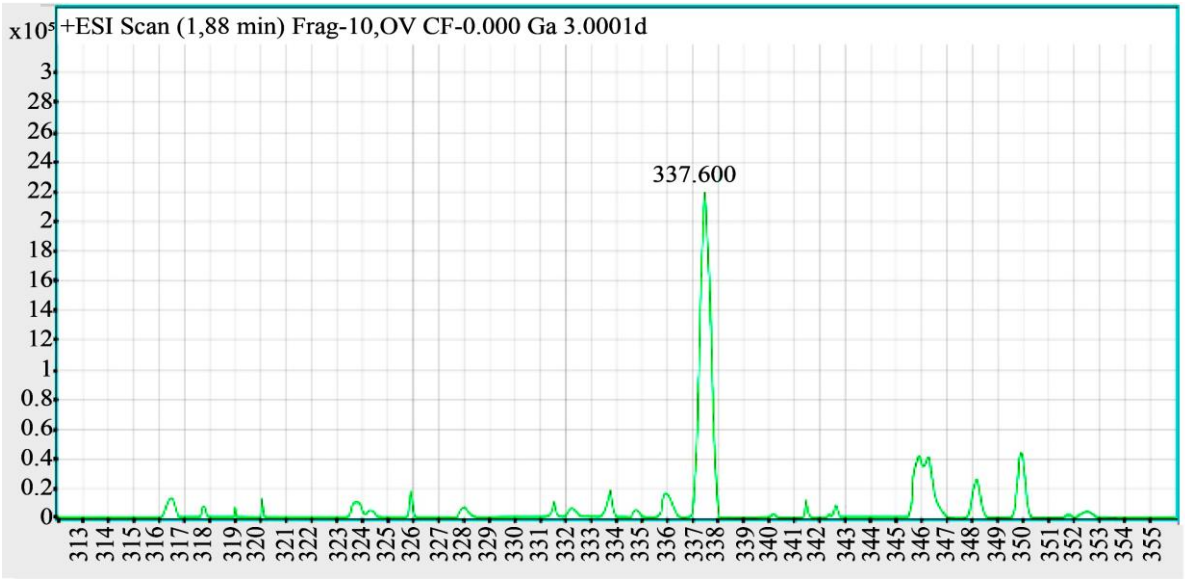

Fig. 4 Chromatomass-spectrum analysis of complex compound containing $Zn(L)_2$

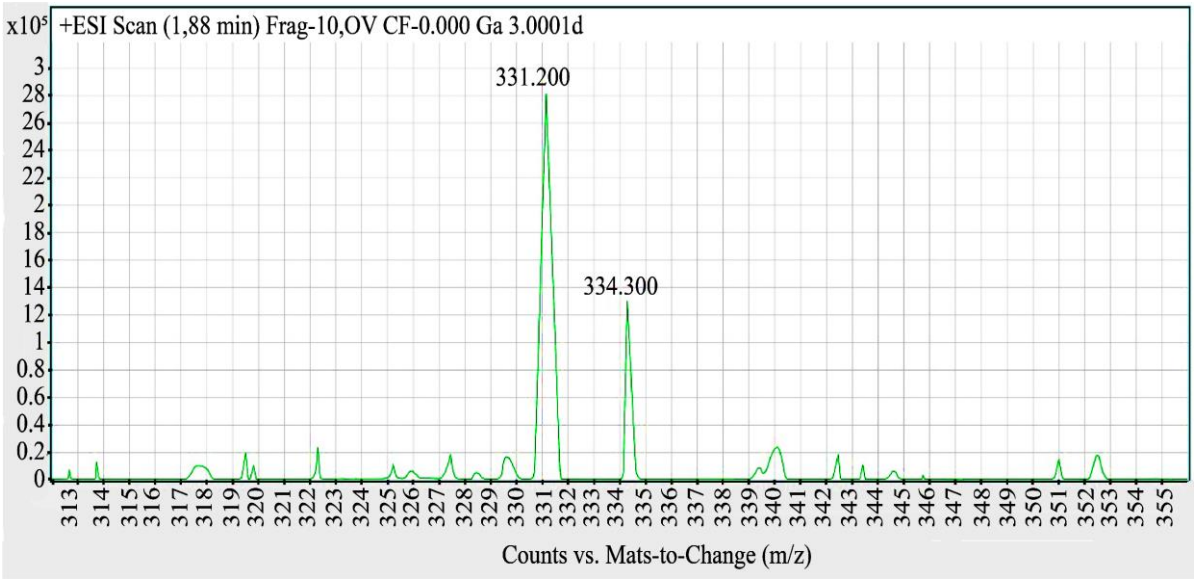

Fig. 5 Chromatomass-spectrum analysis of complex compound containing $Co(L)_2$

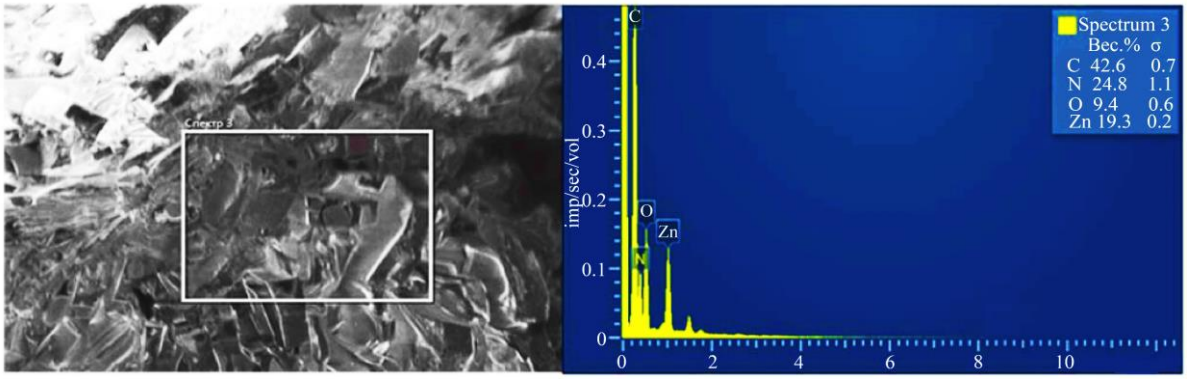

Fig. 6 Scanning electron microscopy (a) Elemental analysis (b) The complex compound $[(C_6H_6N_3O)_2Zn]$

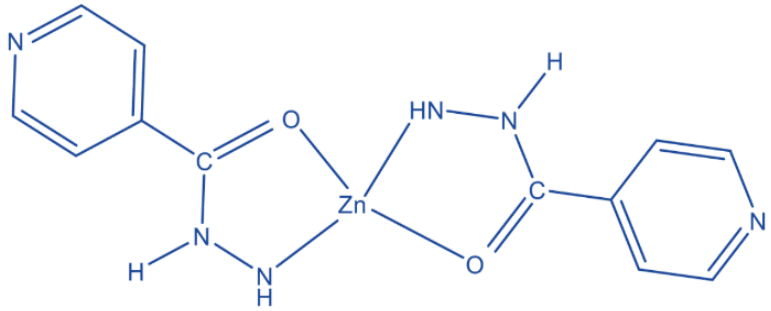

Fig. 7 Graphical structure of the complex compound $[Zn(L)_2]$

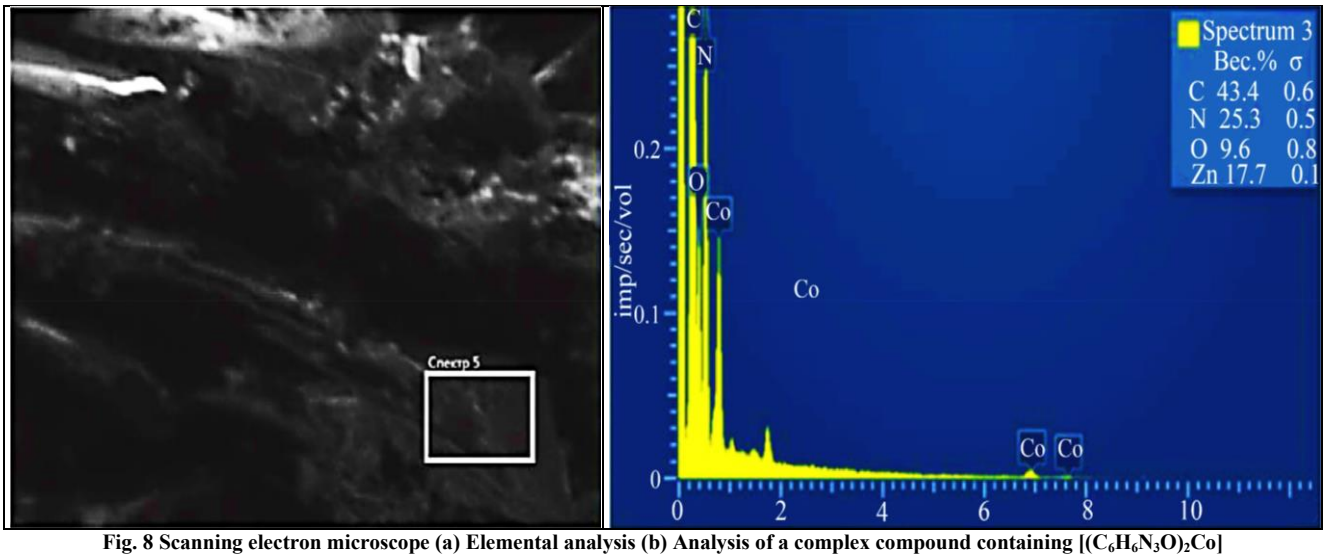

Fig. 8 Scanning electron microscope (a) Elemental analysis (b) Analysis of a complex compound containing $[(C_6H_6N_3O)_2Co]$

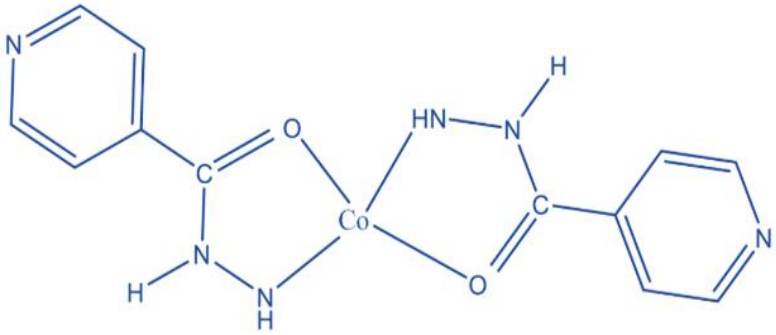

Fig. 9 Graphical structure of the complex compound $[Co(L)_2]$

The thermal stability of the synthesized complex compound containing $[Co(L)_2]$ was analyzed by differential-thermal and thermogravimetric methods on the device of the Japanese company SHIMADZU-DTG 60. It was studied by automatic recording of the derivatogram at the speed of 10 degrees/min, T-900, TG-200, DTA-1/10, and DTG-1/10 galvanometer sensitivity in the derivativeograph.

Thermal analysis of $[Co(L)_2]$ complex compound, 5.18 mg was taken for thermogravimetric analysis of complex compound, and the process was studied at a temperature of 20-600 0C. The obtained analysis showed that the complex compound synthesized on the basis of Co^{+2} and isoniazid takes place in the range of 3 intensively decomposing temperatures. The first decomposition range occurs at a temperature of 111.23-161.62 ^{0}C, where the mass change is 0.261 mg 5.038%. The second thermal decomposition takes place in the temperature range of 204.46 ^{0}C to 274.25 ^{0}C, in which 14.555% of the mass, i.e. 0.754mg of the mass, is lost.

The third thermal decomposition proceeds from 337,03 ^{0}C to 384,72 ^{0}C and is completed with a mass loss of 8,088 due to the decomposition of additives in the mixture. No change is observed after 384,72 ^{0}C [27-29].

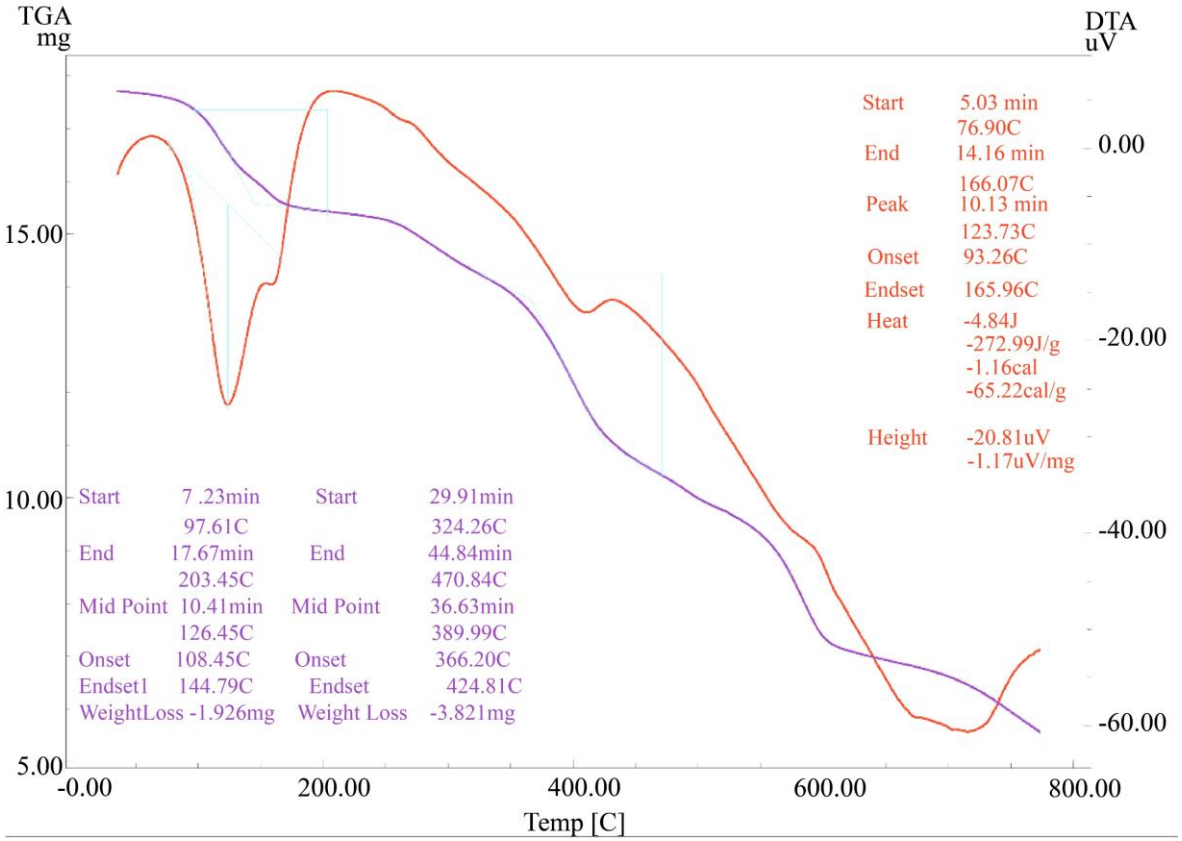

Fig. 10 Thermogravimetric (TGA) and differential thermal analysis (DTA) derivatogram of the complex compound [Zn(L)$_2$]

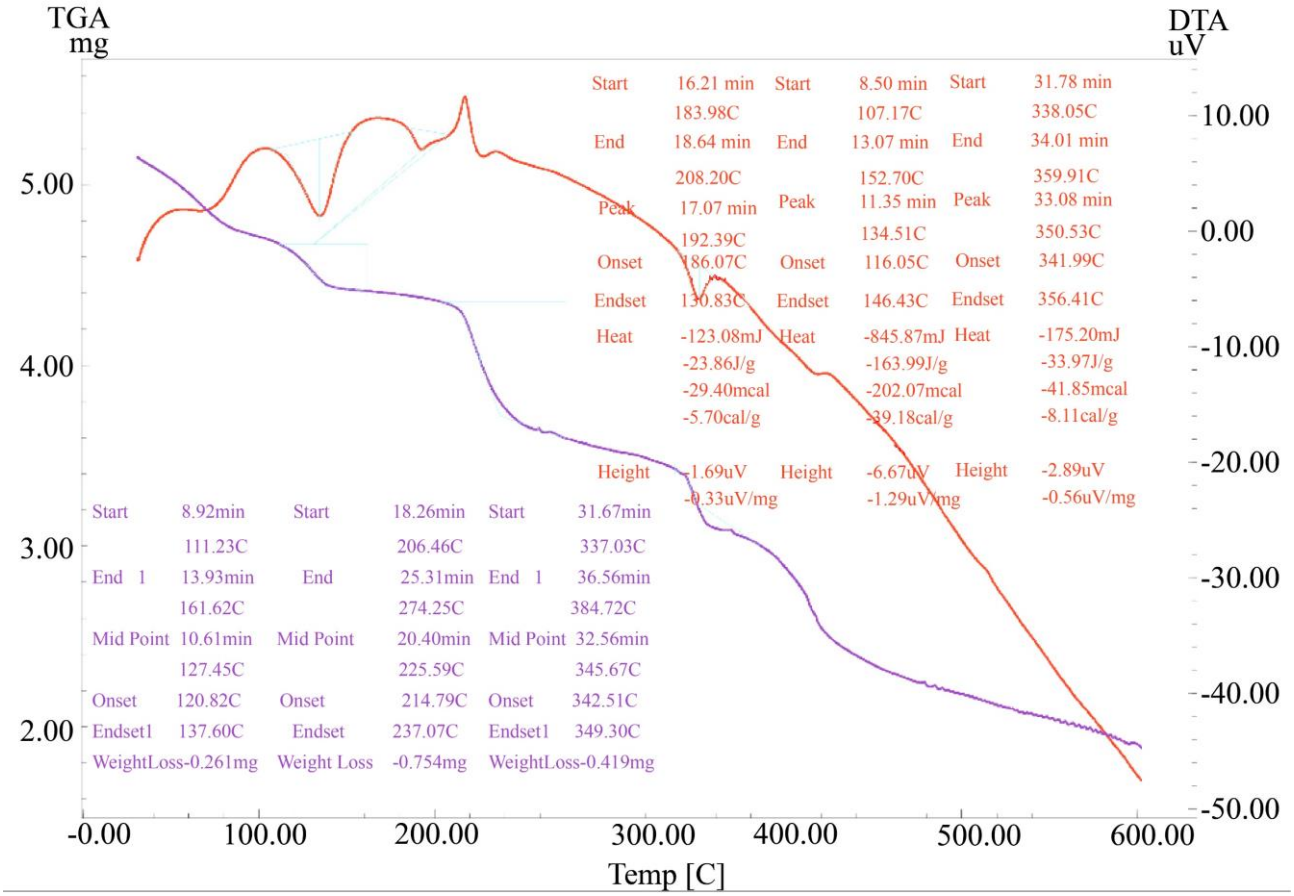

Fig. 11 Thermogravimetric (TGA) and differential thermal analysis (DTA) derivatogram of the [Co(L)$_2$] complex

6. Conclusion

Practical synthesis methods of complex compounds of isoniazid (pyridine-4-carbohydrazide) with nitrate salts of Zn(II) and Co(II) were developed, and complex compounds that are well soluble in water were isolated. Based on the results of modern physico-chemical research, it was found that in the synthesized complex compounds, Zn^{+2} and Co^{+2} ions interacted with the ligand molecule in a ratio of 1:2, forming a monoligand complex compound. Physico-chemical analysis, composition and structure of the synthesized complex compounds based on the results of thermal analysis, chromato-mass-spectroscopic analysis, scanning electron microscope and IR-spectroscopic analysis, the composition of the complex compounds correspond to the formulas $[(C_6H_6N_3O)_2Zn]$ and $[(C_6H_6N_3O)_2Co]$ was determined.

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Authors' Declaration

- We hereby confirm that all the Figures and Tables in the manuscript are ours.
- Ethical Clearance: The project was approved by the local ethical committee at the Termez State University

Authors' Contribution Statement

M.B.A: conducted the drafting, T. Kh.Kh: did the conception, design, drafting, U.I.A: was responsible for the acquisition of data; A.F.H: did the interpretation; A.B.Kh: participated in the conception, design, drafting, and all the authors took part in revision and proofreading.

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