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# SYNTHESIS AND STUDY OF COMPLEX COMPOUNDS OF 3-AMINO-1,2,4-TRIAZOLE WITH SOME 3D METAL ACETATES

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**ABSTRACT:** Methods of synthesis of complex compounds of 3-amino-1,2,4-triazole with Co (II), Ni (II), Cu (II) and Zn (II) salts were elaborated, solubility of synthesized complex compounds in different solvents was studied. In order to determine the coordination centers of the ligand with the central atom, the IR spectra of the synthesized complex compounds were studied. According to the results of IR-spectroscopic research, it was found that the ligand is coordinated through the first nitrogen atom in the triazole ring in the reactions of 3-amino-1,2,4-triazole complex formation. The absorption bands characteristic of the acetate acid ligand indicate that acetate anions are connected to metals monodentately through an oxygen atom. And also the results of the IR spectra were confirmed by the Raman spectrum method. In the Raman spectrum, absorption associated with the M-N bond was observed in the region of 314, 438 cm-1. As a result of thermal analysis, the rate of decrease in the mass of the sample, the mass of decomposition of complexes, liquefaction, and the thermal stability of complexes and final products were determined. Thermogravigrams of thermolysis of the complexes show that mass loss begins at 150°C and ends at 875°C with the formation of metal oxides. The amounts of elements in the synthesized ligands and complexes were analyzed using the SEM-EDX method. On the obtained data of analysis, the percentage ratios of the masses of the elements in the complex were calculated. In the synthesized complex compounds, two 3-amino-1,2,4-triazole molecules and acidoligands were united in the monodentate state and formed complex compounds with a tetrahedral structure. According to the results of physical and chemical studies, it was found that metal and ligand are combined in a 1:2 ratio at the formation of complex compounds.

KEYWORDS: 3-amino-1,2,4-triazole, raman spectrum, thermal analysis, infrared spectroscopy, scanning electron microscopy, ligand.

## INTRODUCTION

Recently, triazole derivatives have served as the most interesting objects for researchers due to their biological activity among organic substances. Since the discovery of triazoles, it's derivatives have been shown to have various biological activities [1], including anti-HIV [2], antifungal [3], antibacterial [4], anti-tuberculosis [5], anti-inflammatory [6], anticoagulant [7] and its anticancer properties [8]. It is reported in the literature that complex compounds synthesized on the basis of triazole derivatives enhance the pharmacodynamic and pharmacokinetic properties of drugs.

Derivatives of 1,2,4-triazole containing an amino group have attracted much attention owing to their biological activity, for example 3-amino-1,2,4-triazole is a triazole rich in amino group and electron density. It has a strong nucleophilic effect due to the preservation of its ring. Therefore, these compounds have a wide range of biological activity as antibacterial, antitumor, antiviral, fungicidal and anti-weed drugs [11-13]. 1,2,4-triazole-based compounds have chemotherapeutic effects, including potential antibacterial activity against drug-sensitive and drug-resistant pathogens[14].

Itraconazole, fulconazole, voriconazole, clotrimazole were used effecting in medicine against skin cancers. However, studying the properties and structure of 3-amino-1,2,4-triazole derivatives, developing methods for synthesizing new substances from them is important from a practical and theoretical point of view.

## EXPERIMENTAL TECHNIQUE

The ligand used in the work was 3-amino-1,2,4-triazole; for the synthesis of complex compounds, the following salts were used in the form of crystalline hydrates: acetates of Co(II), Ni(II), Cu(II) and Zn(II) - all salts of analytical grade.

Elemental composition (metals, carbon and nitrogen) was determined by Japan Jeol JSM-IT200LA analyzer. Duplicate elemental analysis was also carried out. The amount of metals in the synthesized compounds was determined according to [15]. Nitrogen was determined using the Dumas micromethod [16], carbon, and hydrogen were determined by combustion in a stream of oxygen. A comparison of the results obtained showed the agreement between the SEM-EDX data and the chemical methods of elemental analysis.

IR spectrums of the complexes were recorded on a Perkin Elmer FT-IR spectrophotometer in rangefrom 4000 to 400 cm-1 using KBr pellets.

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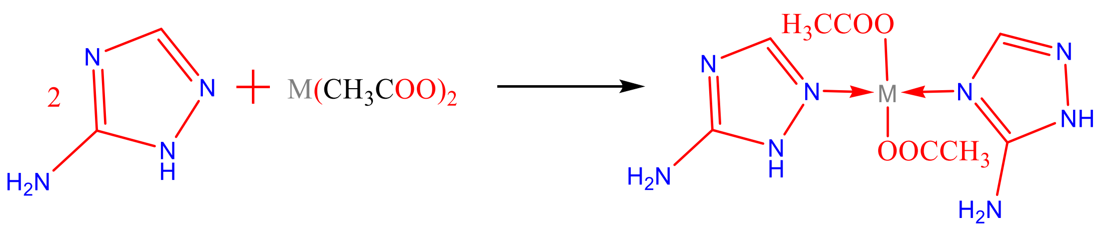
Thermogravimetric analysis (TGA) was performed on a Shimadzu simultaneous DTG-60A compositional analysis instrument from room temperature to 1000oC under N2 atmosphere at a heating rate of 5oC/min.

Preparation of complex compounds based on 3-amino-1,2,4-triazole (L) and ions of transition metals Co2+, Ni2+, Cu2+, Zn2+.

A hot solution of 0.001 mol of cobalt (II) acetate crystalline hydrate in 10 ml of ethanol was poured into a flask equipped with a reflux condenser. With constant stirring, a hot solution of 0.002 mol of 3-amino-1,2,4-triazole ligand in 10 ml of ethanol was added dropwise to a hot solution of the metal salt. The mixture was boiled for 1 hour, filtered while hot, and left to crystallize. After 3 days, a red precipitate formed, which was filtered, then washed several times with ethanol and dried in air. Yield 70%. Tpl. 180-181oC. Complex compounds of Ni(II), Cu(II) and Zn (II) acetates were obtained similarly.

Synthesis of complex acetate salts of Co(II), Ni(II), Cu(II) and Zn(II) with 3-amino-1,2,4-triazol is the following scheme:




## RESULTS AND THEIR DISCUSSION

The newly formed complexes were studied using a scanning electron microscope (SEM) (Fig. 1). The amounts of carbon, oxygen, nitrogen and metals in the complex compounds were determined [17]. Based on the data obtained as a result of SEM, a change in the microstructure of the ligands was observed due to the coordination of metal ions with the ligand.

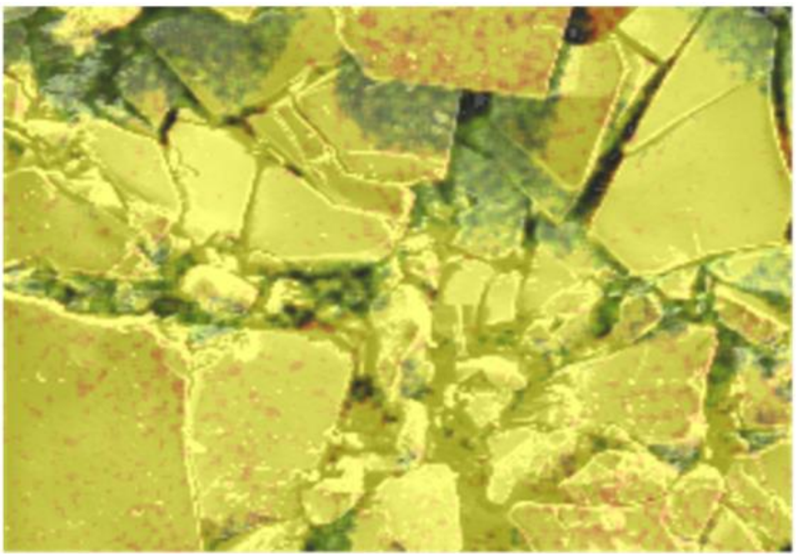


**Fig. 1. [Ni(L)2(CH3COO)2] of microstructure**
**Рис. 1. Микроструктура [Ni(L)2(CH3COO)2]**

The amounts of elements in the synthesized ligands and complexes were analyzed using the SEM-EDX method. In the obtained dataof analysis, the percentage ratio of the masses of the elements in the complex was determined. What has allowed to derive the gross formula of the complexes. Based on the given formulas, the composition of the complexes was determined.

The values of the liquidus temperature of the complex compounds and the results of elemental analysis are presented in Table 1.

**Table 1**

**3-Amino-1,2,4-triazole and the liquefaction temperature values of complex compounds based on it and the results of elemental analysis**

**Таблица 1. Элементный анализ и температура плавление комплексных соединений на основе 3-амино-1,2,4-триазола**

| № | Composition | Color | % | $T_s$, $^0C$ | Brutto formula | Found, % / Calculated, % | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | C | H | N | M |
| 1 | L | White | 80 | 157-159 | $C_2N_4H_4$ | 28,6/29,1 | 4,76/4,26 | 33,3/32,9 | - |
| 2 | $[Co(L)_2(CH_3COO)_2]$ | Red | 70 | 180-181 | $CoC_8N_8H_{14}O_2$ | 30,7/31,6 | 4,47/4,55 | 35,8/36,3 | 18,8/19,5 |
| 3 | $[Ni(L)_2(CH_3COO)_2]$ | Purple | 74 | 186-187 | $NiC_8N_8H_{14}O_2$ | 30,8/31,5 | 4,48/4,53 | 35,9/36,6 | 18,7/19,4 |
| 4 | $[Cu(L)_2(CH_3COO)_2]$ | Green | 75 | 198-199 | $CuC_8N_8H_{14}O_2$ | 30,2/30,9 | 4,40/4,47 | 35,2/36,1 | 20,1/20,9 |
| 5 | $[Zn(L)_2(CH_3COO)_2]$ | White | 72 | 179-180 | $ZnC_8N_8H_{14}O_2$ | 30,1/30,7 | 4,39/4,44 | 35,1/35,9 | 20,4/20,7 |

To determine the coordination centers in the synthesized complex compounds of Co(II), Ni(II), Cu(II) and Zn(II) acetates with 3-amino-1,2,4-triazole (L), their spectra were studied using the IR spectra method. The results of IR spectroscopy of metal complexes based on L are presented in table 2 and in fig 2.

IR spectroscopy is a technique that provides important information at investigation of the structure of amino compounds. Absorption frequencies of amines appear in the areas of 3500-3300, 1650-1500 and 1360-1000 cm-1, these areas are absorption frequencies related to different vibrations of the amino group. The value of frequencies depends on the type of amino group, with which groups is connected and whether or not there is a hydrogen bond in the molecule. For aromatic compounds, the region of deformation vibrations (900-650 cm-1) of the C-H group in the ring is the most important. The presence of an aromatic ring in the molecule can also be proved by the presence of C-H group valence vibrational frequencies in the region 3000 cm-1. An in-depth study of these areas can also determine the number of substituents in the ring and how they are positioned relative to each other on the ring [18].

The valence vibrations of the C-H group located in the 3-amino-1,2,4-triazole ring produced were observed in the 3069 cm-1 area, and the deformation vibration in the 822 cm-1 area. The valence vibrations of the NH2 bond in the ligand showed asymmetric absorption spectrum in the 3416 cm-1 area and symmetric absorption spectr - in the 3333 cm-1 area. The absorption region belonging to the C=N group showed a strong intense vibrational frequency at 1528 cm-1.

**Table 2**

**Main frequencies in the IR -spectrum of ligand L and its complexes, cm-1**

**Таблица 2**

**Основные частоты в ИК спектрах лигандаL и его комплексов, см-1**

| Compound | ν C-H | νC-N | νC=N | ν $NH_2$ | δ $NH_2$ | M→O | M→N |
|---|---|---|---|---|---|---|---|
| L | 3069<br>3211 | 1206 | 1528 | 3333<br>3416 | 1646 | - | - |
| $[Co(L)_2(CH_3COO)_2]$ | 3307 | 1204 | 1526 | 3456 | 1623 | 640 | 466 |
| $[Ni(L)_2(CH_3COO)_2]$ | 3268 | 1213 | 1533 | 3460 | 1683 | 652 | 410 |
| $[Cu(L)_2(CH_3COO)_2]$ | 3330 | 1219 | 1542 | 3440 | 1636 | 691 | 398 |
| $[Zn(L)_2(CH_3COO)_2]$ | 3090<br>3213 | 1246 | 1547 | 3417 | 1643 | 619 | 426 |

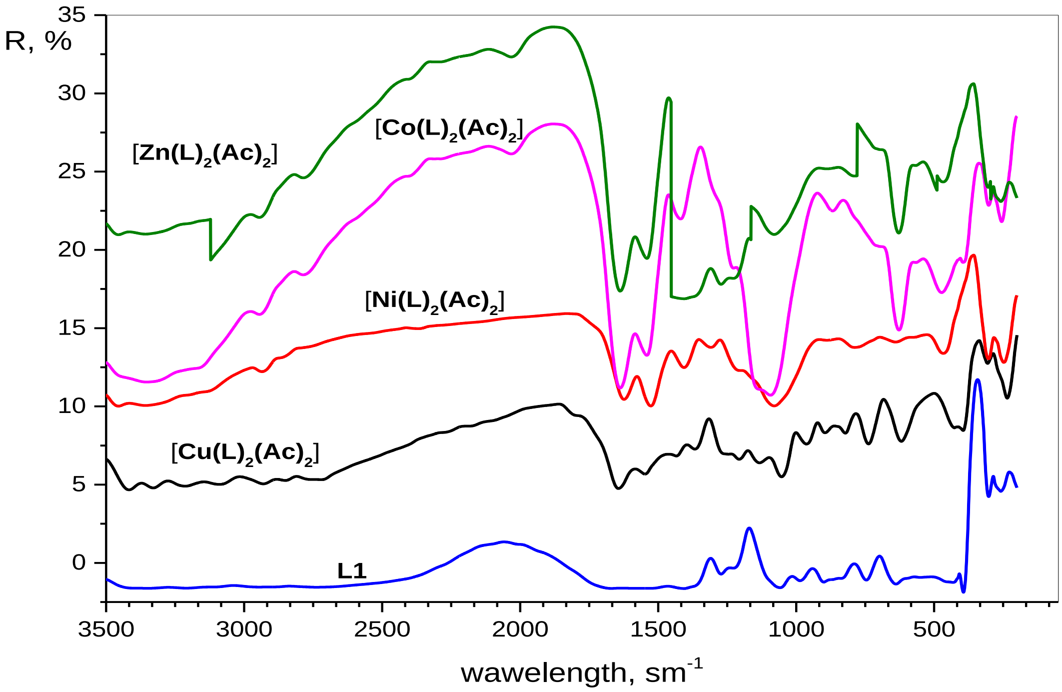
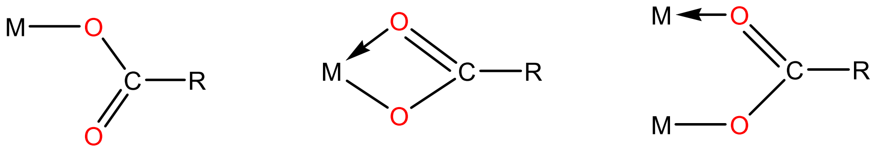



**Fig. 2.IR spectrums of 3-amino-1,2,4-triazole and its complex compounds based on Co(II), Ni(II), Cu(II), Zn(II) acetates**

**Рис. 2.ИК спектры 3-амино-1,3,4-триазола и его комплексных соединений на основе ацетатов Co(II), Ni(II), Cu(II), Zn(II)**

It is known from the literature that in the complexes of metal acetates, the carboxyl ion can coordinate with the metal atom in the following forms [19]:




I II III

- inmonodentate complexes (structure I) (Δ= [νas(CO2-) – νs(CO2-)] = 220-230 sm-1)theΔ difference in value will be somewhat higher;
- forbidentatechelate complexes (II structure) (Δ=40-80 cm-1) the Δ difference is significantly smaller;
- in bridged complexes (III structure) (Δ=140-180 cm-1), the Δ difference is higher compared to bidentate chelate complexes.

The complex obtained on the basis of L with manganese acetate was studied in the IR spectrum. There are weak absorption maxima of valence vibrations of sp2 hybridized C-H bonds in the complex compound in the range 2945 cm-1, deformation vibrations in the range 837 cm-1. We can see that the valence vibrations of the NH2 bond relative to the ligand shifted to asymmetric absorption regions at 3393 cm-1 and symmetric absorption regions at 3349 cm-1. Weak non-intense absorption lines belonging to the C=N group at 1523 cm-1have indicated that the ligand molecule is monodentately coordinated with the central atom through the first nitrogen atom in the ring of reagent. In addition, in the IR spectrum of the [ZnL2(CH3COO)2] complex, a new absorption line appeared at 1366 cm–1, which, according to [19], is the symmetric COO group, and the COO group at 1132 cm–1 corresponds to an asymmetric valence vibrations of this group. The difference between the asymmetric and symmetric valence vibrations of the carboxyl group is equal to Δ = 234 cm-1 and corresponds to the structure I, and the metal atom combines with the carboxyl ion in a monodentate state. In the 398-466 cm–1 range, we can observe the absorption maxima caused by the valence vibrations of broad linear Mn-N bonds was observed.

Raman spectroscopy is important in the spectral analysis of triazole derivatives due to its acquisition of signals of oscillations in a wide absorption range and separation of vibration frequencies more accurately than in IR spectroscopy. According to the literature, the amino group appears in range 3300-3500 cm-1. The valence asymmetric vibrations (□ass) of the NH2 group appeared in the region 3265 cm-1 and the valence symmetric vibrations (□ss) appeared in the regionof 3217 cm-1. The appearance of deformation vibrations of the NH2 group in the 1150-900 cm-1 range is mentioned has been literature [20].

The valence vibrations of the NH2 group of 3-amino-1,2,4-triazole showed were observed in the region of 3189 cm-1. Deformation vibrations of the NH2 group were observed in the regions 1073, 1045, 978, 964 cm-1. Another characteristic vibration of the molecule is the vibrations of the C-N bond in the triazole ring. Which are corresponding in many areas in the Raman spectroscopic analysis. For example, the □C-N vibration were formed in the regions 1588, 1530, 1439, 1435 cm-1. In addition, we can find the absorption belonging to the □C=N group in the region of 1672 cm-1. In

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Raman spectroscopy the absorptions related to the M-N bond in the region of 450-150 cm-1 was observed (Fig. 3).

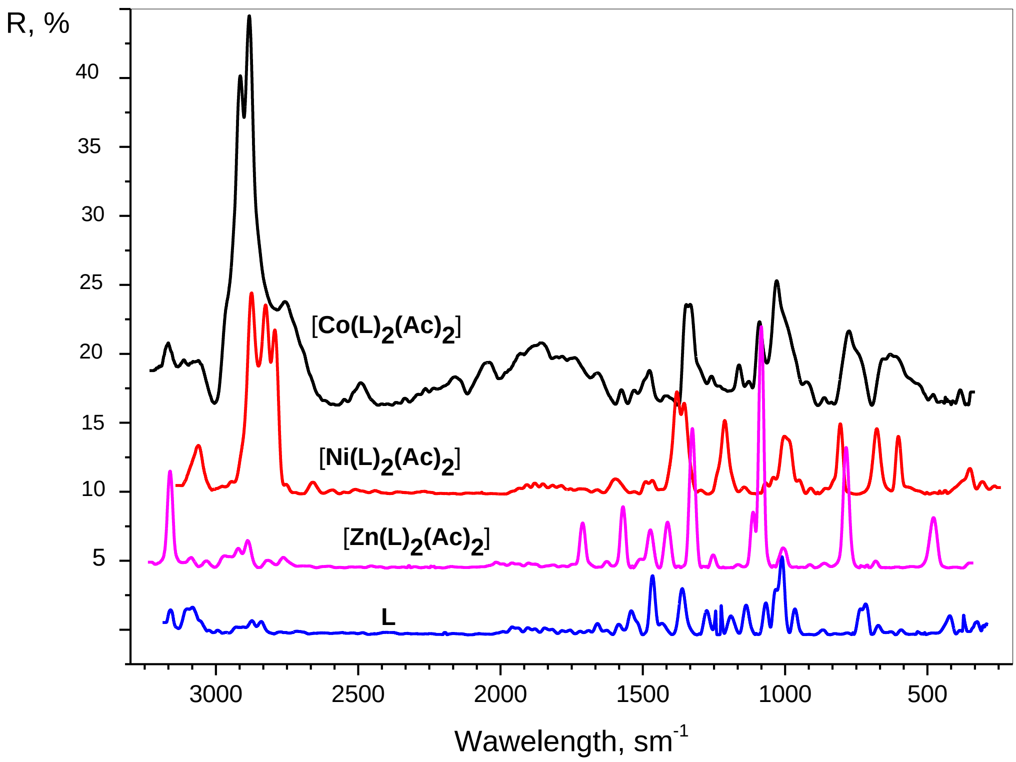


**Fig. 3. Raman spectrums of 3-amino-1,2,4-triazole and its complex compounds based on Co(II), Ni(II), Zn(II) acetates**

**Рис. 3. Раман спектры 3-амино-1,3,4-триазола и его комплексных соединений на основе ацетатов Co(II), Ni(II), Zn(II)**

In the Raman spectrum of the complex compound with nickel acetate the valence vibrations of the NH2 group were observed in the region of 3239 cm-1. Deformation vibrations of the NH2 group were formed in the region of 1073, 978, 964 cm-1. Another characteristic vibrations of the molecule are the vibrations of the C-N bond in the triazole ring. Which this observed in many areas in the Raman spectrum. For example, the C-N vibrationswere observed in the regions of 1589, 1469, 1436 cm-1. In addition, the absorption belonging to the C=N group in the region of 1626 cm-1. Was observed in Raman spectrum, the absorptions related to the M-N bond in the region of 314, 438 cm-1.

The investigation of the composition and structure of the synthesized coordination compounds was completed by obtaining their thermogravigrams. In the results of thermal analysis, the nature of thermal effects, the observation of thermal decomposition of compounds, the temperature range of effects and its nature, and the percentage mass loss in the same range of effects are presented. Were determined as a result of the thermal analysis, the decomposition and liquefaction of the complexes, the coordination of the ligand and the final products of the thermolysis process of the complexes were determined [21-22].

First, the thermal analysis of 3-amino 1,2,4-triazole was performed in range 20 - 600°C. General decomposition was observed in the range of 150-300°C. Initially, an endothermic effect was observed in the range of 159.22oC, which corresponds to the liquefaction temperature of the ligand. The next endothermic effect was observed at 279.91oC. No changes after 300oC were observed.

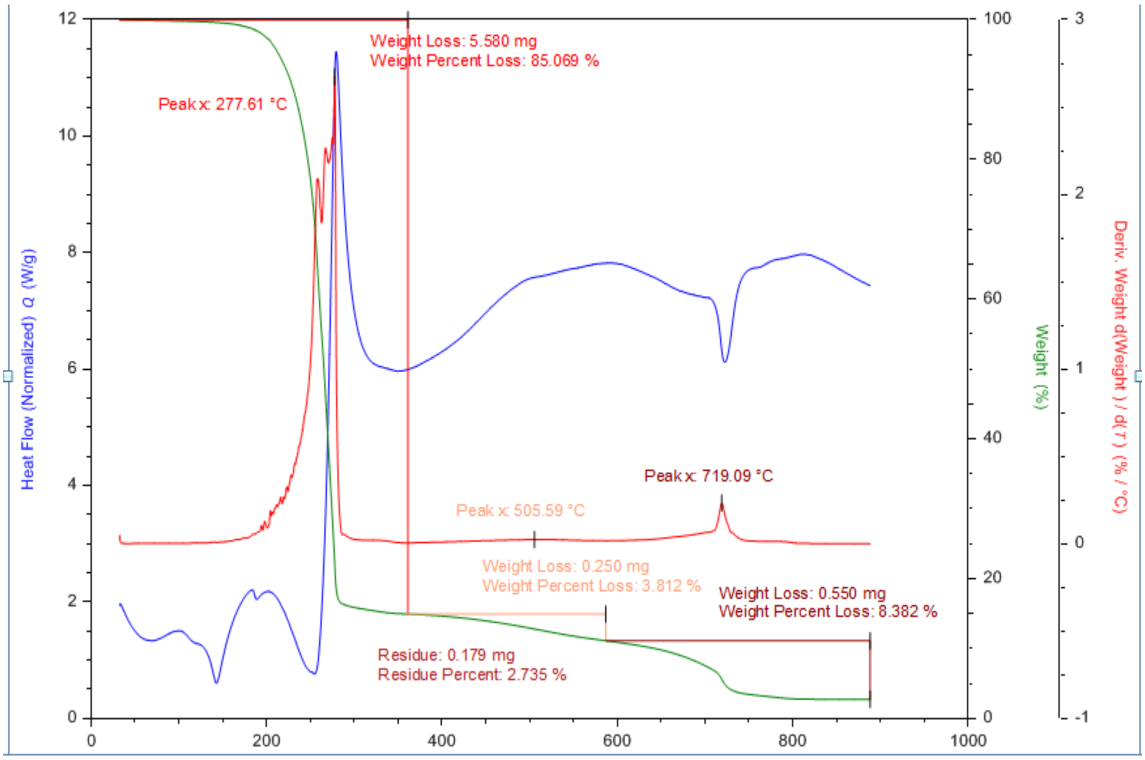


**Fig. 4.Derivatogramm of [Co(L)2(CH3COO)2]**

**Рис. 4.Дериватограмма [Co(L)2(CH3COO)2]**

[Co(L)2(CH3COO)2] in the heating curve of the complex compound, several exoeffects were observed at temperatures of 277, 505, 719oC (Fig. 4). Intensive mass loss in the range of 365-580oC was 5,580 mg, i.e. 85,069%, and an exothermic effect was observed. The last effects on the thermogravigrams of the complexes synthesized at 580-875oC are related to the formation of metal oxides. The amount of decomposition in this interval is 8.382% of the decomposition, i.e. 0.550 mg. No change is observed after 875 oC. As a result of thermolysis, metal oxide remains.

CONCLUSION

A method for the synthesis of complexes of 3-amino-1,2,4-triazole with Co(II), Ni(II), Cu(II) and Zn(II) acetates has been developed and new complex compounds have been synthesized on its basis. Based on the results of physicochemical studies, it was established that in the synthesized complex compounds the metal ion is coordinated with the ligand molecule through the first nitrogen atom in the triazole ring. Based on the results of elemental, thermal, scanning electron microscopy, IR and Raman spectroscopy, it was established that the composition of the synthesized complex compounds is in a metal-ligand ratio of 1:2.

**CONFLICT OF INTEREST**

The authors declare the absence a conflict of interest warranting disclosure in this article.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

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