SYNTHESIS AND CHARACTERIZATION OF COORDINATION COMPOUNDS OF 3d-METAL MALEATES WITH THIOSEMICARBAZIDE

Complexes of copper(II), nickel(II), cobalt(III), and zinc(II) maleates with thiosemicarbazide were synthesized. The resulting compounds were characterized by the elemental analysis data, infrared spectroscopy, diffuse reflectance spectroscopy, and thermogravimetry.

Keywords: coordination compounds, 3d-metals, thiosemicarbazide

Thiosemicarbazide is an interesting ambidentate ligand able to form five-membered chelate rings or monodentately coordinate by sulfur. It is interesting to trace the mutual effect and competition for the inner-sphere sites in coordination compounds with different inorganic and organic anions. We have previously obtained thiosemicarbazide (HL) complexes with inorganic salts [1], carboxylates [2-4], naphthalenesulfonates [5]. Depending on the central atom and salt anion, compounds with different metal:HL ratios are realized, and the ligand can act in a molecular or deprotonated form.

Thiosemicarbazide coordination compounds where anions of unsaturated carboxylic acids, in particular maleic (H2Mal), act as counterions, have been studied relatively little. The structure was described only for the complex [Ni(HL)2(H2O)2](HMal)2H2O [6].

The aim of this work was studying the products of reaction of copper(II), nickel(II), cobalt(II), and zinc(II) maleates with thiosemicarbazide.

EXPERIMENTAL SECTION

Copper(II), nickel(II), cobalt(II), and zinc(II) nitrates, maleic acid and thiosemicarbazide were of analytical grade.

The metal content in the isolated compounds was determined by atomic absorption spectrometry on a Shimadzu 7000AA instrument, the nitrogen content, according to the Dumas method [7], the sulfur content, by the Schoeniger method [7].

The IR spectra were taken on a Perkin-Elmer SPECTRUM BX II FT-IR SYSTEM instrument, samples were prepared in the form of tablets with KBr. The diffuse reflection spectra were recorded on a Lambda-9 (Perkin-Elmer) spectrophotometer with MgO (100βMgO) as the standard. Thermogravigrams were taken in air on a Paulic–Paulic–Erdei derivatograph at the heating rate of 10 deg/min.

The synthesis was carried out according to the methods developed earlier [2-4].

**Synthesis of** [Cu(HL)₂][HMal]. 0.01 mol of Cu(NO₃)₂·3H₂O was dissolved in water, to the resulting solution was added 8 g of NaOH. The precipitated hydroxide was filtered through a paper filter on a Buchner funnel and washed with water, transferred...
to a beaker with a portion of 2.38 g of maleic acid. The mixture of hydroxide with acid was thoroughly mixed adding drops of water. When the precipitate was completely dissolved, the solution was added in small portions to a solution of 1.82 g (0.02 mol) of thiosemicarbazide in 100 ml of water. The precipitate formed was separated, washed with water and air dried to constant weight.

**Synthesis of \([\text{Ni}(HL)_2(H_2O)_2](\text{HMal})_2\cdot2\text{H}_2\text{O}\).** 0.01 mol of \(\text{Ni(NO}_3\)\)_2·6\(\text{H}_2\text{O}\) was dissolved in water, to the resulting solution was added 8 g of \(\text{NaOH}\). The precipitated hydroxide was filtered through a paper filter on a Buchner funnel and washed with water, transferred to a beaker with a portion of 2.38 g of maleic acid. The mixture of hydroxide with acid was thoroughly mixed adding drops of water. When the precipitate was completely dissolved, the solution was added in small portions to a solution of 1.82 g (0.02 mol) of thiosemicarbazide in 100 ml of water. The precipitate formed was separated, washed with water and air dried to constant weight.

**Synthesis of \([\text{Ni}(HL)_2](\text{HMal})_2\).** 0.005 mol of \(\text{Ni(NO}_3\)\)_2·6\(\text{H}_2\text{O}\) was dissolved in water, to the resulting solution was added 4 g of \(\text{NaOH}\). The precipitated hydroxide was filtered through a paper filter on a Buchner funnel and washed with water, transferred to a beaker with a portion of 1.18 g of maleic acid. The mixture of hydroxide with acid was thoroughly mixed adding drops of water. When the precipitate was completely dissolved, the solution was added in small portions to a solution of 1.82 g (0.02 mol) of thiosemicarbazide in 100 ml of water. The precipitate formed was separated, washed with water and air dried to constant weight.

**Synthesis of \([\text{Co}(HL)_2]_2[\text{HMal}]_2\).** 0.01 mol of \(\text{Co(NO}_3\)\)_2·6\(\text{H}_2\text{O}\) was dissolved in water, to the resulting solution was added 8 g of \(\text{NaOH}\). The precipitated hydroxide was filtered through a paper filter on a Buchner funnel and washed with water, transferred to a beaker with a portion of 2.38 g of maleic acid. The mixture of hydroxide with acid was thoroughly mixed adding drops of water. When the precipitate was completely dissolved, the solution was added in small portions to a solution of 2.73 g (0.03 mol) of thiosemicarbazide in 150 ml of water. The mixture was allowed to stand until complete homogeneity of the precipitate, which was separated, washed with water and air dried to constant weight.

**Synthesis of \([\text{Zn}(HL)_2][\text{HMal}]_2\).** 0.005 mol of \(\text{Zn(NO}_3\)\)_2·6\(\text{H}_2\text{O}\) was dissolved in water, to the resulting solution was added 4 g of \(\text{NaOH}\). The precipitated hydroxide was filtered through a paper filter on a Buchner funnel and washed with water, transferred to a beaker with a portion of 1.18 g of maleic acid. The mixture of hydroxide with acid was thoroughly mixed adding drops of water. When the precipitate was completely dissolved, the solution was added in small portions to a solution of 1.82 g (0.02 mol) of thiosemicarbazide in 100 ml of water. The precipitate formed was separated, washed with water and air dried to constant weight.

**RESULTS AND DISCUSSION**

As can be seen from the results of chemical analysis (Table 1), for copper (II) is realized a complex with a ratio of metal: thiosemicarbazide 1:2, for zinc – 1:4, for cobalt (III) – 1:3, with cobalt (II) is oxidized by air oxygen to cobalt (III). For nickel (II), depending on the ratio of reagents, two compounds are formed – 1:2, which was described by the authors [6] and not described in the literature a compound 1:4. The ratio of metal: maleate in all complexes corresponds to a single-charged anion.
Complexes of 3d-metal maleates with thiosemicarbazide

Table 1

| № | Compound | M,% | N,% | S,% | Color |
|---|----------|-----|-----|-----|-------|
| I | [Cu(HL)L](HMal) | 18.0 | 17.8 | 23.2 | Brown |
| II | [Ni(HL)2(H2O)2](HMal)2·2H2O | 10.5 | 10.9 | 15.8 | Blue |
| III | [Ni(HL)4](HMal)2 | 9.2 | 9.0 | 25.4 | Green |
| IV | [Co(HL)2L](HMal)2 | 10.2 | 10.5 | 22.9 | Dark-pink |
| V | [Zn(HL)4](HMal)2 | 10.3 | 9.9 | 25.1 | White |

Analysis of the IR spectra of thiosemicarbazide, 3d-metal maleates, and complexes (Table 2) shows that for complexes with a composition of 1:4 they are practically identical regardless of the central atom (green Ni(HL)4(HMal)2 and Zn(HL)4(HMal)2).

At the same time, some common features are observed in the spectra of cobalt (III) and copper (II) complexes.

The thioamide I band behaves similarly for each group of complexes with similar stoichiometry. For complexes with a composition of 1:4, the band frequency increases by about 40 cm⁻¹. For the rest of the complexes, it splits into a doublet, while for the complexes of cobalt (III) and copper (II) the difference in the frequencies of the bands of the doublet is much larger (68 and 82 cm⁻¹, respectively) than for the nickel complex with the composition Ni(HL)2(HMal)2(H2O)2 (25 cm⁻¹). The thioamide II band shifts towards higher frequencies and splits into a doublet for all complexes except the last one. Wherein the frequency differences of the doublet bands for the 1:4 complexes slightly exceed those for the cobalt (III) and copper (II) complexes. In all cases, the thioamide III band does not undergo noticeable changes, while the thioamide IV band decreases.

For complexes of cobalt (III) and copper (II), very intense absorption bands appear in the IR spectra in the region of about 2000-2100 cm⁻¹. For copper (II), the frequencies of the bands of the doublet (2173, 2102 cm⁻¹) for the complex of maleate with thiosemicarbazide [Cu(HL)L](HMal) obtained in this work practically coincide with the bands in the spectra of previously obtained thiosemicarbazide complexes with similar stoichiometry: glycinate with the composition Cu(L)(Gly) (2174 and 2100 cm⁻¹) and glycyl-glycinate with the composition Cu(HL)L(HGG) (2174 and 2082 cm⁻¹) [2]. Previously, the interaction of a number of aliphatic copper (II) carboxylates Cu(CnH2n+1COO)2 with thiosemicarbazide was studied and it was found that at n≥4 the carboxylate anion is not included in the reaction products, while thiosemicarbazide appears in a deprotonated form and forms the compound CuL2 [1]. In the spectrum of this compound, absorption bands in the region of about 2100 cm⁻¹ were also found. The region 2200-1900 cm⁻¹ is characteristic of stretching vibrations of cumulated double bonds [8], in particular, for thiocyanate ions. The NCS group, similar to the thiocyanate group, in our case can arise only when the metal is bonded to the nitrogen atom adjacent to the carbon atom. Consequently, for deprotonated thiosemicarbazide, it is necessary to sug-
Table 2

The IR spectroscopy data (cm⁻¹) of complexes, maleates and ligand HL.

| Compound                  | υ(NH) + υ(OH)                  | Thioamide bands | υ(NCS) | υₗ(COO⁻) | υᵣ(COO⁻) | Δυ(COO⁻) | ΔΔυ(COO⁻) |
|---------------------------|-------------------------------|-----------------|--------|----------|----------|----------|-----------|
|                           |                               | I   | II  | III | IV |           |           |           |           |
| HL                        | 3370, 3260, 3170              | 1530| 1315| 1000 | 800 | 1578     | 1385     | 193       |
| Cu(II) Mal. H₂O          |                               |     |     |     |     |           |           |           |
| [Cu(II)HL] (HMal)        | 3164                         | 1585| 1385| 1360| 1000| 763      | 2173, 2102| 1585     | 1360     | 225       | 32        |
| Ni(II) Mal. 3H₂O        |                               |     |     |     |     | 1551     | 1418     | 133       |
| [Ni(II)HL₂(H₂O)₂ (HMal)₂]₂H₂O | 3564, 3390, 3330, 3202, 3154, 3099, 2990, 2881 | 1559| 1534| 1382| 1009| 736      | -        | 1534     | 1427     | 107       | -26       |
| [Ni(II)HL₂] (HMal)₂      | 3414, 3261, 3187, 2890        | 1571| 1385| 1352| 1003| 731      | -        | 1571     | 1385     | 186       | 53        |
| Co(II) Mal. 3H₂O        |                               |     |     |     |     | 1579     | 1440     | 139       |
| [Co(II)HL₂] (HMal)₂      | 3326, 3206, 3162              | 1589| 1521| 1395| 1365| 1003| 736  | 2055     | 1521     | 1395     | 126       | -13       |
| Zn(II) Mal. 2H₂O        |                               |     |     |     |     | 1575     | 1432     | 143       |
| [Zn(II)HL₄] (HMal)₂      | 3408, 3263, 3190, 2885        | 1572| 1385| 1352| 1003| 731      | -        | 1572     | 1385     | 187       | 44        |
gest the formation of a four-membered ring, where the metal is covalently bonded to nitrogen and coordinated to sulfur. Thus, for the obtained compounds of cobalt (III) and copper (II), taking into account the analysis data, it should be supposed the complex contains two forms of thiosemicarbazide: molecular and deprotonated. For the complex [Ni(HL)2(H2O)2](HMal)2, the formation of a chelate also occurs, but the cycle is five-membered with the participation of sulfur and nitrogen of the amino group, which coincides with the X-ray diffraction data of [6]. In coordination compounds with a composition of 1: 4, thiosemicarbazide is obviously monodentate with coordination only through sulfur. It is necessary to pay attention to the presence of a much larger number of absorption bands in the region of about 3000 cm−1 in the spectrum of the [Ni(HL)2(H2O)2](HMal)2H2O complex in comparison with the spectra of all other complexes. This can be explained by the presence of bound water in the composition of this compound; separate identification of the N-H and O-H bands is not possible due to the presence of numerous hydrogen bonds.

The values of ΔΔν(COO−) for all synthesized complexes are small, and for [Ni(HL)2(H2O)2](HMal)2H2O and [Co(HL)2L](HMal)2, are negative at all, that allows to suppose in all cases the outer-sphere character of maleate ions.

In the IR spectra of all synthesized complexes of maleates with thiosemicarbazide, except for the cobalt (III) complex, there is a shoulder at about 1700 cm−1. There are no such bands in the spectra of nickel (II) maleate complexes with phenylacethydrazide and cobalt (II) maleate with benzhydrazide, for which the presence of the Mal2− anion in the compounds was established by X-ray diffraction analysis [9]. The presence of bands around 1700 cm−1 is related with the presence in the composition of thiosemicarbazide complexes of one COOH group from monodeprotonated maleic acid, for which absorption bands of COOH appear at 1707 cm−1 [10].

Diffuse reflection spectra (Table 3) indicate the octahedral structure of the nickel complex of composition 1: 2 and the cobalt (III) complex, the tetrahedral structure of the nickel (II) complex of composition 1: 4 and the pseudotetrahedral structure of the copper (II) complex.

| Compound                          | ν, cm−1 | Assignment          |
|-----------------------------------|---------|---------------------|
| [Cu(HL)L](HMal)                   | 18230   |                     |
| [Ni(HL)2(H2O)2](HMal)2H2O        | 17210   | 3T1 → 3T1(P)        |
|                                   | 13110   | 3T1 → 1E            |
|                                   | 4450    | 3T1 → 3A2           |
| [Ni(HL)2](HMal)2                 | 17180   | 1A2g → 3T1g         |
| [Co(HL)L](HMal)2                 | 18150   | 1A1g → 3T1           |

Table 3

The diffusion reflectance spectroscopy data of complexes
Thermal analysis data (Table 4) show that the highest thermal stability has the complex \([\text{Ni}(HL)_2(H_2O)_2](\text{HMal})_2\cdot2\text{H}_2\text{O}\), the other complexes decompose at fairly close temperatures. For all synthesized compounds on thermogravimetric analysis, the first few effects are endothermic, exo-effects are observed above 500 °C, and about 700 °C there are effects (exothermic for Co(III) and endothermic for other complexes), accompanied by a small increase in mass. Similar increases were also in thiosemicarbazide complexes with malonates, glutarates [3], p-hydroxybenzoates and p-aminobenzoates [4].

| Compound | Endo-effects | Exo-effects | Total mass loss,% |
|----------|-------------|-------------|------------------|
|          | t, °C       | Δm,%        | t, °C            | Δm,%          |                  |
| [Cu(HL)L](HMal) | 90-110(100) 4.4 | 500-700(620) 23.0 | 67.5 |
|          | 170-190(180) 7.8 |                   |                  |
|          | 260-330(270) 14.2 |                   |                  |
|          | 700-820(750) +2.5 |                   |                  |
| [Ni(HL)_2(H_2O)_2](HMal)_2\cdot2\text{H}_2\text{O} | 170-220 (180) 6.4 | 570-660 (620) 23.7 | 80.0 |
|          | 220-260 (230) 20.7 |                   |                  |
|          | 660-800(690) +1.0 |                   |                  |
| [Ni(HL)_4](HMal)_2 | 100-130 (110) 14.3 | 500-630 (560) 24.0 | 85.0 |
|          | 200-240(220) 19.7 |                   |                  |
|          | 630-800(750) +3.0 |                   |                  |
| [Co(HL)_2L](HMal)_2 | 100-120 (110) 7.2 | 550-670(580) 23.6 | 82.5 |
|          | 180-280(240) 28.7 | 670-830 (690) +2.6 |                  |
| [Zn(HL)_4](HMal)_2 | 90-110(100) 10.7 | 600-730(700) 17.8 | 81.3 |
|          | 150-200(180) 6.1 |                   |                  |
|          | 730-880(770) +3.1 |                   |                  |

The studies carried out suggest the following structures of the synthesized compounds:

\[
\begin{align*}
\text{Cu} & \quad \text{S} \quad \text{N} \quad \text{NH} \quad \text{2} \\
\text{H}_2\text{N} & \quad \text{C} \quad \text{S} \quad \text{NH} \quad \text{2} \\
\text{NH}_2 & \quad \text{C} \quad \text{C} \quad \text{C} \quad \text{NH}_2
\end{align*}
\]

\[
\begin{align*}
\text{HO} & \quad \text{O} \\
\text{HO} & \quad \text{O} \\
\text{HO} & \quad \text{O}
\end{align*}
\]
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Thus, coordination compounds of copper(II), nickel(II), cobalt(III), and zinc(II) maleates with thiosemicarbazide were synthesized. Their structure was studied by the IR and diffuse reflection spectroscopy methods. In coordination compounds with a composition of 1:4, thiosemicarbazide is monodentate with coordination only through sulfur. There is five-membered cycle with the participation of sulfur and nitrogen of the amino group for the complex \([\text{Ni(HL)}_2(\text{H}_2\text{O})_2](\text{HMal})_2\cdot 2\text{H}_2\text{O}\). For the obtained compounds of cobalt (III) and copper (II), it should be supposed the complex contains two forms of thiosemicarbazide: molecular and deprotonated.
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Complexes of 3d-metal maleates with thiosemicarbazide

 Kovaleвним зв’язком з азотом і координаційним зв’язком із сіркою. Тіосемікарбазид у молекулярній формі в комплексах складу 1:2 і 1:3 координований з утворенням п’ятичлененого циклу за участю сірки і азоту аміногрупи. У координаційних сполуках складу 1:4 тіосемікарбазид монодентатний з координацією лише через сірку. Малеат-іони у всіх випадках є зовнішньосферними. При цьому в ІЧ спектрах присутні смуги близько 1700 см⁻¹, що підтверджує наявність однієї протонованої карбоксильної групи і узгоджується з тим, що малеат-іон однозарядний.

Спектри дифузного відбиття вказують на октаедричну структуру [Ni(HL)₂(H₂O)₂](HMal)₂ и [Co(HL)₂L(HMal)]₂, тетраедричну структуру [Ni(HL)₄](HMal)₂ і псевдотетраедричну структуру [Cu(HL)₂L](HMal).

Дані термічного аналізу показують, що найбільшу термічну стабільність має комплекс [Ni(HL)₂(H₂O)₂](HMal)₂·2H₂O, інші комплекси розкладаються при досить близьких температурах. Перші ефекти на термогравіметрах є ендотермічними, екзоефекти спостерігаються вище 500°C, а близько 700°C присутні ефекти, що супроводжуються невеликим збільшенням маси.

Ключові слова: координаційні сполуки, 3d-метали, тіосемікарбазид

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СИНЕЗ И ХАРАКТЕРИСТИКА КООРДИНАЦИОННЫХ
СОЕДИНЕНИЙ МАЛЕАТОВ 3d-МЕТАЛОВ
С ТИОСЕМИКАРБАЗИДОМ

Синтезированы комплексы малеатов меди(II), никеля(II), кобальта(III) и цинка(II) с тиосемикарбазидом. Полученные соединения охарактеризованы методами элементного анализа, инфракрасной спектроскопии, спектроскопии диффузного отражения и термогравиметрии.

Ключевые слова: координационные соединения, 3d-металлы, тиосемикарбазид.

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