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SYNTHESIS OF COMPLEXES OF Cu(II) AND VO(IV) WITH HYDRAZIDE OF MALEIC AND HYDRAZINEDIACETIC ACID

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Abstract: The work demonstrated that hydrazide of maleic and hydrazine diacetic acid(L-2,2'-(2-(3-carboxyacryloyl)hydrazine-1,1-diyl)diacetic acid) forms mononuclear complexes [CuL(H₂O)₂] and [VOL(H₂O)₂]. The structure and physical properties of these complex compounds have been studied by IR, UV spectroscopy and thermogravimetric methods of analysis. On the basis of the data obtained, the structure of the complexes was proposed, in which the metal ion is coordinated by two oxygen atoms and one nitrogen atom of the aminodiacetic fragment. In the electronic spectra of the copper complex, there are absorption bands of the transition at ~240 nm (related to charge transfer) and one d-d transition 740 nm. In the electronic spectra of the vanadyl complex, several absorption bands are observed at 300 nm (inflection), ~625 nm (inflection), and 780 nm, which can be attributed to d-d transitions of the VO²⁺ in the low symmetry ligand environment. The five-coordinated structure of the complex is carried out by two oxygen atoms of two water molecules. The EPR spectrum of the polycrystalline [CuL(H₂O)₂] complex at room temperature indicates a low symmetry of the complex and has three g-factor values: g₁=2.30, g₂=2.22; g₃=2.11. Additionally, a hyperfine structure is observed due to ^{63,64}Cu nuclei (nuclear spin 3/2) with a splitting constant of 80 G.

Keywords: hydrazide, maleic acid, mononuclear complexes, Cu(II), VO(II) ions**DOI:** 10.32737/2221-8688-2023-3-269-278

Introduction

The previously described 3d metal maleates and their mixed-ligand complexes are of interest from both practical and theoretical points of view. These compounds can be used in the field of nonlinear optics [1-4]. Thermolysis of maleates can be used to obtain small metal or metal oxide nanoparticles stabilized in a polymer matrix. An example is the M(OOCCH=CHCOO)_{n-m}H₂O (M = Fe(III), Co(II)) complex, which upon thermolysis forms both individual particles of metal oxides and aggregates of three to ten such types, embedded in a polymer matrix [5].

Cobalt bis (hydromaleate) tetrahydrate

exhibits luminescent properties. The theoretical interest in maleate complexes is due to the fact that maleic acid can bind to a metal ion in mono- and dianion form as a mono-, bi-, tri-, or tetradentate ligand to form various structures [6-10]. The maleate ion can be monodentate, bidentate, tridentate, tetradentate, bridged, and chelated [11-14]. We have obtained for the first time a hydrazide of maleic and hydrazine diacetic acid(L-2, 2'-(2-(3-carboxyacryloyl)hydrazine-1,1-diyl)diacetic acid), by reacting which with salts of transition metals (Cu(II) and VO(II)) mononuclear complexes [CuL(H₂O)₂] and [VOL(H₂O)₂].

Experimental part

All chemicals that took part in the synthesis were used without further purification. Maleic acid was used analytically pure, from Sigma-Aldrich. IR spectra of samples in vaseline were recorded in the range of 4000–400 cm^{-1} using a NicoletIS10 spectrophotometer. Electronic absorption spectra were recorded on a UV-VISE 60S spectrophotometer in ethanol solutions in the range of 200–400 nm. Hydrazine diacetic acid was obtained according to a known method [15].

Preparation of maleic acid hydrazide with hydrazine diacetic acid (L-2,2'-(2-(3-carboxyacryloyl)hydrazine-1,1-diyl)diacetic acid). Maleic acid hydrazide was obtained by reacting maleic anhydride with N,N-hydrazine diacetic acid. To 1 mmol of N,N hydrazine diacetic acid dissolved in 8 ml of water was added 1 mmol of maleic anhydride. The mixture was stirred for 2 hours. A day later, white transparent crystals fell out of the solution. $T = 202^\circ\text{C}$. Yield 0.80 g (85%). For $\text{C}_8\text{H}_{10}\text{O}_7\text{N}_2$

calculated %: C 39.03 H 4.09 N 11.38. Found %: C 39.09 H 4.03 N 11.40

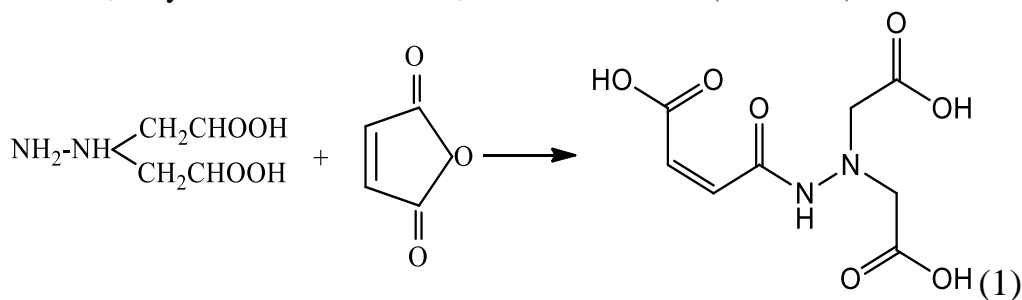
Synthesis of $[\text{CuL}(\text{H}_2\text{O})_2](\text{L}-2,2'-(2-(3\text{-carboxyacryloyl)hydrazine-1,1-diyl)diacetic acid})$. To 0.004 mol (120 mg) of maleic acid hydrazide dissolved in ethanol solution, taken in a ratio of 1:1, was added 0.005 mol (100 mg) of copper acetate $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$ dissolved in 5 ml of methyl alcohol. The solutions were mixed and stirred on a magnetic stirrer at a temperature of $30\text{--}40^\circ\text{C}$. After 2 days, blue crystals are formed, which are separated and dried in air $T_m > 250^\circ\text{C}$. The crystals are soluble in alcohol. Yield 0.70 g (75%). For $\text{Cu}(\text{C}_8\text{H}_{10}\text{O}_7\text{N}_2)(\text{H}_2\text{O})_2$ calculated %: C 27.90 H 2.90 N 8.13 Cu 18.49 Found %: C 27.88 H 2.94 N 8.18 Cu 18.45

The complex $[\text{VOL}(\text{H}_2\text{O})_2]$ was obtained by the method described above. For $\text{VO}(\text{C}_8\text{H}_{10}\text{O}_7\text{N}_2)(\text{H}_2\text{O})_2$ calculated %: C 27.66 H 2.88 N 8.06 V 14.61; found %: C 27.70 H 2.96 N 8.02 V 14.66. $T_m > 250^\circ\text{C}$. Yield 0.68 g (72%).

Discussion of the results

For the first time, by reacting maleic anhydride with N,N-hydrazine diacetic acid, a

hydrazide of maleic and hydrazine diacetic acid was obtained (Scheme 1).

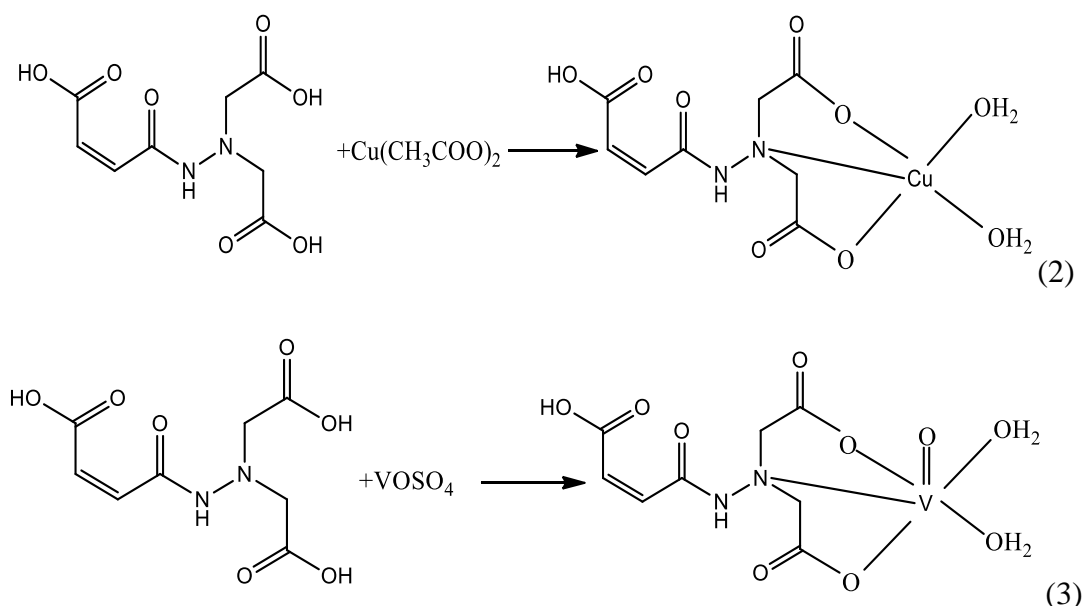


Scheme 1. L-2,2'-(2-(3-carboxyacryloyl)hydrazine-1,1-diyl)diacetic acid

The obtained ligand 2,2'-(2-(3-carboxyacryloyl)hydrazine-1,1-diyl) diacetic acid) in alcohol solutions forms stable complex compounds with Cu(II) and VO(IV) ions with the metal ion: ligand ratio 1:1, having the following compositions: $[\text{CuL}(\text{H}_2\text{O})_2]$ and

$[\text{VOL}(\text{H}_2\text{O})_2]$ (Scheme 2).

The structure and physical properties of these complex compounds have been studied by infrared spectroscopy, UV spectroscopy, EPR, and thermogravimetric analysis.



In the IR spectrum of the ligand (2,2'-(2-(3-carboxyacryloyl)hydrazine-1,1-diyl)diacetic acid), a wide absorption band related to O–H stretching vibrations is located in the region of 3390 cm^{-1} , in the copper complex is in the region of 3462 cm^{-1} , and in the vanadyl complex in the region of 3582 cm^{-1} (Fig.1). The absorption band in the region of 3116 cm^{-1} related to the N–H group of the ligand is shifted, in the copper complex, to the region of 3296 cm^{-1} (Fig.2). The carboxyl group, located in the 1746 cm^{-1} region of the ligand, in the copper

complex is shifted to a lower frequency region of 1662 cm^{-1} , and in the vanadyl complex to the 1644 cm^{-1} region. The band at 2349 cm^{-1} , which belongs to the O=C=O group of the ligand in the copper complex, is shifted to the region of 2359 cm^{-1} , and in the vanadyl complex to the region of 2341 cm^{-1} (Fig.3). The shift of the bands in the complexes suggests that the metal ion is coordinated by two oxygen atoms and one nitrogen atom of the aminodiacetic fragment [16-17].

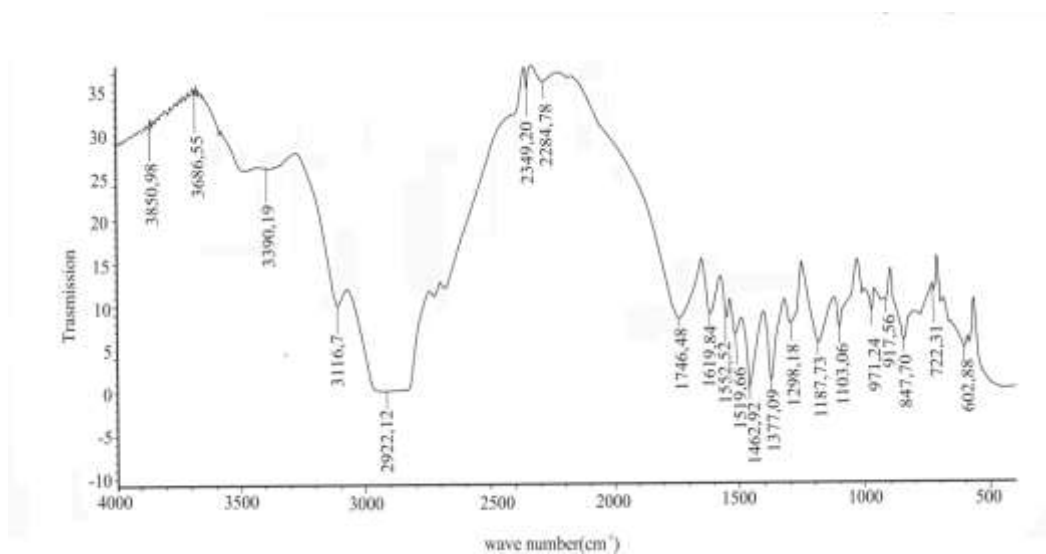


Fig.1. IR spectrum of 2,2'-(2-(3-carboxyacryloyl)hydrazine-1,1-diyl) diacetic acid in vaseline oil.

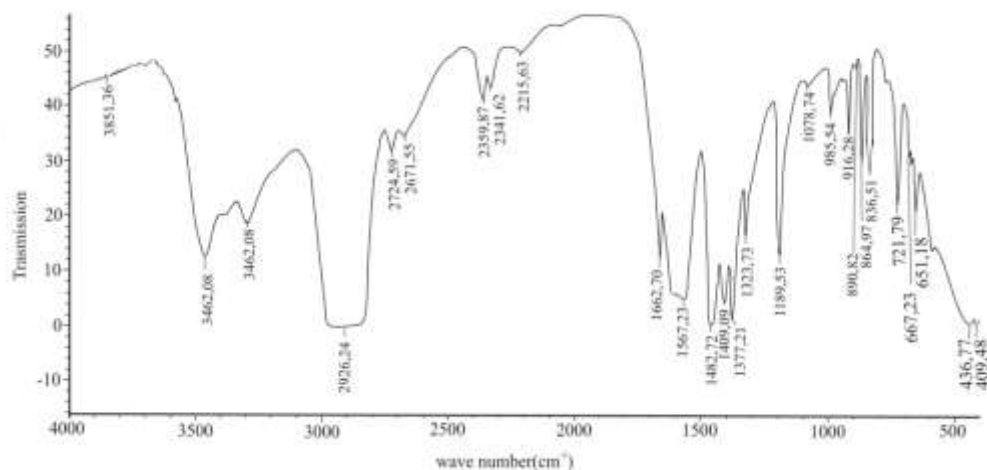


Fig. 2. IR spectrum of the $[\text{CuL}(\text{H}_2\text{O})_2]$ complex in vaseline oil

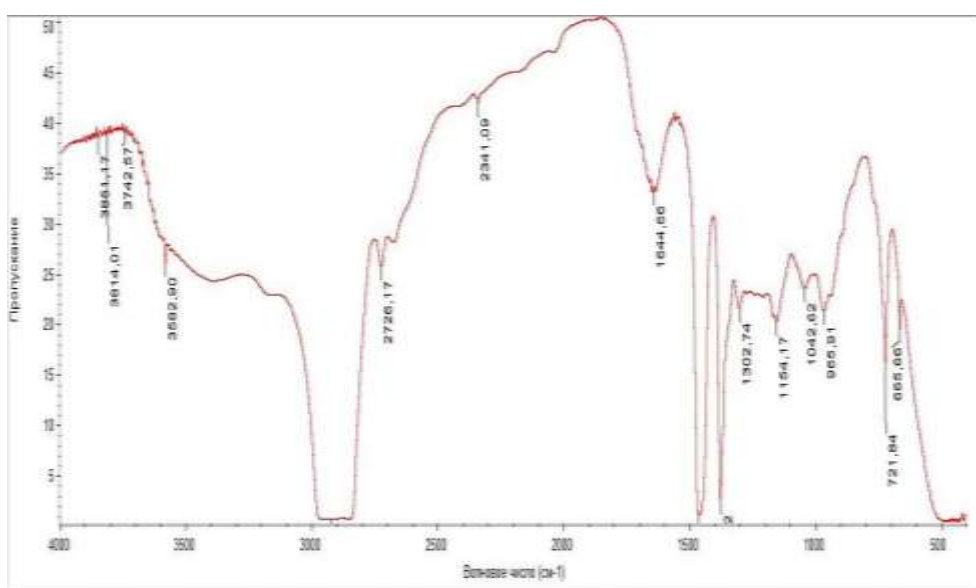


Fig. 3. IR spectrum of the $[\text{VOL}(\text{H}_2\text{O})_2]$ complex in vaseline oil

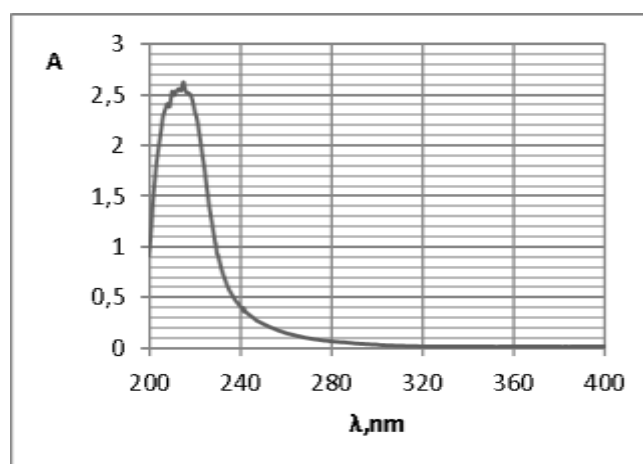


Fig. 4. Electronic spectrum of 2,2'-(2-(3-carboxyacryloyl)hydrazine-1,1-diyl) diacetic acid in ethyl alcohol

There is a band at 210 nm in the electronic spectrum of the ligand (Fig.4). The electronic spectrum of the copper complex is shown in Figure 5. In the electronic spectra of

copper complex there are absorption bands at ~240 nm (related to charge transfer) and one d-d transition 740 nm [18].

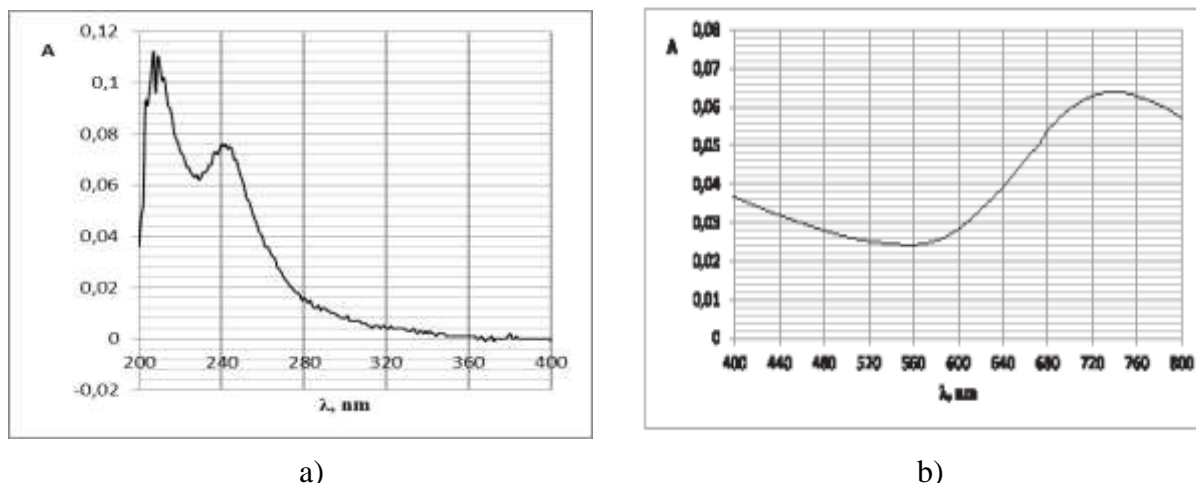


Fig. 5. Electronic spectra of $[\text{CuL}(\text{H}_2\text{O})_2]$ complex in ethyl alcohol: a) in the UV region; b) in the visible region.

In the electronic spectra of the vanadyl complex, several absorption bands are observed at 300 nm (inflection), ~625 nm (inflection),

and 780 nm, which can be attributed to d-d transitions of the VO^{2+} ion in the low symmetry ligand environment (Fig.6) [19-22].

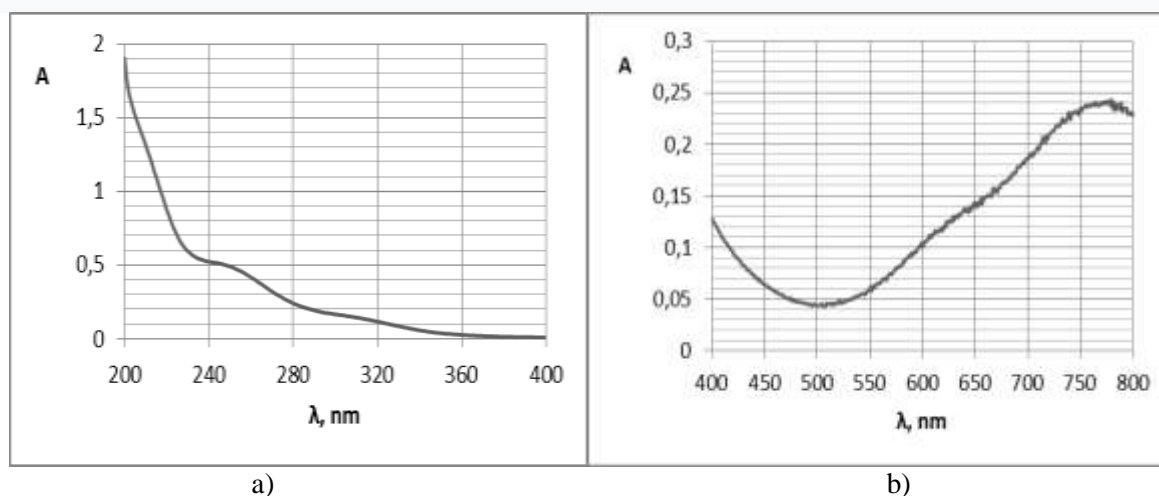


Fig. 6. Electronic spectra of the $[\text{VOL}(\text{H}_2\text{O})_2]$ complex in ethyl alcohol: a) in UV region; b) in the visible region.

The thermogravimetric analysis of the $[\text{CuL}(\text{H}_2\text{O})_2]$ complex occurred in 4 stages. In the range of 157–230°C, weight loss is observed due to the removal of water (loss of two molecules of coordinated water per formula unit is 9.15% (calculation) and 8.10% (experiment) (Fig. 7). At the next stages in the

temperature range 330–797.1°C, a weight loss of 24.59% is observed, probably due to the decomposition of carboxylate ligands (loss of carboxylate ligands per formula unit is 24.75% (calculation) 24.59%, (experiment) and the presence of a precipitate in the form of metal oxide indicated the end of the process.

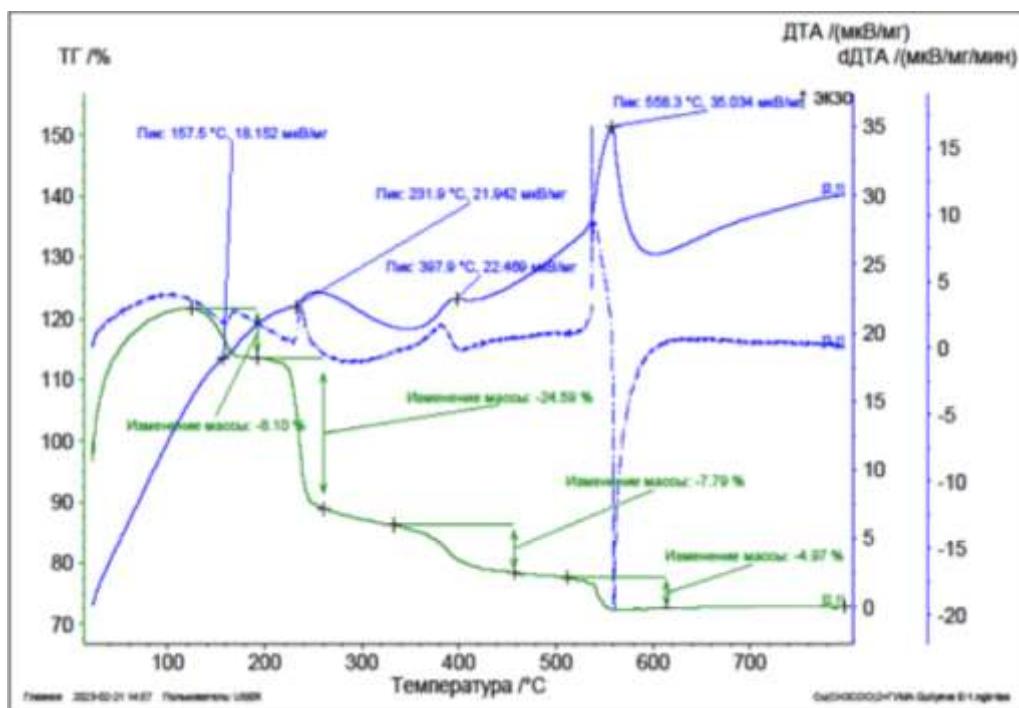


Fig. 7. Derivatogram of the [CuL(H₂O)₂] complex

In the vanadyl complex [VOL(H₂O)₂], the decomposition also occurs in four stages. At the first and second stages, in the range of 110–355°C, a mass loss is also observed due to the elimination of water molecules (loss of two coordinated water molecules per formula unit is

7.51% (calculation) and 6.13% (experiment) (Fig. 8). In the next two stages, in the temperature range of 468–618.4°C, the decomposition of carboxylate ligands also takes place, with the formation of vanadium oxide as a result.

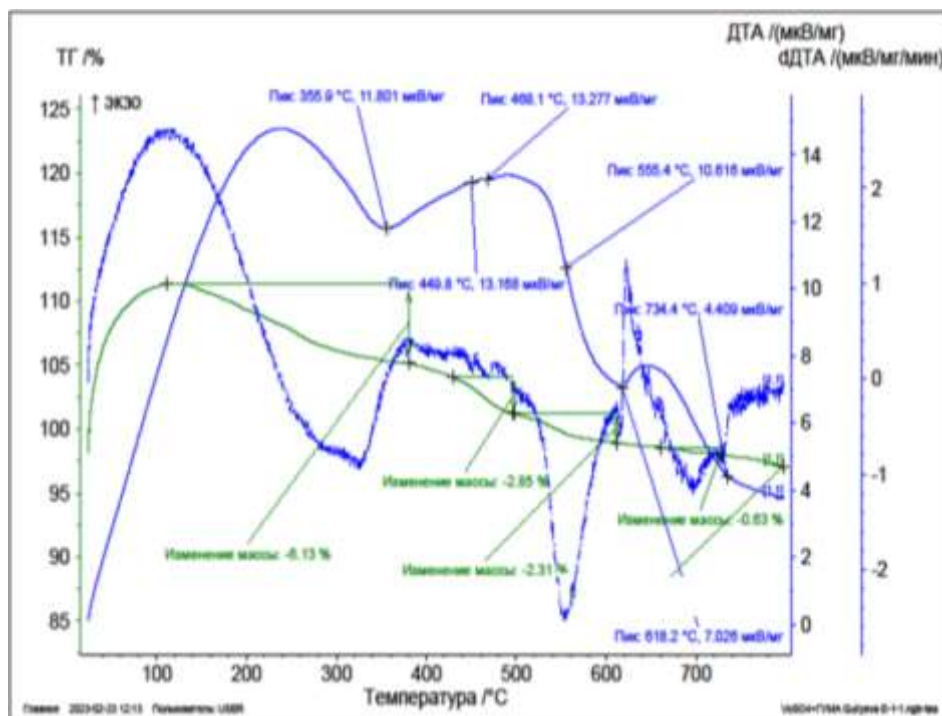


Fig. 8. Derivatogram of the [VOL(H₂O)₂] complex

The EPR spectrum of the polycrystalline complex (Fig.9) $[\text{CuL}(\text{H}_2\text{O})_2]$ at room temperature indicates a low symmetry of the complex and has three values of the g-factor:

$g_1=2.30$, $g_2=2.22$; $g_3=2.11$. Besides, a hyperfine structure is observed due to $^{63,64}\text{Cu}$ nuclei (nuclear spin 3/2) with a splitting constant of 80 G.

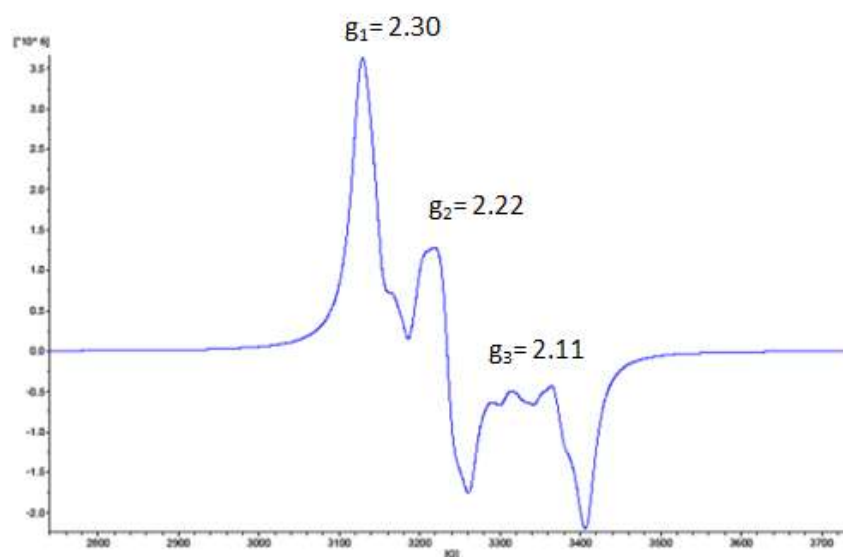


Fig. 9. EPR spectrum of the $[\text{CuL}(\text{H}_2\text{O})_2]$ complex

Conclusion

Thus, a hydrazide of maleic and hydrazine diacetic acid was obtained, which forms with Cu(II) and VO(IV) ions the mononuclear complexes $[\text{CuL}(\text{H}_2\text{O})_2]$ and $[\text{VO}(\text{H}_2\text{O})_2](\text{L}-2,2'-(2-(3\text{-carboxyacryloyl)hydrazine-1,1-diyl)diacetic acid))$. On the basis of the data obtained, the structure of the obtained complexes was proposed, in which the metal ion is coordinated by two oxygen atoms and one nitrogen atom of the aminodiacetic fragment. The five-

coordinated structure of the complex is due to the additional binding of two oxygen atoms of two water molecules. The binding spectrum of the polycrystalline $[\text{CuL}(\text{H}_2\text{O})_2]$ complex at room temperature shows a low symmetry of the complex and has three g -factor values: $g_1=2.30$, $g_2=2.22$; $g_3=2.11$. Aside from this, a hyperfine structure is observed due to $^{63,64}\text{Cu}$ nuclei (nuclear spin 3/2) with a splitting constant of 80 G.

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MALEİN HİDRAZİD VƏ HİDRAZİNDİASETAT TURŞUSU İLƏ Cu(II) VƏ VO(IV) KOMPLEKSLƏRİNİN SİNTEZİ

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Xülasə: Bu işdə göstərilmişdir ki, malein turşusunun hidrazidi və hidrazin diasetat turşusu (L-2,2'-(2-(3-karboksiakrilol)hidrazin-1,1-diil)diasetat turşusu) mononüvəli komplekslər əmələ gətirir [CuL(H₂O)₂] və [VOL(H₂O)₂]. Bu kompleks birləşmələrin strukturu və fiziki xassələri İQ, UB spektroskopiyası və termogravimetrik analiz üsulları ilə öyrənilmişdir. Alınan məlumatlar əsasında metal ionunun iki oksigen atomu və aminodiasetat fraqmentinin bir azot atomu ilə əlaqələndirildiyi əldə edilmiş komplekslərin strukturu təklif edilmişdir. Mis kompleksinin elektron spektrlərində ~240 nm-də (yük ötürülməsi ilə bağlı) keçidin udma zolaqları və bir d-d keçidi var 740 nm. Vanadil kompleksinin elektron spektrlərində bir neçə udma zolaqları: 300 nm (əyilmə), ~625 nm (əyilmə) və 780 nm-də müşahidə olunur ki, bu da VO²⁺ aşağı simmetriyalı liqand mühitində d-d keçidlərinə aid edilə bilər. Kompleksin beş koordinasiyalı quruluşu iki su molekulunun iki oksigen atomu tərəfindən həyata keçirilir. Otaq temperaturunda polikristal [CuL(H₂O)₂] kompleksinin EPR spektri kompleksin aşağı simmetriyasını göstərir və üç g-faktorlu qiymətə malikdir: g₁=2.30, g₂=2.22; g₃=2.11. Bundan əlavə, 80 G parçalanma sabiti olan ^{63,64}Cu nüvələr (nüvə spini 3/2) hesabına hiperincə struktur müşahidə olunur.

Açar sözlər: hidrazid, malein turşusu, mononüvəli komplekslər, Cu(II), VO(II) ionları

СИНТЕЗ КОМПЛЕКСОВ Cu(II) И VO(IV) С ГИДРАЗИДОМ МАЛЕИНОВОЙ И ГИДРАЗИНДИУКСУСНОЙ КИСЛОТЫ

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Аннотация: Показано, что гидразид малеиновой кислоты и гидразиндиуксусной кислоты образует с ионами Cu(II) и VO(IV) моноядерные комплексы [CuL(H₂O)₂] и [VOL(H₂O)₂] (L-2,2'-(2-(3-карбоксиакрилоил)гидразин-1,1-диил)диуксусная кислота). Строение и физические свойства этих комплексных соединений изучены ИК-, УФ-спектроскопией и термогравиметрическим методами анализа. На основе полученных данных предложено строение полученных комплексов, в котором координация иона металла осуществляется двумя атомами кислорода и одним атомом азота аминодиуксусного фрагмента. В электронных спектрах комплекса меди присутствуют полосы поглощения перехода при ~240 нм (относящиеся к переносу заряда) и один d-d переход 740 нм. В электронных спектрах ванадиевого комплекса наблюдается несколько полос поглощения при 300 нм (перегиб), ~625 нм (перегиб) и 780 нм, которые можно отнести к d-d переходам иона VO²⁺ с лигандным окружением с низкой симметрией. Пятикоординированное строение комплекса осуществляется за счет двух атомов кислорода двух молекул воды. Спектр ЭПР поликристаллического комплекса [CuL(H₂O)₂] при комнатной температуре указывает на низкую симметрию комплекса и имеет три значения g-фактора: g₁=2.30, g₂=2.22; g₃=2.11. Дополнительно наблюдается сверхтонкая структура за счет ядер ^{63,64}Cu (ядерный спин 3/2) с константой расщепления 80 Гс.

Ключевые слова: гидразид, малеиновая кислота, моноядерные комплексы, ионы Cu(II), VO(II)