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Synthesis Of A Mixed Heteroligand Complex Of Copper Salts With Acetylacetone And Acetamid

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Abstract: In recent years, scientists worldwide have been conducting research on the synthesis of heteroligand complex compounds, identifying coordination centers in heteroligand compounds, studying their geometric structure, composition, and properties, elucidating mechanisms of complex formation reactions, investigating complex compounds in solutions, and isolating them in solid form. This article discusses the results of synthesis and analysis of such complex compounds.

Keywords: Coordination number, ligand, acetylacetone, acetamide, complex compound, spectroscopy.

INTRODUCTION:

The synthesis and study of complex compounds of intermediate metals with oxygen-containing heteroligands is of particular interest. The high degree of coordination of acetylacetone is explained by the presence of two carbonyl groups in the molecule. The structure of complex compounds of intermediate metals with acetylacetone has been studied by scientists before, and their structural properties are characterized by various factors, including the nature of the metal, the presence of a substituent in the ligand, and its type. The synthesis of complex compounds with different structures and physicochemical properties is characterized by these very properties of acetylacetone [1].

Currently, complex compounds with heteroligands are widely used in many fields, including medicine, polymers, analytical chemistry, light-sensitive materials, the production of corrosion-resistant inhibitors, as plant growth accelerators, and radiochemistry. From the data presented, it is clear that the formation of a complex with a mixed ligand the study of mixed ligand complexes (MLC) is of great importance for the scientific substantiation of their composition, structure and properties [2]. The study of mixed ligand complexes (MLC) indicates the need to clarify some of the fundamental concepts of coordination chemistry, since mixed ligand complexes play an important role in oxidation-reduction processes and participate in physiological processes of various biological systems and enzymatic reactions [3]. The study of MLC is of great importance for the study of exchange reactions in complexes. The individuality of elements is clearly manifested in the formation of MLC, which opens up prospects for increasing the selectivity and sensitivity of the detection, separation and concentration of components [4].

LITERATURE ANALYSIS

The stability of ALK is usually compared with the stability of the corresponding binary complexes, since this allows us to determine the type of coordination, the coordination number (k.s.) of the metal ion, the nature of the ligands involved in the formation of ALK, etc.

ALKs in solutions have been studied mainly by spectrophotometric, spectrographic methods, and potentiometric titration methods. To determine the stability constants of ALK, the Irving Rossotti method is used, as well as the Berrum, Thompson, and Loraas methods for determining the stepwise formation constants [5].

A fundamentally new approach to determining the stability constants of complexes - the potentiometric surface method, proposed by Lefebvre, deserves special attention, which can be applied to systems in which two different ligands are simultaneously attached to the complex-forming ion. The most complete information about the formation of ALKs of metals is provided by the mathematical modeling (MM) method [6].

Currently, mixed-ligand complexes have been widely studied, in which the main ligand is a complexon, and the additional ligands can be different. In complexes of intermediate elements with mixed ligands, the coordination number of metals is 6, and the dentancy of the ligands is less than 6 [7]. Binary complexes of d-elements contain water molecules MA·nH2O, which, when forming ALK, can be replaced by a second ligand without changing the structure of the main complex. The formation of ALK of d-metals, the coordination sphere of which contains two different complexons, can occur in two ways: when the complex is formed, the main complex is more stable, and the remaining part of the second complexon is not destroyed by the addition, or the two ligands interacting with the d-metal ion have approximately the same complex-forming ability, and the composition of the complex is determined by the ratio of ligand concentrations [8].

METHODOLOGY

In the synthesis of complex compounds, the metal salts in the form of crystalline hydrates were used: copper (II) chloride, acetate and nitrate salts of "chemically pure" grade. The organic solvents used were purified and dried according to the known method. The IR absorption spectra of the compounds were studied in the range of 400-3800 cm-1 on an Irrafinity-1S "Shimadzu" (Japan) spectrophotometer using a sample in the form of a KBr tablet with a diameter of 7 mm. The identification of complex compounds was carried out on a powder diffractometer XRD-6100 (Shimadzu, Japan). It was carried out under the influence of CuKα radiation (β-filter, Ni, λ=1.54178Å, current and voltage in the X-ray tube 30 mA, 30 kV). The detector rotation speed was 4 degrees/min, in 0.02o steps (ω/2θ-coupling), and the scanning angle was from 40o to 80o. Derivatography results were analyzed to determine the thermal stability of the complexes and the presence of water molecules in the complex. The composition and structure of the complexes of metal salts were studied using thermal analysis. The complex compounds were synthesized according to the known [9] methodology. According to it, copper salts and ligands in different 1:1:1 ratios, i.e. (0.01 mol) 0.59 g of acetamide (AA), 1 mol of acetylacetone with a mass of 100 g/mol, density of 0.98 g/ml (0.01 mol), 0.103 ml of acetylacetone (AcAc) and 0.25 g (0.01 mol) of copper (II) chloride crystalline hydrate were mixed in a mortar. The mixture was dissolved in 50 ml of ethanol, kept in a water bath for 30 min., then removed for crystallization. After 3 days, small crystals fell out, which were filtered and washed several times in ethanol. The yield of the resulting blue mixed ligand complex of the copper salt = 68 %. Melting point = 260- 270 °C

RESULTS

The color, yield, liquefaction temperature, and elemental analysis results of the complex compound are presented in Table 1, and the solubility of the complex compound in solvents is presented in Table 2.

Table 1.
Characteristics of the ligand and synthesized complex compounds

Compounds	Color	Yield, %	Melting point, °C	C (%)	N (%)	M (%)	Empirical formula
[CuAcAcAACl]	Blue	68	260–270	32.62	5.44	24.8	$C_7H_{12}O_3NClCu$

Table 2.
Solubility of the synthesized mixed-ligand complex compounds

Compounds	Water	Ethanol	Acetic acid	Benzene	DMF	DMSO
[CuAcAcAACl]	SP	SP	SP	NS	SP	SP

Note: SP – slightly soluble; S – soluble; NS – not soluble.

In the IR spectrum of the complex compound [CuAcAcAACl], the symmetric and asymmetric vibration frequencies of the carbonyl group in the acetamide molecule are displayed in the 1806 and

1576 cm-1 regions. In addition, absorption lines at 416 cm-1 appear in the short-range, which belong to the N⟶M valence vibrations, indicating that the acetamide molecule is coordinated through the nitrogen atom in the amino group. In the 534 cm-1 region of the spectrum, the M-O bond vibrates, in which the bonding occurs through the oxygen atom in acetylacetone.

As a result of the bonding of one of the two oxygen atoms in the acetylacetone molecule to the metal atom through an ionic bond and the other through a coordination bond, the double bonds of the oxygen atoms are opened, and the ν (C=C) + (C=C) bond, which occurs due to the delocalization of the bond in the formed chelate, is observed to vibrate at 1520 cm-1.

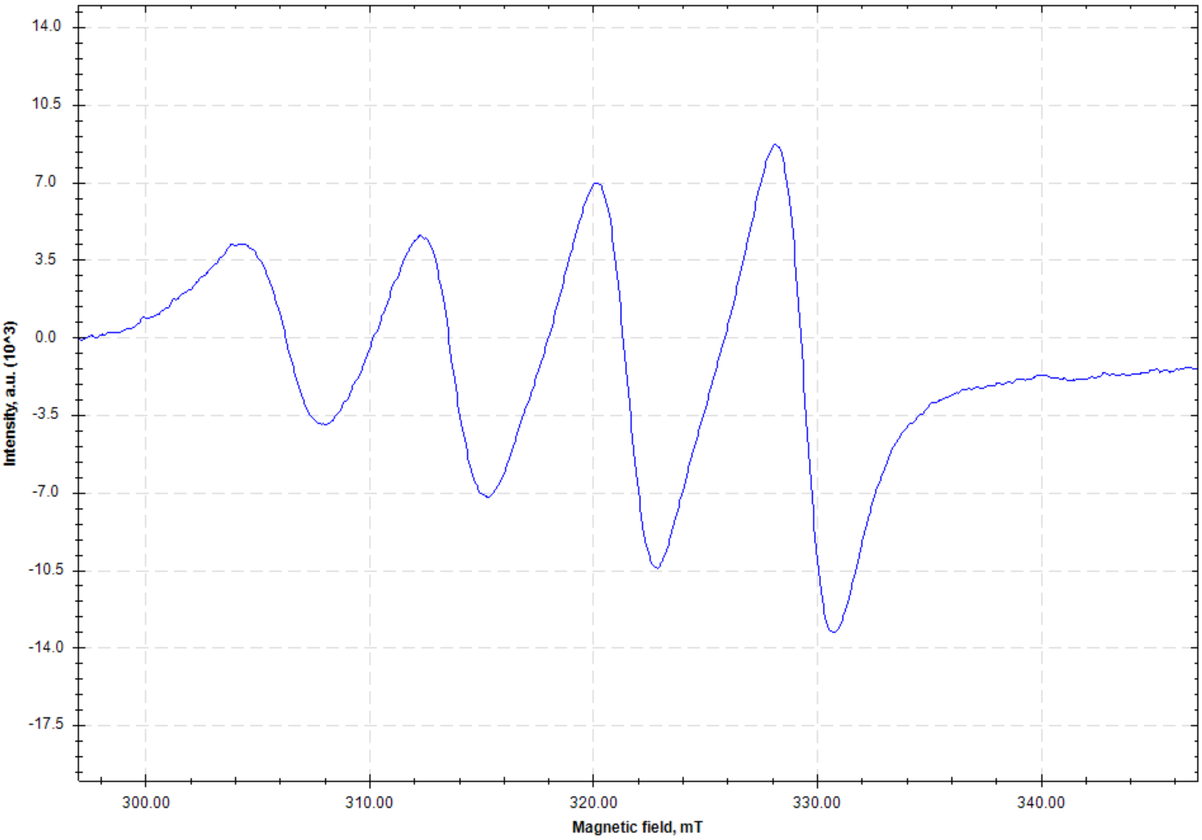
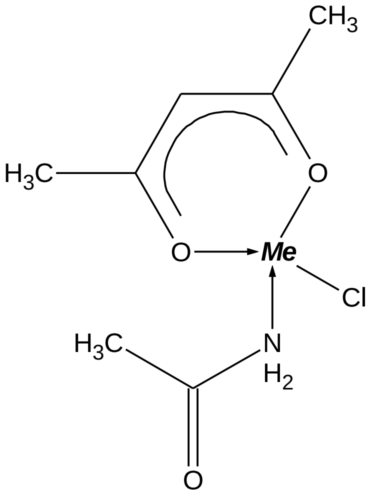


Figure 1. EPR spectrum of [CuAcAcAACl].

The EPR spectra of 3d-metal complexes contain a lot of information about the electronic structures of these complexes. The EPR spectra of 3d-metals are determined depending on the state of the d electrons in them in a magnetic field. These features cause orbital and zero-field effects. The EPR spectra of the synthesized complex compounds were determined in the X-band (λ=3 cm, ν = 9500 MHz, Ho=3400 e). Conclusions The obtained complex compounds have an effective magnetic moment of 1.73-1.74 B.M., calculated according to the formula $\mu_{эфф} = \sqrt{8\chi^{1} \cdot m^{T}}$ which is almost independent of temperature. This indicates that the metal chelate is stable. The EPR spectra of solid polycrystalline samples of these complexes do not contain lines of "forbidden" transitions at low fields (N≅1700 e). These values of the magnetic moments indicate that all molecules of the complexes are monomers and that antiferromagnetic exchange interactions do not occur between them. The influence of the substituents in the hydrazone fragment on the electronic properties of copper (II) complexes was studied by the EPR method in alcohol or chloroform solutions at room temperature. The complexes with a planar-square structure studied by us have isotropic EPR spectra in solutions, which are similar to the spectra of previously studied compounds and are characteristic of mononuclear complexes of copper (II). It was concluded that the copper (II) complex has a planar-square structure. Based on physicochemical studies, it was concluded that the structure of the complex compounds is acetamide metalacetylacetone in a 1:1:1 ratio, and the structure of the complex compound synthesized with Cu (II) metal salts was suggested as follows:



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