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NMR SPECTROSCOPIC INVESTIGATION OF SUCCINIC ACID DIHYDRAZONE WITH 1-FERPOCENYLBUTANEDIONE-1,3

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Annotation: *Succinic acid dihydrazone 1-ferrocenylbutanedione-1,3 (H_2L) was synthesized by the reaction of dicarboxylic acid dihydrazide with ferrocenoylacetone. The NMR spectra of the synthesized compounds in DMSO-d_6+CCl_4 solution were studied.*

Keywords: *monoacetylferrocene, dihydrazone, Claisen ester condensation, NMR spectroscopy.*

The interaction of an alcoholic solution of 1-ferpocenylbutanedione-1,3 and an ethanol suspension of succinic acid dihydrazide in molar at a ratio of 2:1, a new ligand (H_4L) was isolated, in which two hydrazone fragments are connected via methylene bridges. To establish the composition and structure of the obtained ligand in the solid state, we used the data of elemental analysis, IR spectroscopy, and in solution it was studied using the IR spectroscopy data. The ^{1}H NMR spectrum of the H_4L ligand in a DMSO-d_6+CCl_4 solution indicates the retention of the rectilinear diketo form [1,2,3,4,5, 6,7].

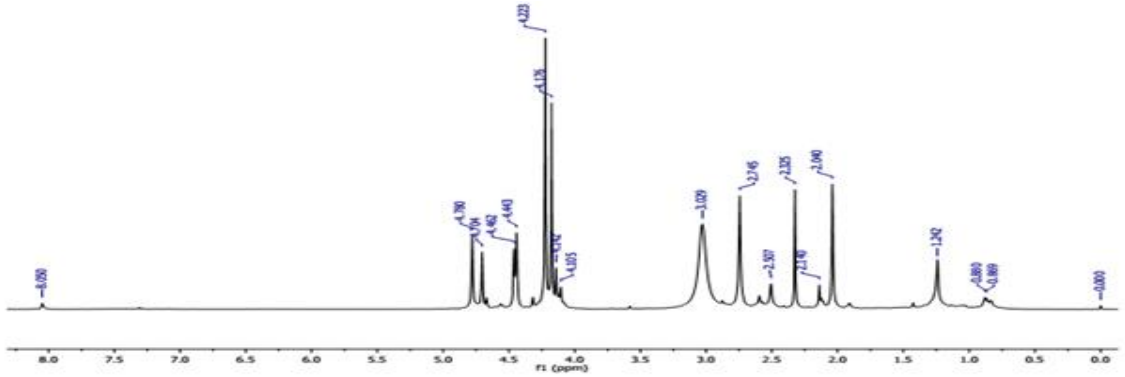

Fig. 1. ^{1}H NMR spectrum of H_4L ligand in ДМСО-d_6+CCl_4. solution.

Signals from two bridging –$(CH_2)_2$– groups bound to amide C=O substituents of the ligand with an intensity of four protons were noted in the region at δ 2.75 ppm. [1,2]. Unequal proton signals of two cyclopentadienyl rings were fixed at 4.46 (2H), 4.78 (2H), and 4.70 (5H) ppm. The signals of protons of two methyl groups (6H) in the spectrum were recorded in the high-field region at δ 1.24 ppm. in the form intense singlet. And protons of the N–H groups (2H) resonate in the region of weak fields in the form of singlet signals at δ 10.01. Thus, the most weak-field signal was assigned by us to the proton of the hydrazone group. After 4-5 minutes, a second set of signals appears, belonging to the enhydrazine form. The change in the H_4L spectra stops after a few days and equilibrium sets in between the stereoisomers of the hydrazone, enhydrazine, and cyclic forms [8,9,10].

The diketone form of the H_4L ligand is confirmed by the ^{13}C NMR spectrum (Fig. 2). The ^{13}C NMR spectrum of the H_4L ligand showed signals at δ 16.83 (CH_3); 39,52; (CH_2); 67,96 ($C^{2,5}$ Fc); 69,54 ($C^{3,4}$ Fc); 70,60 (5C Fc); 78,72 (C Fc); 171,56 (C=O); 176,36 (C=N) ppm. Low intensity signal at δ 171.56 ppm refers to the carbon atom of the C=O group [11,12,13].

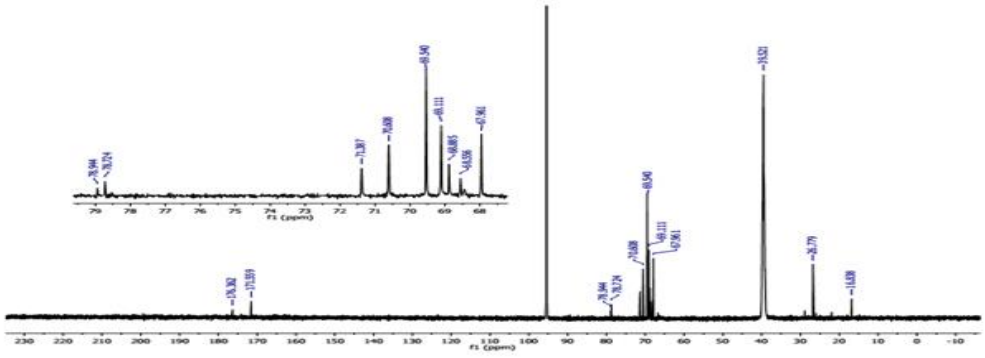

Fig. 2. ^{13}C NMR spectrum of H_4L ligand in DMSO-d_6+CCl_4 solution.

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INVESTIGATION OF COMPLEX COMPOUNDS OF TRANSITION METALS WITH DIHYDRAZONE OF SUCINIC ACID BASED ON FERROCENOLACETONE

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Annotation: *We obtained by Claisen condensation β-diketone - 1-ferrocenyl-butanedione-1,3. The dicarboxylic acid dihydrazone of 1-ferrocenylbutanedione-1,3 (H_4L) was synthesized by reacting succinic acid dihydrazide with ferrocenoylacetone in a 2:1 ratio. Based on them, homobinuclear complex compounds with copper(II), zinc(II), and nickel(II) ions were obtained. The IR spectra of the synthesized organic compounds have been studied. According to the results of spectroscopic studies, the complexes were assigned a square planar structure, where the four times deprotonated ligand residue is coordinated by each metal atom through two oxygen atoms and a nitrogen atom of the hydrazone fragment. The fourth position in the planar square of the trans-N2O2 coordination site is occupied by the ammonia molecule. Planar five- and six-membered metal cycles of synthesizers are practically coplanar with each other.*

Key words: *Claisen ester condensation, ferrocenoylacetone, succinic acid dihydrazone, tautomerism, spectroscopy*

We have synthesized the H_4L ligand by the condensation of 1-ferpocenylbutanedione-1,3 with succinic acid dihydrazide. It should be noted that such ligands are characterized by the formation of complexes of a homo- and heterobinuclear nature. We have synthesized complexes with a homobinuclear structure [1,2,3,4,5].

Using the data of elemental analysis and IR spectroscopy, we determined the composition and structure of the synthesized ligands in the solid state, and the structure in solutions was studied by the 1-speak method.

Using the data of elemental analysis and IR spectroscopy by us the composition and structure of the synthesized ligands in the solid state, and the structure in solution was studied by ^{1}H NMR spectroscopy.

The complexing ability of the H_4L ligand is due to the presence in the connection of several donor centers connected by a system of conjugated bonds, and in addition, a mobile hydrogen atom. Upon the interaction of an alcoholic solution of the H_4L ligand with aqueous ammonia solutions of Ni(II), Cu(II), and Zn(II) acetates in a molar ratio of 1:2, complex compounds were isolated. Based on the results of elemental analysis of the complexes, the general formula $M_2L{\cdot}2NH_3$ [6,7,8,9,10].