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SYNTHESIS AND STUDY OF COMPLEX COMPOUNDS OF SOME 3d-METALS WITH 1,3-DIBENZYLQUINAZOLINE-2,4-DIONE

Каримова Гавхар Шовкатжановна
Candidate of Chemical Sciences, Senior Researcher,
National Research Institute of Renewable Energy
Sources under the Ministry of Energy of the Republic of Uzbekistan

Abstract

The ligand 1,3-dibenzylquinazoline-2,4-dione and its complex compounds of chlorides, nitrates and zinc(II acetates) were synthesized. The composition and structure of the synthesized compounds were studied by methods of elemental analysis, IR and NMR^{1}N-spectroscopy. The displacement of all signals responsible for hydrogen-containing functional groups in the ligand molecule to the low field region, as well as the appearance of a new signal from acetate acidoligand protons, indicates what is happening coordination to the complexing ion. The results of NMR^{1}H spectroscopic study of the structure of the synthesized complexes confirmed the conclusions obtained by IR spectroscopy in terms of the location of acidoligands in the internal sphere of the complex compound.

Keywords: d-metal y,1,3-dibenzyl quinazolin-2,4-dione, ligand, IR and PMR spectrum, synthesis, analysis, preparation, herbicide, fungicide, bactericide, rost-regulating

Currently, the chemistry of heterocyclic compounds is developing rapidly, which is associated with the physiological properties of these compounds. Among them there are highly effective drugs, chemical plant protection products against pests. On their basis, dyes, monomers, heat-resistant fibers, polymeric materials and many other practically valuable substances have been created. The most widely used preparations containing nitrogen-containing five- and sixth-weighted heterocyclic substances System. Such systems include quinasolins [1], which have condensed pyrimidine and benzene nuclei and possess unique chemical and pharmacological properties.The bulk of the substances used in the national economy are organic compounds and every day the demand for them is growing. Therefore, the development of convenient, simple methods for obtaining heterocyclic compounds is one of the urgent tasks facing chemists.In solving this problem, the purposeful synthesis of coordination compounds of transition metals with physiologically active organic compounds can provide significant assistance.

It is known that the introduction of vital metal ions into the composition of biologically active preparations not only reduces their toxicity, but in most cases increases biological activity and often leads to new biological properties [2,3].

In coordination chemistry, there are a large number of different ligands that vary greatly in properties and structure [4-5]. One of the important classes of such compounds are quinazolin-2,4-dione and their derivatives [6], which are quite widespread in plant and biological objects. Consequently, the study of the complexing abilities of quinazolin-2,4-dione derivatives is an urgent problem in the chemistry of coordination compounds. Quinazolins have also important from a practical point of view[7-9]. On the basis of these compounds, many biologically active substances have been created - herbicides, pesticides, fungicides, bactericides and pharmacologically active drugs [10].Therefore, the search for biologically active compounds in this series is of particular practical interest.

The determination was carried out by the method of double serial dilutions in a liquid nutrient medium for pharmacopoeial strains: *Staphylococcusaureus*ATTS 6538-P, *Escherichiacoli*ATSS 25922. The bacteriostatic effect of the studied compounds was compared with the action of dioxidine and ethacridinalacate.As a result of the studies, it was found that it is most appropriate to search for highly active compounds with this type of activity in the series N-acylhydrazids N-acyl-5-bromo(iodine)anthranyl acids, 6-bromo(iodine)-2-phenyl-3-aznlaminoquinazolin-4-(ZN)-ons and 2-substituted-Z-(K-benzilidenylino)-6-bromo(iodine)quinazolin-4(ZN)-onov[11].

The interaction of 1,3-dibenzoyl quinazolin-2,4(1 N,3*N*)-dione with potassium carbonate in the environment of anhydrous DMFA at room temperature leads to the formation of a potassium salt of 3-benzoylquinazolin-2,4(1 N,3*N*)-dione, the alkylation of which *insitu*benzyl chloride gives 1-benzyl-3-benzoylquinazoline-2,4(1 N,3 *N*)-dion. A one-step method for obtaining N1-mono-substituted derivatives of quinazolin-2,4(1 N,3*N*)-dione has been developed. The influence of the nature of the alkaline reagent on the yield and ratio of debenzoylation products was studied [12].

Despite a significant number of works devoted to the study and properties of quinazolin-2,4-dione and their derivatives, their complex compounds are practically little studied.

Quinazolin derivatives are also able to form complex compounds with various metals. For example, the interaction of 3-mercaptoethyl quinasolin-2,4-dione, salicylic aldehyde and o-aminotionphenol with displaced salts of rhenium(V) leads to the formation of (2-(3-quinazolin)ethyl thiolato)-N-(2-mercaptophenyl)-salicyldeniminato-oxo-rhenium(V).When 3-mercaptoethylquinazolin-2,4-dione reacts with 3-thiapentan-1,5-dithiolate in the presence of mixed rhenium salts (V)) dimethylformamide produces dimethylformamide solvate (2-(1H, 3H-quinazolin-2,4-dione-3-yl)ethyl thiolato)-(3-thiapentane-1,5-dithiolato)-oxo-rhenium(V) [13].

It should be noted that the quinazolin-2,4-dione analogue quinazolin-4-one can form complex compounds with d-metal salts. For example, [14] its interaction with $CuCl_2$ leads to the formation of diaqua-dichloro-bis[quinazolin-4(1H)-on-kN^3]-copper(II).Trimethylolol(IV) chloride) with 2-mercaptoquinazoline-4 forms a bond with both the sulfur atom and the nitrogen atom, resulting in trimethyl-(2-mercapto-4-hydroxyquinazolin-N,S)-tin(IV) [15].As a result of the interaction of quinazolin-4-one with cadmium chloride(II)) a complex compound is formed [16]. K.M. Fitchet and P.J. Steml managed to obtain a complex compound based on 2,2'-dichinazolin. The interaction of the latter with palladium(II) chloride carried out the synthesis of (2,2'-dichinazolin)-dichloro-palladium(II) [17].The same authors managed to synthesize the cadmium complex 2,2'-dichinazolin - aqua-(2,2'-dichinazolino)-dinitrate-cadmium(II) [18 Quinazolin derivatives with both substituents in the heterocol and in the aromatic nucleus can also enter the chelation reaction. For example, 6-bromo-1,4-dihydro-4-hydroxy-1-methyl-4-(2'-pyridyl)-quinazolin with $CuCl_2$ forms bis-(μ 2-chloro)-bis-(6-bromo-1,4-dihydro-4-hydroxy-1-methyl-4-(2'-pyridyl)-1,3-quinazolin-N,N')-dichlorodimedi(II) [19]:

The purpose of this work is to develop methods for the synthesis and production of complex compounds of chlorides, nitrates and zinc(II) acetates with1,3-dibenzylquinazoline-2,4-dioneom. Study of the structure and properties by physicochemical methods of analysis.

In this work, the following salts were used in the form of crystal hydrates: chlorides, acetates and nitrates; zinc(II) - all salts of the brand "c.d.a.". An end was used to synthesize the ligands . HCl, tetrabutylammonium bromide, benzene, benzyl chloride, propionic acid and acetic acids of grade "h".The organic solvents used were also purified and dried by known methods [20].

The analysis of synthesized complex compounds for metal content was carried out on the atomic absorption spectrophotometer "Perkin-Elmer-432" (USA). Elemental analysis for nitrogen and oxygen content - on the device "EA 1108" company Carlo-Erba (Italy).

Irspectra were filmed on the Fourier SpectrumGX spectrometer by Perkin-Elmer (USA), in tablets, recorded in the region of 400-4000 $^{cm-1}$compressed with KBrdiameter with a resolution of -4 13 мм$^{cm-1}$on the Perkin-Elmer model 2000 device in petroleum jelly.

NmR ^{spectra} ^{1}H were filmed on the VarianInova-400 UNITY 400^{+} (400 MHz) spectrometer in DMSO, an internal TMS standard. The values of $R_f$are determined on the plates "Sorbfil" (Russia) and "Whatman®UV-254" (Germany),, the eluent is a mixture of benzene with ethanol 5: 1; developers: $KMnO1$ г$_4$ + 4 ml H_2SO_4 + 96 ml H_2O, UV light.Melting point of synthesized substances was determined on the device "Boetius" (Germany) and "MEL-TEMP" (USA).

Thermal analysis was recorded on the derivatograph of the Paulik-Paulik-Erdey system at a speed of 10 degrees / min and a hitch at the sensitivity of galvanometers T-9000,10 г ^{O}S, TG-200, DTA-1 /80, CTG-1 / 10. The recording was carried out under atmospheric conditions with constant use of a pump. The holder was a corundum crucible with a diameter without a cover. Al10 мм $_2O_3$ was used as a standard.

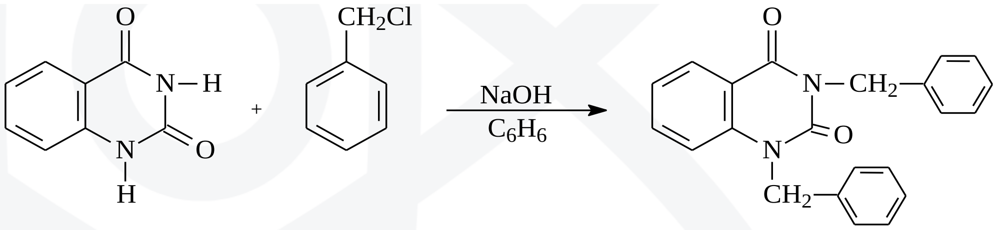
Diffractograms of the complexes for the purpose of their identification were filmed on a DRON-3.0 diffractometer in the following parameters: X-ray generator mode-36 kV, 15 mA; CuK- radiation (=1,542A), -filter, Ni; /20-clutch; exposure interval 5 0 30; constant rotation speed of the detector 1 degree/min; write speed 12 mm/min.$_\infty$λβω°≤≤°

X-ray diffraction analysis (PCA) of single crystals of 1,3-dibenzylquinazoline-2,4-dione was carried out on the automatic diffraktometerOxfordDiffraction-2009 (MoK, graphite monochromator, /2-scan, $2\lambda\alpha\theta\theta\theta_{max} = 56^{o}$). The structure was deciphered by a direct method using a complex of programs SHELXS-97 [21] and refined using the SHELXL complex -97 [22]. Hydrogen atoms were set geometrically and refined according to the rider's model. Molecular graphics were carried out by the XP program in SHELXTL-Plus [23].

Quantum-chemical calculation of the reactivity of ligands was carried out by semi-empirical quantum-chemical methods for calculating MNDO, AM1 and PM3 given in the HyperChemData program complex [24].

For the first time, methods for synthesis were developed and coordination compounds of zinc (II) with 1,3-dibenzylquinazolin-2,4-dione were obtained. As a result of the research, 3 previously unknown metal complexes in the literature were synthesized.

The ligand 1,3-dibenzylquinazoline-2,4-dione (L) was synthesized according to the following reaction scheme:



To a mixture of (0.01 mol) of quinazolin-2,4-dione, (0.1 mol) NaOH in 40 ml of water, 1.28 g (0.004 mol) of tetrabutylammonium bromide (TBAB) and 40 ml of benzene were added (0.03 mol) of benzyl chloride. Heated to 601,62 г4 г3,79 г^{o}C and kept at this temperature for 6 hours. The organic layer was separated, washed to a neutral reaction, dried over Na_2SO_4, benzene evaporated, the residue was recrystallized from benzene. Yield of 1,3-dibenzylquinazolin-2,4-dione (**L**) (88%). T.pl.=125-127C. 3,0 r°$\mathbf{R_f}$=0,82 (benzene : acetone=5:1). Lit. t.pl. 123-125 °S.

The synthesis of the complexes was carried out according to the following technique: a saturated solution (0.002 moles) of zinc 0,684 racetate in 50 ml of xylene, heated to 125-130 0 C, was applied to a hot solution (0.002 moles) of zinc acetate in 50 ml of xylene with constant stirring and heated for 1 hour in a water bath with a reverse refrigerator. After cooling, a fine-crystalline white precipitate falls out of the solution, after two days the precipitate was filtered out, thoroughly washed with alcohol, ether and air dried. Product yield (51%), t.p. 130-132 0,219 r3,11 r°S.

Similarly, complex compounds of chloride, acetate and zinc(II) nitrate based on 1,3-dibenzyl quinazolin-2,4-dione were synthesized. The composition and individuality of the synthesized complexes were established using elemental analysis.

The main characteristics of all synthesized complex compounds are given in Table 1. From the analysis of the data obtained from the determination of the elementary composition of complex compounds, it can be concluded that in all synthesized complex compounds, the composition of the complexes corresponds to M: L 1: 2.

Table 1 Resource requirements by component

Characteristics of synthesized complexes based on 1,3-dibenzylquinazoline-2,4-dione

Compound	Exit %	T.pl. °S	Naydeno, %			Gross formula	Color	Inyhisleno,%		
			N	About	Me			N	About	Me
L	95	134-136	10.81	12.3	-	C_4:$_{14}$ p.m. N 2 O_2	White	10.53	12.0	-
$ZnLCl_2$	80	177-179	6.34	7.63	15.89	$ZnC_{16}H_{14}$N 2 O 2 Cl $_2$	White	6.96	7.96	16.17
$ZnL(NO_3)_2$	48	154-156	12.58	28.41	14.33	ZnC_4:$_{14}$p.m.N_6O_8	White	12.31	28.13	14.28
$ZnL(CH_3COO)_2$	55	166-168	6.42	21.30	14.07	ZnC_8:$_{20}$p.m.N_2O_6	White	6.24	21.38	14.48

L- 1,3-дибензилхиназолин-2,4-диона

The structure of the synthesized compounds is established using spectroscopic methods of analysis [25-26].

In the IR spectrum of 1,3-dibenzylquinazoline-2,4-dione (Fig.1), characteristic valence oscillations of carbonyl groups at positions 2, 4 are observed for ν_{as} (C = O) at 1763-1731 $^{cm-1}$, ν_s (C = O) at 1656-1697 $^{cm-1}$. Group of intense absorption bands in the mid-frequency region at 1607-1483 $^{cm-1}$ is attributed to fluctuations of the C-N heterocycle bond. Oscillations of methylene groups of benzene rings were detected in the high frequency region at 2969-3088 $^{\mathrm{cm-1}}$.

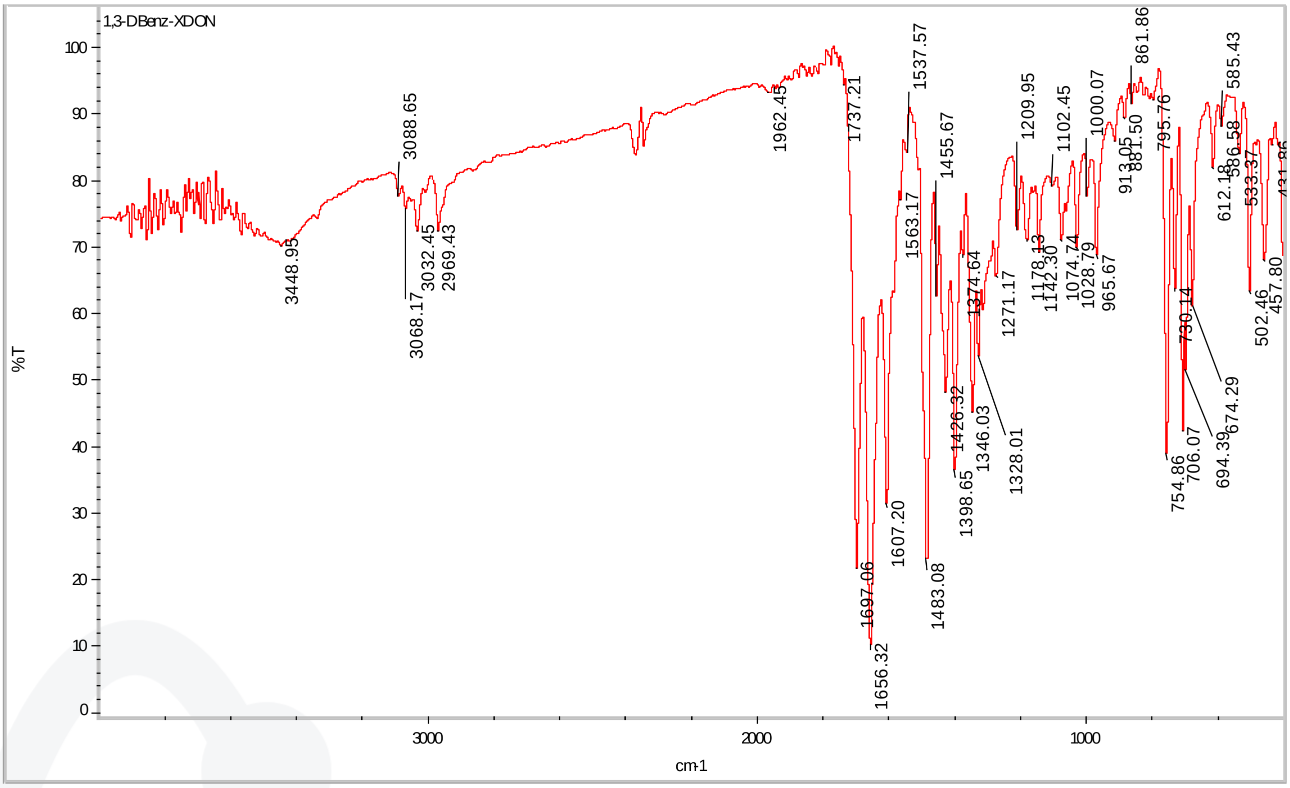


Fig.1. IR spectrum ligand 1,3-dibenzylquinazoline-2,4-dione

In the region of low frequencies, groups of bands with high intensity were found related according to [27-28] to oscillations of CH_2 groups of the ligand molecule. In the region of high frequencies at 3448 cm-1 in the form of an expanded band with low intensity, an absorption band is observed, attributed to oscillations of water molecules. Probably, moisture is present in the ligand.

The structure of the synthesized ligand1,3-dibenzylquinazoline-2,4-dione was additionally established by the NMR^{1}N spectroscopy method. In the NMR ^{1}N spectrum of the ligand in the weak field region, a group of multiplane signals is observed at 7.26-7.42, 7.85-.d., which are related to signals from protons of hydrogen-containing methylene groups of aromatic nuclei and from protons CH7.99 м$_2$ groups associated with aromatic cycles. Duplicate-doublet signals recorded at 8.19-.d. are assigned to the signals of neighboring CH8.38 м$_2$ groups of the quinazolindione ring [29-32].

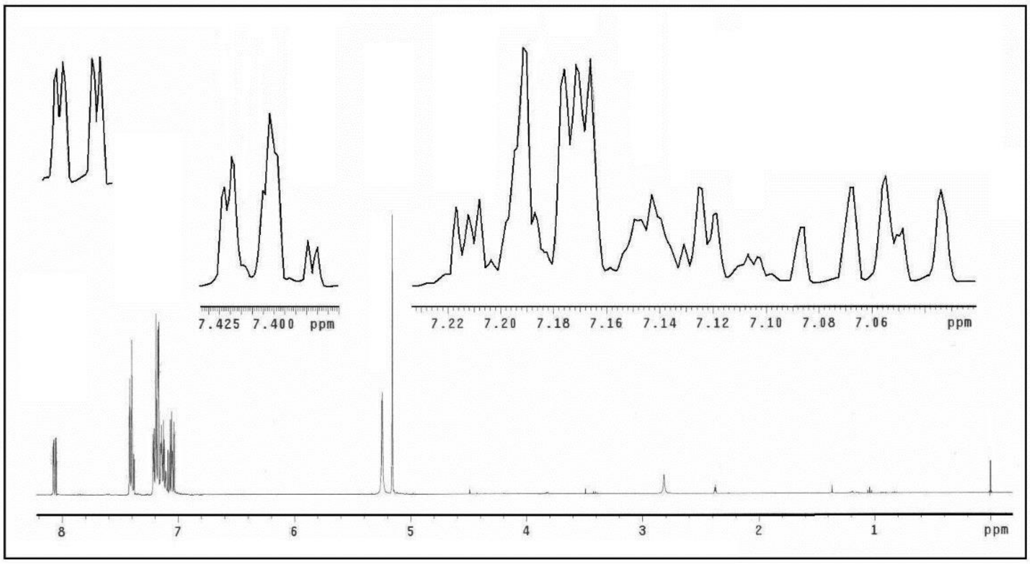


Fig.2. NMR ^{1}N-spectrum ligand 1,3-dibenzylquinazoline-2,4-dione

By the interaction of ligand solutions with the corresponding metal chlorides, nitrates and acetates in a mole ratio of L: M 2: 1, complexes with the formula ML_2X 2 common to all synthesized compounds were obtained, where: M - Zn (II); L-1,3-di benzyl quinazolin-2,4-dione; X - CI^-, NO_3^- and CH_3COO^-.

Table 2 Resource requirements by component
The main frequencies in the IR spectra are 1,3-dibenzylquinazoline-2,4-dionye of its complexes ($^{cm-1}$) (L)

Connection	$\nu_{(s)}$(C=O)	$\nu_{(as)}$ (C=O)	ν(C-N)	ν(N-C=O)	ν(O-M)
L	1656	1731	1182	2237	-
$ZnCl_2L{\cdot}H_2O$	1671	1710	1184	2242	546
$Zn(NO_3)2\ L{\cdot}H_2o$	1682	1725	1220	2240	542
$Zn(CH_3COO)_2\ L{\cdot}H_2o$	1674	1721	1237	2270	562

Analysis of the IR spectrum of the $ZnCl_2L{\cdot}H_2O$ complex showed that the acetate acidoligand is coordinated. This conclusion is made on the basis of finding in the IR spectrum a complex of new absorption bands absent in the spectrum of the free ligand at 1386 and 753 $^{cm-1}$, related according to [33-35] to symmetric valence oscillations of the COO-group bond and valence oscillations of the M-O bond.

A comparison of the IR spectrumof a$Zn(NO_3)_2L{\cdot}H_2O$ with the spectrum of the ligand showed that in the spectrum of the complex there is a band at 748 $^{cm-1}$, absent in the spectrum of the ligand, characteristic of oscillations, M-O bonds.

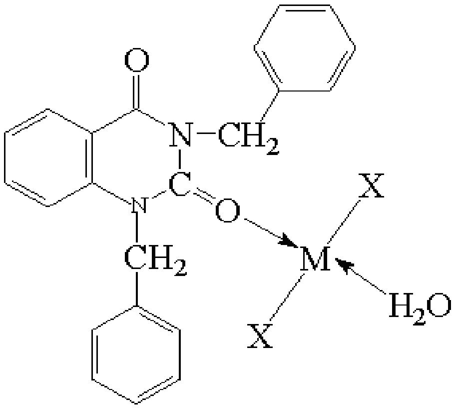
In addition, the new band manifested in the IR spectrum of the complex at 827 cm-1 is attributed to the $^{\text{extra-plane}}$ deformation oscillations of the coordinated nitrate group. As indicated, above, according to [36-37], during coordination, the valence oscillations of this group should be split into two bands. The bands found in the IR spectrum of the complex at 1473 and 1278 $^{\text{cm-1}}$ also testify in favor of the fact that nitrate acidoligand is intrasphere.

To supplement the results obtained by IR spectroscopy, a PMR spectroscopic study of the structure of synthesized zinc diamagnetic complexes was carried out.

As is known, the PMR method makes it possible to establish the presence of hydrogen-containing functional groups in the compound molecule by the resonance frequencies in the magnetic field, which are fixed by the PMR method, which are fixed by the PMR method. At the same time, the main characteristics of signals in the spectra of PMR are the values of chemical shifts, spin-spin interaction and the magnitude of the integral intensity of the curve. The magnitude of chemical shifts of protons is affected by the electronegativity and inductance of neighboring atoms in such a way that the greater the shielding of the electron density of a given proton under the influence of the local field of neighboring atoms, the more in strong fields a resonant signal of the proton is observed in the spectrum of PMP and, conversely, the phenomenon of descreening shifts the signal to the region of the weak field. That is, the position of the resonant signal (chemical shift) is determined by the totality of the electron density around the proton in question and the anisotropic effects of neighboring groups.

PMRspectra of chloride, nitrate and acetate complexes are slightly different from the PMR spectrum of the free ligand. In the PMR spectrum of complexes, all signals of hydrogen-containing functional groups are slightly shifted to the region of a weak field in comparison with their location in the PMR spectrum of the ligand, which indicates the ongoing reaction of complexation. In the PMR spectrum of the $ZnCl_2L{\cdot}H_2$complex, the dudelet-doublet signals of the benzene ring protons shift to the low field region and have centers at δ 7.32 and .d. In the NMR7.84 м$^{\text{of 1}}$N spectrum of the Zn(II) acetate complex in the strong field region at δ 1.90-.d. there is a singlet signal [1.94 м38-41], attributed to the protons of the methyl group acidoligand. The shift of all signals responsible for hydrogen-containing functional groups in the ligand molecule to the region of the weak field, as well as the appearance of a new signal from the acetate acidoligand protons, indicates the ongoing coordination to the complexing ion. The results of the PMR spectroscopic study of the structure of the synthesized complexes confirmed the previously obtained conclusions by ir spectroscopy about the location of acidoligands in the internal sphere of the complex compound.

Based on the discussion of the spectra of Zn(II) compounds with 1,3-dibenzylquinazoline-2,4-dione,it is possible to represent the spatial structure of the synthesized complex compounds in the form of:



гдеM = Zn(II),X-Cl$_2$, NO$_{3,}$

Based on the conducted spectroscopic studies, it can be concluded that the heterocyclic ligand is coordinated by the oxygen atom of the quinasolindione ring, which is an experimental confirmation of the theoretical conclusions obtained during the quantum chemical assessment of the reactivity of competing donor centers in the polydentate heterocyclic ligand molecule. Acidoligands are coordinated and are located in the internal sphere of coordination polyhedra.

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