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Theoretical aspects of weak exchange interaction in the ESR spectra of homobinuclear complexes of copper (II)

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ABSTRACT: The method of ESR is widely used for the investigation of electronic construction and geometrical structure of the polynuclear complexes of the transition metals. The majority of the well-known experimental works had been done using monocrystals and polycrystal examples. Construction of homobinuclear complexes of the copper(II), the nature of antiferromagnetic exchange interaction and its value was the object of discussion by the scientists and that issue is being checked in the article.

KEYWORDS: Homobinuclear complexes of the Cu(II), bicyclic hexadentate ligands, intermolecular antiferromagnetism, energy of exchange interaction..

I. INTRODUCTION

The ESR method is widely used to study the electronic structure and geometric structure of polynuclear transition metal complexes [1,2]. The vast majority of known experimental work was performed on single crystals, polycrystalline samples. In these cases, the ESR spectra of polynuclear complexes, as a rule, are not observed, due to broadening caused by anisotropic spin-spin interactions [3].

This report presents the results of a study of new binuclear Cu(II) complexes in liquid solutions of which the ESR spectra exhibit an ultrafine structure (CTC) from two equivalent Cu(II) atoms.

II. MATERIALS AND RESEARCH METHODS

The complex compounds were synthesized by the interaction of alcohol solutions of the corresponding organic ligands with pyridine solutions of metal acetate in a molar ratio of 1: 2.

$Cu_2L^1 \cdot 2Py$. To a hot solution of 3.26 g (0.01 mol) of oxohydrazone of salicylic aldehyde (H_4L^1) in 100 ml of ethanol was added with stirring a solution of 4.00 g (0.02 mol) of copper(II) acetate in a minimum amount of pyridine. The reaction mixture was heated for 30 minutes under reflux. The green solution was filtered and left in the air. 4.44 g (73%) of the $Cu_2L^1 \cdot 2Py$ complex of the composition $Cu_2C_{26}H_{20}N_6O_4$ were obtained.

Polycrystalline powders of copper(II) complexes with a coordinated pyridine molecule were obtained in a similar manner.

To obtain the heterobinuclear complexes Ni(II) and Cu(II) $NiCuL^6 \cdot 2Py$, it is necessary to mix the complex $Ni_2L^6 \cdot 2Py$ and $Cu_2L^6 \cdot 2Py$ in equimolar amounts. The yields and results of elemental analysis of the complexes in the table. 1.

The ESR spectra of polycrystalline samples, liquid and frozen solutions in toluene and other solvents were recorded on a SE/X-2542 radio spectrometer (Radiopan) with an operating frequency of 9.4 GHz. The stable radical diphenylpicrylhydrazyl (DPPH) was used as a standard. The experimental spectra were transferred through a special interface to the SM-4 computer, with which a theoretical reconstruction of the ESR spectra was performed. The parameters of the spectra were found by the best approximation between the experimental and theoretical spectra by minimizing the error functional according to the technique described in [4, 6, 17].

The ESR spectra of all compounds were studied in a solution of toluene, chloroform, previously degassed in vacuum. This was done in order to remove oxygen from the solutions, which broadens the ESR lines. Toluene and chloroform were chosen as a solvent because they do not form adducts and, as a result, do not contribute to the ESR

parameters. The concentration of solutions was $10 \cdot 10^{-2} \cdot 10^{-6}$ M. In some cases, measurements, to facilitate interpretation of the ESR spectra, were carried out on samples enriched with the ^{63}Cu and ^{65}Cu isotope to 99.8%.

Table 1.

Yields and results of elemental analysis of homo- and heterobinuclear complexes of copper(II) and nickel(II) with bis-(salicylidene) hydrazone of dicarboxylic acids

Compound	Output, %	$T_{\text{Melt}}, ^\circ\text{C}$	Gross formula	Found, %/ Calculated, %			
				Cu, Ni	C	H	N
$\text{Cu}_2\text{L}^1 \cdot 2\text{Py}$	73	210	$\text{Cu}_2\text{C}_{26}\text{H}_{20}\text{N}_6\text{O}_4$	21,06/20,92	51,33/51,40	3,24/3,32	13,92/13,83
$\text{Cu}_2\text{L}^2 \cdot 2\text{Py}$	75	232	$\text{Cu}_2\text{C}_{27}\text{H}_{22}\text{N}_6\text{O}_4$	20,80/20,54	52,06/52,17	3,45/3,57	13,63/13,52
$\text{Cu}_2\text{L}^3 \cdot 2\text{Py}$	80	248	$\text{Cu}_2\text{C}_{28}\text{H}_{24}\text{N}_6\text{O}_4$	20,10/19,99	52,82/52,91	3,74/3,81	13,31/13,22
$\text{Cu}_2\text{L}^4 \cdot 2\text{Py}$	72	253	$\text{Cu}_2\text{C}_{29}\text{H}_{26}\text{N}_6\text{O}_4$	19,83/19,56	53,54/53,62	3,96/4,03	13,05/12,94
$\text{Cu}_2\text{L}^5 \cdot 2\text{Py}$	68	265	$\text{Cu}_2\text{C}_{30}\text{H}_{28}\text{N}_6\text{O}_4$	19,32/19,15	54,18/54,29	4,17/4,25	12,75/12,66
$\text{Cu}_2\text{L}^6 \cdot 2\text{Py}$	80	276	$\text{Cu}_2\text{C}_{34}\text{H}_{36}\text{N}_6\text{O}_4$	17,88/17,66	56,61/56,73	4,95/5,04	11,78/11,68
$\text{NiCuL}^6 \cdot 2\text{Py}$	65	256	$\text{NiCuC}_{34}\text{H}_{36}\text{N}_6\text{O}_4$	8,35/8,21; 8,98/8,89	57,03/57,12	5,01/5,08	11,83/11,76

The static magnetic susceptibility of polycrystalline samples in the range of 77–300K was measured by the Faraday and Guy method using the facilities described in [16–17]. The obtained values were corrected for temperature-independent paramagnetism and ligand diamagnetism calculated according to the Pascal scheme [16-17]. $\text{Co}[\text{Hg}(\text{CNS})_4]$ was used as a reference sample.

Values μ_{eff} monomer complexes of Cu(II) was calculated by the formula:

$$\mu_{\text{eff}} = 2,83 \cdot (\chi_{\text{at}} \cdot T)^{1/2} \quad (1)$$

where $\chi_{\text{at}} = \chi_{\text{M}} - \chi_{\text{dia}}$.

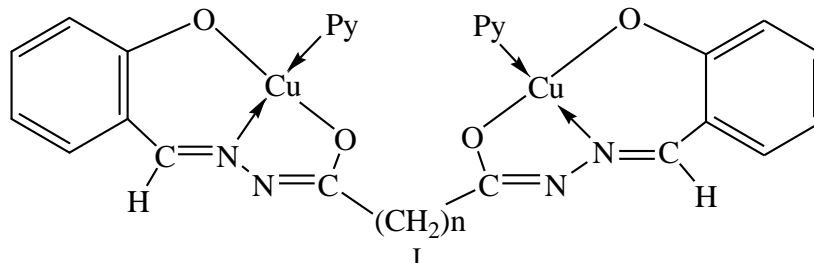
Values μ_{eff} Cu(II) dimeric complexes was calculated by the formula:

$$M_{\text{eff}} = 2,83 \cdot (\chi_{\text{Cu}} \cdot T)^{1/2} \quad (2)$$

where $\chi_{\text{Cu}} = 0.5 \chi_{\text{M}}$; $\chi_{\text{M}} = \chi_{\text{u,d}} \cdot M - \chi_{\text{dia}} - 2\alpha N$ (M - is the molecular weight of the monomer, α - is the admixture of the monomer). Error in the determination of μ_{eff} was ± 0.01 M.B.

III. RESULTS AND DISCUSSIONS

Homobinuclear copper (II) complexes of a new type, in which two chelated copper complexes with tridentate bicyclic ligands, interconnected by polymethylene bridges $-(\text{CH}_2)_n-$, $n = 0, 1, 2, 3, 4, 8$ schematically have a structure (I):



$n=0$ ($\text{Cu}_2\text{L}^1 \cdot 2\text{Py}$), 1 ($\text{Cu}_2\text{L}^2 \cdot 2\text{Py}$), 2 ($\text{Cu}_2\text{L}^3 \cdot 2\text{Py}$), 3 ($\text{Cu}_2\text{L}^4 \cdot 2\text{Py}$), 4 ($\text{Cu}_2\text{L}^5 \cdot 2\text{Py}$), 8 ($\text{Cu}_2\text{L}^6 \cdot 2\text{Py}$);

The synthesis of these compounds and preliminary data on their magnetic properties are given in [4,7].

The static magnetic susceptibility $\chi(T)$ was measured in the temperature range 77–300K for polycrystalline $\text{Cu}_2\text{L}^1 \cdot 2\text{Py}$ - $\text{Cu}_2\text{L}^6 \cdot 2\text{Py}$ (I) samples by the relative Faraday method. The obtained χ values were corrected for temperature-independent paramagnetism and ligand diamagnetism [5].

The ESR spectra of polycrystalline samples of liquid and frozen solutions of $\text{Cu}_2\text{L}^1 \cdot 2\text{Py}$ - $\text{Cu}_2\text{L}^6 \cdot 2\text{Py}$ in toluene or in a mixture of toluene + chloroform were recorded at 77 and 300K on a SE/X-2542 spectrometer of the Polish

company Radiopan. The theoretical processing of the ESR data was carried out on PC286 by the best approximation method between the experimental and theoretical spectra according to the technique described in [6].

To facilitate the interpretation of the ESR spectra, in some cases, samples enriched with the isotope ⁶³Cu and ⁶⁵Cu up to 99.8% were used.

The study of the static magnetic susceptibility showed that in Cu₂L¹·2Py the effective magnetic moment μ_{eff.} = 8χ_mT decreases from 1.62 at 300K to 1.26 MB at 77K. The experimental dependence μ_{eff.} (T) is well described within the framework of the model of isotropic antiferromagnetic exchange for two centers with spins S₁ = S₂ = 1/2 [5] for the values of the exchange parameter -2J = 80 cm⁻¹.

In the case of the complexes Cu₂L²·2Py-Cu₂L⁶·2Py, the effect is 1.8 M.B. and does not depend on temperature. This means that the compounds Cu₂L²·2Py-Cu₂L⁶·2Py contain only Cu(II) ions with S_i = 1/2. Since the lower boundary of the temperature range is T = 77 K, in the presence of exchange interactions in these compounds, estimates show that the exchange parameter should not exceed 10 cm⁻¹.

The ESR spectra of polycrystalline Cu₂L¹·2Py-Cu₂L⁶·2Py samples are exchange-narrowed lines. Anisotropy of the g-factor is allowed only for Cu₂L¹·2Py, which can be explained by the peculiarities of the crystal structure or the weakening of intermolecular interaction due to strong intramolecular antiferromagnetism. In addition, the forbidden transition Δm=±2 is observed in the ESR spectra of the Cu₂L¹·2Py complex at H ≈ 0.17 Tl. As is known [1-3], the intensity of this transition is ~|D|², where D is the parameter of the anisotropic spin-spin interaction. It follows that for the compound Cu₂L¹·2Py, the value of |D|² is maximum.

The ESR spectra of frozen Cu₂L²·2Py-Cu₂L⁵·2Py solutions have the form of a poorly resolved anisotropic HFS, which is generally not typical for mononuclear Cu(II) complexes. Since the fine structure due to anisotropic spin-spin interactions is also not allowed, it is reasonable to interpret such spectra as a superposition of the hyperfine and fine structure (TC) at low values of the TC and HFS constants.

The most interesting are the ESR spectra of liquid solutions, which represent the well-resolved HFS from two equivalent copper (II) ions (Fig. 1). Since similar spectra for binuclear copper(II) complexes are observed for the first time, let us dwell on their interpretation in more detail.

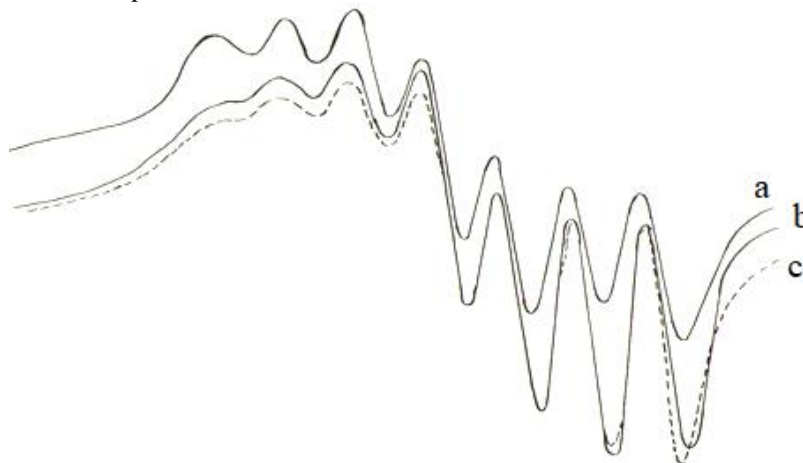


Fig. 1. ESR spectra for binuclear copper (II) complexes: Cu₂L³·2Py - (a) and Cu₂L⁵·2Py - (b) in a toluene solution at 300K, theoretical - (b).

As is known, the ESR spectra of mononuclear complexes s = 1/2 without taking into account additional HFS from ligands are described by the spin Hamiltonian (SH) [3]:

$$H_i = \beta S_i g_i H + S_i A_i I_i, \quad (3)$$

where g_i is the g-tensor; A_i is the HFS tensor from the central ion.

If two mononuclear fragments form a dimer in which paramagnetic ions are connected by isotropic exchange, then SH in the first approximation takes the form [4]:

$$H = H_1 + H_2 - 2gS_1S_2 = \beta(S_1+S_2)gH + (S_1+S_2)(A/2)(I_1+I_2) + (S_1-S_2)(A/2)(I_1-I_2) - 2gS_1S_2 = \beta S g H + S(A/2)(I_1+I_2) + (S_1-S_2)(A/2)(I_1-I_2) - g[S(S+1)-S_1(S_1+1)-S_2(S_2+1)] \quad (4)$$

where for $S_1 = S_2 = 1/2$ the total spin $S = S_1 + S_2$ takes the values $S = 0$ or ± 1 . There are no transitions inside the singlet state. However, as shown, for example, in [7], with a small value of $|g|$, singlet-triplet transitions or the nuclear states of a singlet and a triplet may be mixed by the operator $(S_1 - S_2)(A/2)(I_1 - I_2)$ included in (2). In both cases, a spectrum should be observed that is very different from a simple sequence of seven equivalent lines. Since precisely simple spectra are observed experimentally, the exchange parameter in (2) must satisfy the relation $|J| \gg \hbar\nu, |AA|$.

Subject to this condition, equation (2) is reduced to the effective SH of the triplet state (5):

$$H = \beta SgH + S(A/2)(I_1 + I_2) \quad (5)$$

From very general considerations it follows (3) that SH (3) should contain the contribution of the anisotropic spin-spin interaction, so that the total contribution of SH for the triplet state has the form

$$H = \beta SgH + S(A/2)(I_1 + I_2) + SDS, \quad (6)$$

where the tensor D is the sum of the so-called exchange (D_{ex}) and dipole-dipole (D_{dd}) contributions. For D_{ex} , the estimate [2] holds:

$$D_{ex} \sim (\Delta g / g)^2 J, \quad (7)$$

In the approximation of point dipoles [7]

$$D_{dd} \sim \beta^2 / r^3 \quad (8)$$

where r - is the metal-metal distance. In the complexes $Cu_2L^2 \cdot 2Py - Cu_2L^5 \cdot 2Py$, the value of r seems to exceed 5 \AA ; therefore, for them, $D_{dd} < 0,01 \text{ cm}^{-1}$. The estimate of D_{ex} depends on the degree of anisotropy of the g -factor and the magnitude of the exchange parameter. As is known [3], for complexes of the type in question $(\Delta g/g) \sim 0,1$. Therefore, for them $D_{ex} \sim 0,01J$. From this, in particular, it follows that for $Cu_2L^2 \cdot 2Py$, D_{ex} can reach $\sim 1 \text{ cm}^{-1}$. Such a large value of $|D|$, apparently, explains the features of the ESR spectra of this compound. For $Cu_2L^2 \cdot 2Py - Cu_2L^5 \cdot 2Py$, the values of J are unknown. However, the results obtained and formula (7) allow us to evaluate them.

Indeed, in the experimental spectra of liquid solutions, the effects of rotational averaging lead to the complete disappearance of anisotropy. In accordance with the theory of rotational relaxation [8], this means that $|\Delta gH|, |AA|, |AD|$ are much lower than the frequency of rotation of the molecules $1/j_R$. In mononuclear complexes, $\Delta g\beta H, |AA| < 1/j_R$. However, it follows from the form of the spectra of frozen $Cu_2L^2 \cdot 2Py - Cu_2L^4 \cdot 2Py$ solutions that $|D_{ex}| \sim |D| \sim |\Delta g\beta H|, |AA| \sim 0,01 - 0,02$ for them. Therefore, for them, the averaging condition $|D| < 1/j_R$ is satisfied. In addition, from the relations $|D_{ex}| \sim |D| \sim |\Delta g\beta H|, |AA| \sim 0,01 - 0,02 \text{ cm}^{-1}$ and $|g| \sim 100 |D_{ex}|$, we easily find that $|J| \sim 1 - 3 \text{ cm}^{-1}$.

It should be noted that this estimate does not contradict the magnetochemical data and is in good agreement with the experimental value of $-2J = 1,5 \text{ cm}^{-1}$ obtained in [9] for the bidentate complex $Cu(II)$, which assumes the presence of an exchange channel through a saturated chain $-(CH_2)_2-$.

Table 2.

ESR parameters for complexes $Cu_2L^2 \cdot 2Py - Cu_2L^5 \cdot 2Py$ and $NiCuL^6 \cdot 2Py$

Соединение	$g \pm 0,003$	$A_{Cu} \cdot 10^{-4}, \text{ cm}^{-1}$	α	β	γ	Δ
$Cu_2L^2 \cdot 2Py$	2.092	43.6	27.5	6.1	0.28	-0.1
$Cu_2L^3 \cdot 2Py$	2.094	44.5	27.8	7.0	0.46	-0.29
$Cu_2L^5 \cdot 2Py$	2.097	45.7	26.1	7.2	0.56	6.7
$Cu_2L^6 \cdot 2Py$	2.105	≈ 82.0	-	-	-	-
$NiCuL^6 \cdot 2Py$	2.105	$76.6^* ; 82.1^{**}$	28.3	13.3	2.5	-

Note: * ACu for isotope 63. ** ACu for isotope 65.

Since the inequalities $|J| \gg \hbar\nu, |AA|, |D| < 1/j_R$ are valid for $Cu_2L^2 \cdot 2Py - Cu_2L^5 \cdot 2Py$, the ESR spectra of their liquid solutions should be well described by SH (5). In order to verify this conclusion, we processed the experimental data by the best approximation method between the experimental spectra and the theoretical ones corresponding to SH (5). Since the splitting of the HFS is small ($\sim 0,04 \text{ T}$), we used the solutions of SH (5) in the first order of perturbation theory:

$$\hbar\nu = g_0\beta HM + (A/2)M(m_1 + m_2) \quad (9)$$

where $M = 1, 0, -1$ are the projections of the electron spin, and $m_1, m_2 = -I_i \dots I_i$ is the nuclear spin (for copper atoms $I_i = 3/2$). From here, the expressions for the resonance fields of the allowed transitions are easily found:

$$H_p = \hbar\nu / g_0\beta - (A/2) \cdot g \cdot \beta \cdot M \cdot (m_1 + m_2) \quad (10)$$

Theoretical spectra were constructed by summing the line shapes of individual transitions centered around H_p . As a function of the line shape, the Lorentz functions [10] were used. In accordance with the relaxation theory [8], the one-center contributions to the line width were specified by the expression

$$\Delta H_{p-p}(i) = \alpha + \beta m_i + \gamma m_i^2 \quad (11)$$

where m_i - is the projection of the nuclear spin; α , β , γ -parameters. Since the studied complexes with bridges $-(CH_2)_n-$ can exhibit stereochemical nonrigidity, the contribution due to the intramolecular motion in the binuclear complex around the polymethylene bridge was also taken into account [13]:

$$\Delta H_{p-p}(1,2) = \delta(m_1 - m_2)^2 \quad (12)$$

The parameters of the theory g_0 , A , α , β , γ , δ were varied until the minimum of the error functional was reached:

$$F = 1/N \left[\sum_{i=1}^N (Y_i^{\text{exp}} - Y_i^{\text{theor}})^2 \right] \quad (13)$$

where Y_i^{exp} and Y_i^{theor} are the experimental and theoretical values of the ESR absorption at B discrete points of the spectra. The reliability of the results was ensured by a large number of experimental points ($N \sim 500-1000$) and small residual values of F .

The found best approximation parameters for the minimum residual values of F are given in the table.

Consider the results.

First of all, we note that the values of $g_0 = 2.10$ are close to those known for mononuclear Cu (II) complexes with a similar coordination sphere [2O, 2N] [12]. This means that dimerization does not lead to significant changes in the geometric and electronic structure of mononuclear fragments. The analysis of the values of the broadening parameters α , β , and γ of the relation between them ($\alpha \gg \beta > \gamma > 0$) is the same as in the majority of mononuclear Cu (II) complexes with plane-square geometry [9]. A slight increase in α, β and γ in the series of $Cu_2L^2 \cdot 2Py - Cu_2L^5 \cdot 2Py$ compounds is easily explained by an increase in the size of molecules, which leads to an increase in the rotational correlation time τ_R .

On the other hand, the HFS constant from copper atoms is two times smaller than in the monomer complexes of copper with the coordination sphere [2O, 2N] and exactly corresponds to the similar constant and dimers of copper, which is observed in polycrystalline powders at the forbidden transition $\Delta m_i = \pm 2$ [1, 13].

The broadening parameter δ during the transition from $Cu_2L^2 \cdot 2Py$ to $Cu_2L^4 \cdot 2Py$ and $Cu_2L^5 \cdot 2Py$ increases almost by an order of magnitude. If we take into account that the appearance of this parameter is associated with dynamic spin exchange, which, in turn, is due to conformational transitions, then this result is quite reasonable: with increasing bridge length $-(CH_2)_n-$ stereochemical nonrigidity of dimeric molecules should increase.

Finally, we consider the $Cu_2L^6 \cdot 2Py$ complex with the bridge $-(CH_2)_n = 8$. As can be seen from Fig. 2a, in a liquid solution of $Cu_2L^6 \cdot 2Py$, it has an ESR spectrum with HFS of four lines characteristic of mononuclear Cu(II) complexes. The values of the ESR parameters given in the table are also typical of mononuclear complexes with the coordination sphere [2O, 2N]. As follows, for example, from [11], the appearance of such a spectrum means that for $V |J| \ll |A|$. Thus, upon the transition from $Cu_2L^1 \cdot 2Py - Cu_2L^5 \cdot 2Py$ to $Cu_2L^6 \cdot 2Py$, the energy of exchange interactions sharply decreases. This result is not surprising, since in the $Cu_2L^6 \cdot 2Py$ complex, exchange interactions are realized through a very long bridge $-(CH_2)_8-$ and their energy is, of course, very small. The noticeably broadened appearance of the HFS components is easily explained by the influence of weak dipole and exchange interactions. However, it should be noted that the distance in the spectrum between the 2nd and 3rd components is significantly less than between the others. The distance between the 1st and 2nd components, counting from the side of a weak magnetic field, is 0.087 T, between the 2nd and 3rd - 0.075 Tl, and between the 3rd and 4th - 0.090 Tl (Fig. 2 a). Usually, in mononuclear metal complexes, according to the Breit-Rabi equation [3,14], on the one side of the spectrum there is a minimum distance between the two extreme components, which gradually increases, on the other side of the spectrum, the distance between the components becomes maximum. For unambiguous proof of the considered version, we synthesized heterobinuclear complexes of nickel (II) and copper (II) with similar ligands. The complete identity of the IR spectra and the values of magnetic susceptibility convinces us of the isostructurality of homobinuclear and heterobinuclear complex compounds. As an example, we give an analysis of the ESR spectrum of one mixed complex of nickel and copper. It is this dependence in the distances between neighboring components that manifests itself in the spectrum of the Cu - Ni heterobinuclear complex with an octamethylene bridge ($NiCuL^6 \cdot 2Py$ compound) of structure (II) (Fig. 2b). In addition, on the high-field component of the HFS from the copper atom, the HFS of three lines from one nitrogen atom of the hydrazone ligand is allowed.

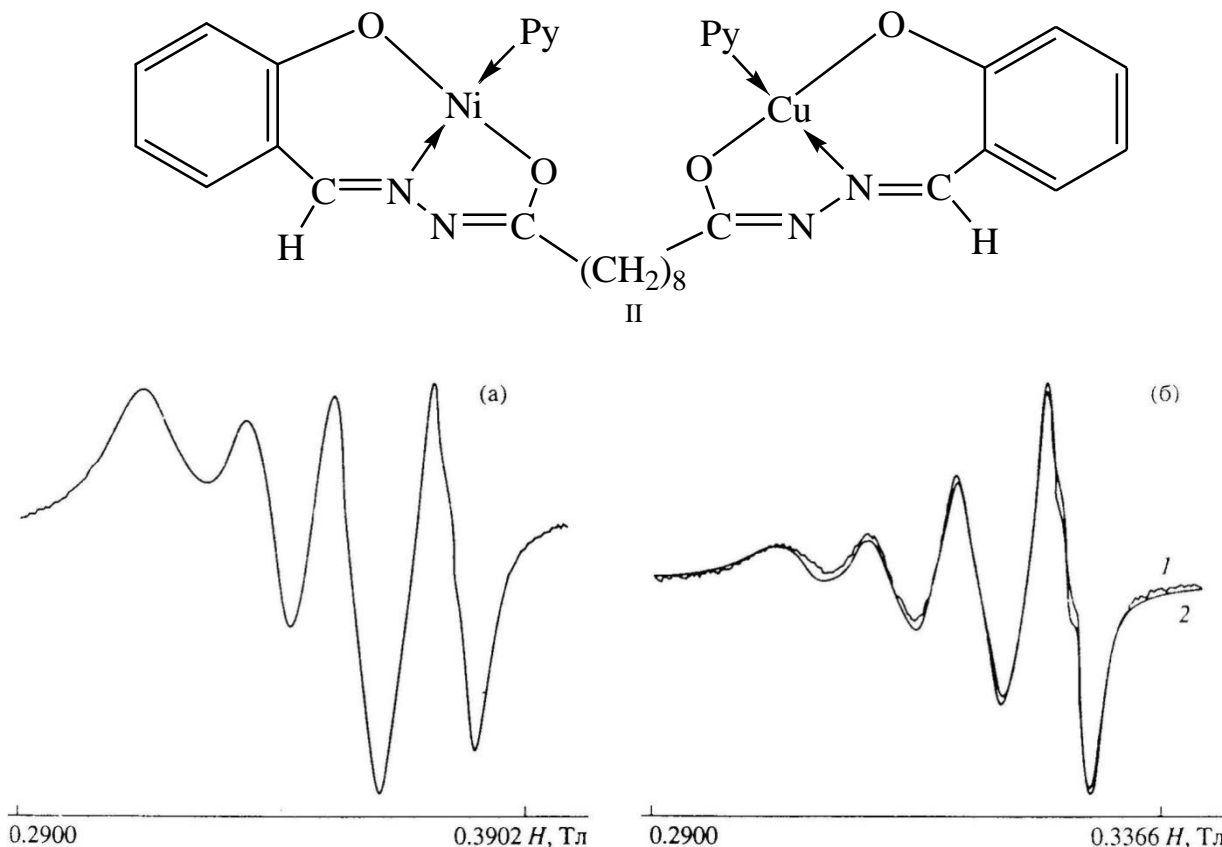


Fig. 2. ESR spectra of a solution of a homobinuclear complex of copper (II) $Cu_2L^6 \cdot 2Py$ [Cu(II)–Cu(II)] – (a) and the heterobinuclear complex $NiCuL^6 \cdot 2Py$ [Ni(II)–Cu(II)] – (b) (1 - experimental spectrum, 2 - theoretical plotted spectrum) in toluene + chloroform.

Apparently, the Breit-Rabi equation in the $Cu_2L^6 \cdot 2Py$ compound is poorly performed due to the fact that even with a polymethylene bridge of eight carbon atoms, there still exist, although very weak, exchange interactions between the unpaired electrons of two copper atoms in the homobinuclear complex, which are manifested in the convergence of the second and third components of the HFS from the copper atom and poor resolution of the HFS from nitrogen atoms. As is known, only σ bonds are realized in the polymethylene chain and π bonds are completely absent. Thus, the studied homobi-nuclear copper compounds $Cu_2L^2 \cdot 2Py$ – $Cu_2L^6 \cdot 2Py$ are characterized by the presence of exchange interactions, which are realized via σ -bonds of the polymethylene bridge. The question of the mechanism for the transfer of antiferromagnetic-type exchange interactions between two unpaired electrons along a chain of σ bonds over such large distances remains open.

IV. CONCLUSION

The value of the exchange integral over the σ -bond chain $-(CH_2)_n-$, where $n = 1-8$, in binuclear copper(II) complexes is estimated. When $n = 1-4$, the value of the exchange integral is $1-3 \text{ cm}^{-1}$.

It was experimentally established that when the polymethylene chain is extended to $n = 8$, the value of the exchange integral decreases by two orders of magnitude.

It is shown that for an adequate description of the width and shape of the ESR spectrum during elongation of the polymethylene chain, it is necessary to take into account the contribution to the line width due to the intramolecular motion in the binuclear complex around the polymethylene bridge.



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