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ESR INVESTIGATION OF COPPER(I) COMPLEXES WITH *o*-SEMIQUINOLATE LIGANDS

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Summary

Solutions of *o*-semiquinolatecopper(I) complexes with neutral ligands, such as PPh_3, $P(OEt)_3$, $AsPh_3$, $AsEt_3$, cyclooctadiene-1,5, PhC≡CPh, PhC≡CH and CO, were investigated by ESR. The spectra of the complexes are characterised by high specificity and sensitivity to configuration of ligand surroundings. The radical character of the *o*-semiquinolate ligand remains constant over the chemical transformation in the metal coordination sphere. These properties allow the use of *o*-semiquinolate ligands as a special "spin mark" in the chemistry of coordination compounds.

Introduction

The ESR method is widely applied for research on the structure and chemistry of copper(II) complexes. The chemistry of copper(I) coordination compounds is also extensive, various copper(I) derivatives being diamagnetic unlike the paramagnetic copper(II) species.

Recently we were successful in obtaining paramagnetic bis(triphenylphosphine)copper(I) complexes with *o*-semiquinolate ligands (SQ) [1]. The unpaired electron spin density in complexes of this kind is localized mainly upon SQ. As a result of the odd electron molecular orbital (MO) interaction with the orbitals of the central atom and phosphinic ligands the spin density appears upon the ^{63}Cu, ^{65}Cu and ^{31}P nuclei, which is sufficient for hyperfine coupling registration by ESR.

In this work some attempts were undertaken to extend the series of paramagnetic copper(I) complexes. For this purpose complexes of the type $L_nCu(SQ)$ with some neutral *n*- and π-ligands (L), which are typical of the chemistry of coordination compounds, have been investigated by ESR. The

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* Dedicated to Professor Ernst Otto Fischer on the occasion of his 60th birthday on November 10, 1978.

TABLE 1

ISOTROPIC ESR SPECTRAL PARAMETERS OF COPPER(I) *o*-SEMIQUINOLATE COMPLEXES L_nCu(SQ) IN THF

L_n	SQ_I				SQ_{II}				SQ_{III}		
	g_i [b]	A(Cu) [a]	A(L) [a]	A(H(SQ)) [a]	g_i [b]	A(Cu) [a]	A(L) [a]	A(H(SQ)) [a]	g_i [b]	A(Cu) [a]	A(L) [a]
$(Ph_3P)_2$	2.0046	10.8	16.9(2 P)	3.4	2.0041	11.0/11.8 [c]	17.6(2 P)	3.1	2.0054	10.2/10.9	15.7(2 P)
Ph_3P	2.0036	3.5	—	3.5	2.0037	3.4	—	3.4			
$[(EtO)_3P]_2$	2.0046	10.8	21.6(2 P)	3.3	2.0044	11.3/12.1	22.7(2 P)	3.2	2.0052	9.9/10.5	20.9(2 P)
$(EtO)_3P$ Ph_3P }									2.0052	9.8	19.5(1 P) 14.8(1 P)
$(Ph_3As)_2$	2.0048	10.3	16.9(2 As)	3.3	2.0048	11.0	18.5(2 As)	3.3	2.0056	10.0	15.1(2 As)
$(Et_3As)_2$	2.0050	10.9	18.1(2 As)	3.2	2.0049	11.6	19.1(2 As)	3.3			
Ph_3P Ph_3As }	2.0047	10.1	17.2(1 P) 17.2(1 As)	3.3							
COD-1.5	2.0035	7.0	—	3.4	2.0037	6.8/7.3	—	3.5			
PhC≡CPh	2.0033	6.3	—	3.6	2.0030	6.5	—	3.3			
PhC≡CH	2.0032	6.4	—	3.6							
CO	2.0032	6.1	—	3.5	2.0034	5.4	—	3.5			

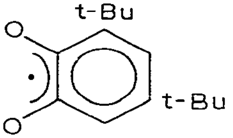
[a] In gauss; error in A(Cu), A(L), and A(H(SQ)) ± 0.2 G. [b] Error in g_i ± 0.0005. [c] In numerator $A(^{63}Cu)$, in denominator $A(^{65}Cu)$.

following ligands were used: PPh_3, $P(OEt)_3$, $AsPh_3$, $AsEt_3$, cyclooctadiene-1,5 (COD), PhC≡CPh, PhC≡CH, CO. All complexes studied were obtained in solution by the exchange reaction of CuCl with the corresponding sodium or thallium *o*-semiquinolate in presence of neutral ligands (eq. 1), where SQ is 3,5-di-t-butylbenzosemiquinolate (SQ_I) or 3,6-di-t-butylbenzosemiquinolate (SQ_{II}). Copper(I) complexes with perchloroxanthrenesemiquinolate (SQ_{III}) were synthesized by the substitution redox reaction shown in eq. 2.

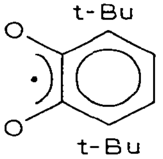
$$CuCl + Na(Tl)SQ + n\,L \xrightarrow{THF} L_nCuSQ + Na(Tl)Cl \qquad (1)$$

$$L_nCuSQ + Q_{III} \xrightarrow{THF} L_nCuSQ_{III} + Q \qquad (2)$$

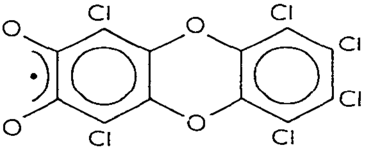
It should be noted that in reaction 1 ESR spectra in absence of neutral ligands are not observed. Isotropic ESR spectral parameters of the complexes L_nCuSQ are shown in Table 1.



(SQ_I)



(SQ_{II})



(SQ_{III})

Analysis of these data shows the observed ESR spectra to be safely identified due to their highly specific features. The variation of the neutral ligands leads to a considerable change of the hyperfine coupling constant *A*(Cu), and the ESR spectra get more specific if the neutral ligands contain magnetic nuclei. Moreover, ligands having the same magnetic nuclei, e.g. PPh_3 and $P(OEt)_3$, $AsPh_3$ and $AsEt_3$, possess different hyperfine coupling constants $A(^{31}P)$ and $A(^{25}As)$ respectively from dependence on ligand organic substituents. Change of SQ facilitates the ESR spectral analysis in some particularly difficult cases, as each semiquinolate ligand has its own specific contribution to the general pattern of hyperfine structure (HFS).

Semiquinolate copper(I) complexes with Ph_3P and $P(OEt)_3$

Recently we have published some results concerning bis(triphenylphosphine)copper(I) complexes [1]. The complex $(Ph_3P)_2CuSQ_I$ was prepared in pure state and characterised by elemental analysis. The ESR spectrum of the complex reflects the interaction of the unpaired electron with a copper nucleus, two equivalent ^{31}P nuclei and a proton of SQ_I.

The ESR spectra of the $(Ph_3P)_2CuSQ_{II}$ and $(Ph_3P)_2CuSQ_{III}$ complexes are analogous, but are solved as a result of decrease in individual line width components corresponding to two different copper isotopes ^{63}Cu and ^{65}Cu. Besides, the ESR spectrum of the $(Ph_3P)_2CuSQ_{II}$ complex shows hyperfine coupling with the two equivalent protons of SQ_{II} (see Fig. 1).

The additional ESR spectrum together with the ESR spectrum of the $(Ph_3P)_2CuSQ_{II}$ complex was registered in reaction 1. When the molar ratio $Ph_3P/CuCl = 2/1$, the HFS of this unusual spectrum corresponds to interaction of the unpaired electron with the copper nucleus and protons, one for SQ_I and

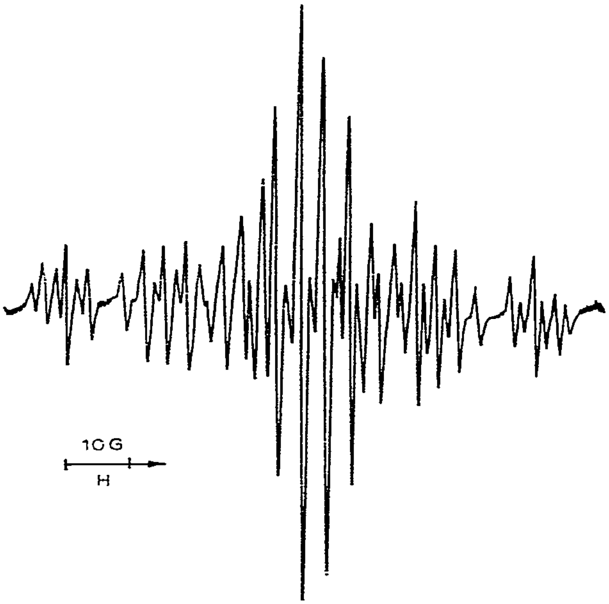


Fig. 1. Superposition of ESR spectra of complexes $(Ph_3P)CuSQ_{II}$ and $(Ph_3P)_2CuSQ_{II}$.

two for SQ_{II}, A(H) and A(Cu) being equal. Hyperfine interaction of the odd electron with the ^{31}P nucleus was not observed. The same ESR spectrum is obtained when the complex $(Ph_3P)_2CuSQ$ is used instead of Ph_3P in reaction 1. On the other hand increasing the molar ratio $Ph_3P/CuCl$ to 2/1 leads to a disappearance of this ESR spectrum and increasing intensivity of the ESR spectrum of the bis-phosphine complexes. These results show that the second ESR spectrum belongs to the monophosphinecopper(I) complex, $(PPh_3)CuSQ$.

Triethylphosphite and triphenylphosphite can be used to form analogous semiquinolate paramagnetic copper(I) complexes. The ESR spectra HFS of the $(EtO)_3P_2CuSQ$ complexes and of the phosphine complexes reflect the coupling of the unpaired electron with the semiquinolate protons, the Cu nucleus and two equivalent ^{31}P nuclei. ESR spectral parameters of monophosphite complexes are the same as for analogous monophosphine derivatives.

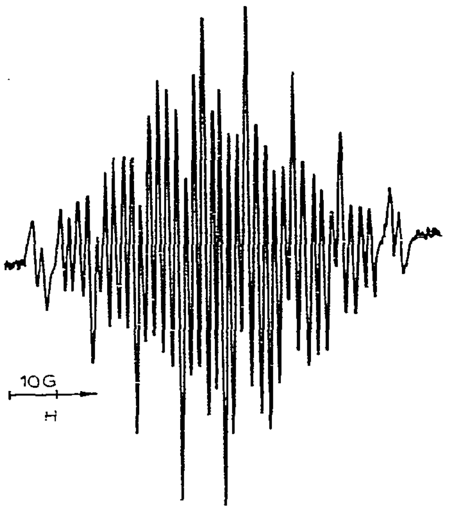
Treatment of the bis-phosphine complexes with triethylphosphite leads to displacement of the phosphine ligands and ultimately to the bis-phosphite complexes. The substitution proceeds stepwise with intermediate formation of mixed phosphinephosphitecopper(I) semiquinolate complexes, which were detected by ESR in the SQ_{III} case.

$$(PPh_3)_2CuSQ + P(OEt)_3 \rightarrow [(EtO)_3P](PPh_3)CuSQ + PPh_3 \qquad (3)$$

$$[(EtO)_3P](PPh_3)CuSQ + P(OEt)_3 \rightarrow [(EtO)_3P]_2CuSQ + PPh_3 \qquad (4)$$

Copper(I) *o*-semiquinolate complexes of $AsPPh_3$ and $AsEt_3$

The use of $AsPPh_3$ or $AsEt_3$ (2 mol to 1 mol CuCl) in reaction 1 leads to the formation of bis(arsine)copper(I) complexes, $(R_3As)_2CuSQ$. Hyperfine coupling



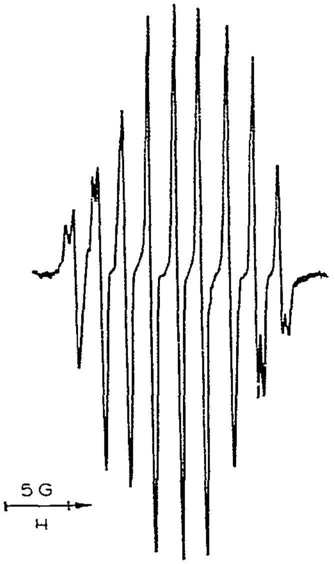


Fig. 2. ESR spectrum of complex $(Et_3As)_2SQ_I$.

Fig. 3. ESR spectrum of complex (COD-1,5)$CuSQ_{II}$.

of the unpaired electron with nuclei of H, ^{63}Cu, ^{65}Cu, ^{75}As observed in the ESR spectra confirms the composition of these complexes. Decreasing the molar ratio arsine/CuCl gives rise to the observance of monoarsine complexes (Et_3As)-CuSQ by ESR in the case of $AsEt_3$ (Fig. 2).

Substitution of arsine ligands takes place when PPh_3 or $P(OEt_3)_3$ is treated with a bis(arsine)copper(I) complex. The substitution proceeds stepwise. The intermediate arsinephosphine and arsinephosphite complexes are easily identified by ESR since their ESR spectra differ strongly from the ESR spectra of the initial bis(arsine) complexes because of the difference in spins of the nuclei ^{31}P (I 1/2) and ^{75}As (I 3/2).

Copper(I) *o*-semiquinolate complexes of cyclooctadiene-1,5

The ESR spectra (Fig. 3) of *o*-semiquinolate—COD complexes contain only components due to the coupling of the unpaired electron with the ^{63}Cu and ^{65}Cu nuclei and the protons of SQ. Hyperfine coupling with the neutral ligand nucleus does not directly appear in the ESR spectrum. The mere fact of ESR signal appearance indicates (COD)CuSQ complex formation, no ESR signals being observed in absence of the ligand. In this complex COD acts as a bidentate ligand and its binding with the copper atom possesses chelate character. This is the usual kind of binding for COD and realized for example in known copper(I) complexes [2].

It should be noted that the copper—COD bond is less stable than the copper—phosphine or —phosphite bonds. The latter two ligands free COD from copper(I) semiquinolate complexes. It is impossible to observe mixed complexes containing both COD and one n-ligand in this case, but ESR spectra of intermediate mono-ligand complexes are observed.

$$\text{(COD)CuSQ} + \text{L} \rightarrow \text{LCuSQ} + \text{COD} \quad (5)$$

$$\text{LCuSQ} + \text{L} \rightarrow \text{L}_2\text{CuSQ} \quad (6)$$

Copper(I) semiquinolate complexes with PhC≡CPh and PhC≡CH

The use of diphenylacetylene in reaction 1 leads to the formation of paramagnetic complexes of copper(I), whose ESR spectral parameters are similar to those of the COD complexes (see Table 1) and differ remarkably from those of mono-ligand semiquinolate complexes. This proves in our opinion the similar structure of the complexes. Hence the alkyne and cyclooctadiene complexes are four-coordinated, the copper atom being bonded to two π-ligands, $(\text{PhC}{\equiv}\text{CPh})_2\text{CuSQ}$.

Phenylacetylene also forms analogous copper(I) complexes, but their lifetime is substantially reduced. The lability of complexes of PhC≡CH is controlled evidently by their rapid transformation in the σ-derivative CuC≡CPh.

Copper(I) semiquinolate complexes with carbon monoxide

Introduction of CO into reaction 1 leads to the appearence of the ESR spectra of copper(I) semiquinolate complexes, their intensity strongly depending on the CO pressure. With increasing pressure the ESR signal intensity increases, and CO removal from the reaction system leads to signal decrease and disappearence. According to ESR data the stability of the carbonyl complexes under constant pressure depends on the nature of the solvent, increasing with their solvating ability. All solvents used may be placed in the following approximate order according to their stabilizing effect: hexane < benzene < diglyme < dimethylaniline ≈ THF < DMSO < triethylamine. It is significant that the hyperfine coupling constant A(Cu) of the copper(I) carbonyl complexes increases in this order (see Table 2). In hexane, which is incapable of coordination the values of the ESR spectrum parameters of the carbonyl complex are very similar to those of the mono-ligand semiquinolate complexes of copper(I). Therefore we suppose the copper(I) carbonyl complexes to be tri-coordinated mono-ligand derivatives, (CO)CuSQ, in absence of solvation.

TABLE 2

EFFECT OF SOLVENT ON THE ESR SPECTRAL PARAMETERS OF COMPLEXES $(\text{CO})\text{Cu}(\text{SQ}_\text{I})$

Solvent	g_i [a]	A(Cu) [b]	A(H(SQ)) [b]
Hexane	2.0026	3.7	3.7
Toluene	2.0033	4.8	3.6
Diglyme	2.0028	5.3	3.6
THF	2.0032	6.0	3.4
Dimethylaniline	2.0034	6.2	3.6
DMSO	2.0038	7.1	3.2
Triethylamine	2.0040	9.5	3.1

[a] Error in g_i ± 0.0005. [b] In G; error in A(Cu) and A(H(SQ)) ± 0.2 G.

Hyperfine coupling in copper(I) semiquinolate complexes. Relation between hyperfine coupling and complex geometry

The unpaired electron mentioned above was localized essentially on the semiquinolate ligand. The fact that in the ESR spectra of the copper(I) semiquinolate complexes the hyperfine coupling constants $A(\mathrm{H(SQ)})$ and g_i is close to the corresponding parameters of "free" semiquinone anion radicals points to such electron localisation.

Spin density transfer from SQ to the central atom and neutral ligands may occur by two possible ways: firstly as a result of direct interaction of the MO occupied by the unpaired electron with orbitals of corresponding energy and symmetry of the Cu atom and ligands and secondly at the expense of spin polarization of complex σ-orbitals.

The comparison between ESR hyperfine coupling constants $A(\mathrm{Cu})$ and $A(\mathrm{L})$ of the complexes studied and their geometry shows that both mechanisms operate in this case. The relative contribution of each of them is determined primarily by the coordination number and the complex geometry which is related to it.

Coordination number 4 is obvious for the complexes $\mathrm{SQCuL_2}$ and their geometry must be very similar to the geometry of analogous bis(triphenylphosphine)copper(I) complexes with β-diketonate, carboxylate and other ligands where the distorted tetrahedral structure has been proved by X-ray structural analysis [3]. The planar trigonal structure characteristic of tri-coordinated copper(I) complexes is the most probable for mono-ligand derivatives with coordination number 3 [4].

In mono-ligand complexes the hyperfine coupling constant $A(\mathrm{Cu})$ is minimal (3.4—3.6 G) and does not depend on the nature of the ligands SQ and L. Hyperfine coupling with magnetic nuclei is not observed. In the ESR spectra of bis-ligand complexes the value $A(\mathrm{Cu})$ is greater than in mono-ligand complexes and strongly depends on the nature of SQ and the neutral ligand. Also a linear relationship between $A(\mathrm{Cu})$ and $A(\mathrm{P})$ is found for the bis-ligand complexes (eq. 7),

$$A(\mathrm{Cu}) = B + KA(\mathrm{P}) \tag{7}$$

where $B = 3.4$ G, $K = 0.44$ for $\mathrm{L = PPh_3}$ and $\mathrm{B} = 3.5$ G, $K = 0.39$ for $\mathrm{L = P(OEt)_3}$.

It is symptomatic, that the constant K in eq. 7 does not depend on the nature of the neutral ligand and is equal (within the experimental error) to the $A(\mathrm{Cu})$ constant in mono-ligand copper(I) semiquinolate complexes. In our opinion equality of both values permits regarding B as the contribution of spin density to $A(\mathrm{Cu})$, which arises from the 4s orbitals of Cu by the spin polarization mechanism. Really, in planar trigonal mono-ligand complexes no MO is suitable by symmetry for overlapping with the MO of the unpaired electron (see Fig. 4). The neutral ligand is situated in the nodal plane of these orbitals. The interaction with the Cu $3d_{xz}$ and $4p_x$ orbitals, which have the same symmetry, is not sufficient to account for the large difference in energies, i.e. spin polarization of the Cu—O bond is the only possible mechanism of spin density transfer to the Cu 4s orbital.

In bis-ligand complexes with distorted tetrahedral configuration a new

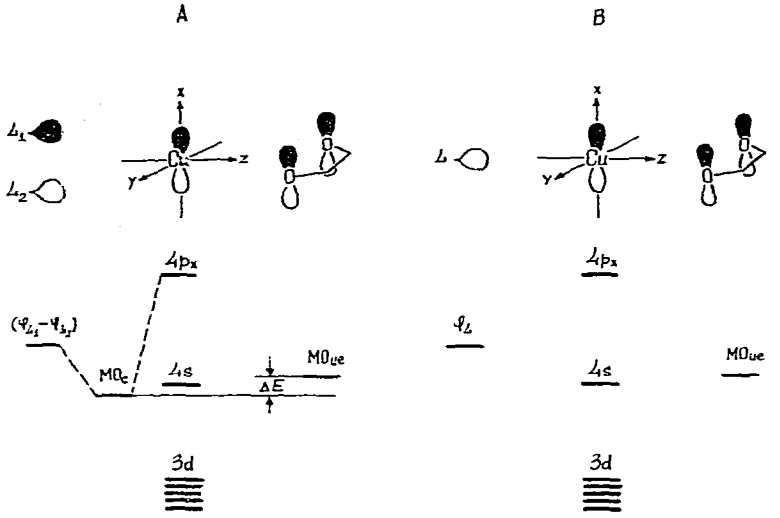


Fig. 4. Probable scheme of molecular orbitals of copper(I) semiquinolate complexes with two (A) and one (B) neutral ligands; orbital ionization potentials are taken for Cu from [5], for L = $P(OEt)_3$ from [6], for SQ = *o*-phenanthrensemiquinone from [7].

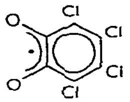
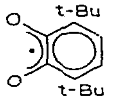
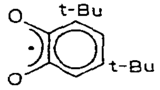
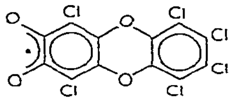
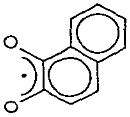
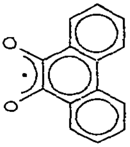
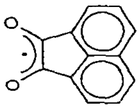
orbital appears, the group orbital of ligands ($\varphi(L_1)-\varphi(L_2)$) being of the same symmetry as the symmetry of the MO containing the unpaired electron (see Fig. 4). Its interaction with the Cu $4p_x$ orbital leads to the formation of an MO having energy much nearer to the unpaired electron energy. As a result the opportunity appears of additional spin density transfer to the Cu and P nuclei caused by delocalisation of the unpaired electron. Thus the variable part in eq. 7 proportional to $A(P)$ is equal (within the limits of the examined model) to the contribution of the one-electron delocalisation mechanism in $A(Cu)$. It should be noted, that the character of the complex MO must be closer to the ligand orbitals than to the $4p_x$ one. Hence the electron transfer takes place more probably to the ligands then to the copper atom.

For series of semiquinolate complexes with the same neutral ligands the value of the delocalizing contribution following the suggested model must be controlled mainly by the value of the energy difference of MO complex and MO unpaired electron (ΔE see Fig. 4). The lower the energy of the unpaired electron MO, the lesser ΔE and the greater the delocalizing contribution value. From the oxidising ability of *o*-quinones the MO of the chloranil derivative of the semiquinolatecopper(I) complex must have the lowest energy, and the MO of the acenaphthosemiquinolate complex the highest. The observed decrease of the $A(Cu)$ value when passing from the *o*-chloranil complex to the acenaphthoquinonic ones (see Table 3) is in good agreement with the order of energy change of the unpaired electron MO. The latter confirms the correctness of the suggested model.

There is good agreement between the model of hyperfine coupling used and the g_i variation in the semiquinolate copper(I) complexes. The g_i value of the mono-ligand complexes, which do not have a fully occupied MO of energy close to the unpaired electron MO, is equal to that of the free semiquinonic

TABLE 3

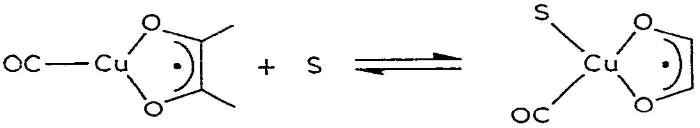
ESR SPECTRAL PARAMETERS OF COPPER(I) o-SEMIQUINOLATE COMPLEXES WITH TRIPHENYLPHOSPHINE AND TRIETHYLPHOSPHITE LIGANDS

SQ	PPH_3		$P(OEt)_3$	
	A(Cu) [a]	*A*(P) [a]	*A*(Cu) [a]	*A*(P) [a]
	11.8	19.4		
	11.0	17.8	11.3	22.7
	10.8	16.3	10.8	21.6
	10.2	15.7	9.8	20.1
	9.5	13.3		
	8.9	11.9	9.2	17.0
	6.7	7.7	6.9	9.8

[a] In gauss; error in *A*(Cu) and *A*(P) ± 0.2 G.

ligand. On the contrary in bis-ligand complexes the observed g_i increase indicates directly the presence of a fully occupied MO close in energy to the unpaired electron MO.

The suggested hyperfine coupling mechanism explains the observed *A*(Cu) dependence on the solvating ability of the solvent in carbonylcopper(I) semiquinolate complexes. The complex solvation causes the transformation of the planar trigonal structure to a distorted tetrahedron with simultaneous reconstruction of the electronic structure and formation, in particular, of the group orbital (φ(S)—φ(CO)) (eq. 8).



(8)

The unpaired electron delocalization on this MO causes the A(Cu) increase. The increase of solvent solvating ability leads either to increase both of the quantity of solvated particles and A(Cu) or to more severe distortion of the complex geometry. The latter leads to increase of the hyperfine coupling constants, A(Cu).

The semiquinolate ligand used as "spin mark" in copper(I) complexes

The results we have obtained in this work allow us to draw a conclusion about the possible use of the semiquinolate ligand as a "spin mark" in copper(I) complexes. This conclusion is based on the following properties of copper(I) semiquinolate complexes. (1) Semiquinolate complexes can be obtained for a rather wide range of copper(I) coordination compounds with neutral n- and π-ligands; (2) these complexes are easily identified by ESR owing to the special features of their ESR spectra; (3) in the ESR spectra of copper(I) semiquinolate complexes the hyperfine coupling value is sensitive to their molecular geometry; (4) paramagnetic SQ bonding to the copper atom retains its radical character in chemical transformation in the metal coordination sphere. The change of ESR spectrum character makes it possible to observe the changes in the coordination sphere.

On the one hand the semiquinolate ligand used as "spin mark" can help in deciding stereochemical questions for example for following the molecular geometry change in solvation processes and reversible addition of neutral ligands to the copper atom. On the other hand the presence of a paramagnetic ligand in the molecule permits the use of ESR for determination of complex concentration during the reaction and hence measurement of kinetic and thermodynamic parameters of such processes, in which this complex takes part.

Evidently the concept of semiquinolate use as a "spin mark" can be extended to other transition metals. Moreover, more several semiquinolate complexes have been prepared for other transition metals, Pd [8,9], Ag [1], Pt [9,10], Ru [9,10], Rh,Ir [10], Co [11].

Experimental

The starting quinones: 3,5-di-t-butylbenzoquinone-1,2, 3,6-di-t-butylbenzoquinone-1,2, perchloroxanthrenequinone-2,3 were prepared by known procedures [12], [13] and [14] respectively. The other reagents used were obtained commercially and, purified by standard techniques.

The solutions of sodium [15] and thallium [16] *o*-semiquinolates were prepared according to the literature.

Bis(triphenylphosphine)- and bis(triethylphosphite)-copper(I) semiquinolate derivatives of *o*-chloranil, phenanthrenquinone-9,10, naphthoquinone-1,2 and acenaphthoquinone-1,2 were obtained according to [1].

The samples for ESR spectra registration were prepared by mixing stoichiometric amounts of deoxygenated solutions in vacuum ampules suitable for ESR spectral measurements.

The ESR spectra were registrated in X-diaposon with a radio spectrometer PE—1301. Mn^{+2} ions in an MgO crystal grating were used as standard for magnetic field calibration.

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