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MAGNETIC AND SPECTRAL STUDIES ON CHROMIUM(III), NICKEL(II) AND COPPER(II) COMPLEXES OF 4-HYDROXI-5-METHOXY ISOPHTHALDEHYDE BIS DIMETHYLHYDRAZONE

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ABSTRACT

A new hydrazone Schiff base, 4-hydroxy-5-methoxy isophthaldehyde bis dimethylhydrazone and its chromium(III), nickel(II) and copper(II) complexes have been synthesized and characterized by elemental analysis, molar conductance and magnetic moment determinations, infrared(IR), electronic and Electronic Paramagnetic Resonance(EPR) spectral studies. The complexes have distorted octahedral symmetry. Ligand field parameters have been calculated for Cr(III) and Ni(II) complexes on the basis of their electronic transitions, using the weak field approach and the strong field approach.

KEYWORDS: hydrazones; chromium(III), nickel(II), copper(II) complexes

RESUMO

Foi preparada a nova base hidrazona, 4-hidroxi-5-metoxi isoftaldeião bis dimetilhidrazona e seus complexos com cromo (III), níquel(II) e cobre (II). Os novos compostos foram caracterizados através de análise elemental, medidas de condutância molar e momento magnético e técnicas de espectroscopia no infravermenlho, ultravioleta-visível e ressonância paramagnética eletrônica (RPE). Os complexos possuem simetria octaédrica distorcida. Parâmetros para o campo ligante foram calculados na base das transições eletrônicas usando os métodos de campo fraco e campo forte.

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INTRODUCTION

Due to their activity, especially as potent tuberculostatic agents, the hydrazone Schiff bases and their metal complexes have been intensively investigated during the last few years[1-8].

In continuation of our studies of the metal complexes of Schiff bases derived from 5-formylvanyline [9-12], we have synthesized a new hydrazone Schiff base, 4-hydroxy-5-methoxy isophthaldehyde bis dimethylhydrazone, and its Cr(III), Ni(II) and Cu(II) complexes.

The hydrazone Schiff base shows a bidentate ON monobasic character in all the complexes.

The splitting of "octahedral" bands of the electronic spectra of chromium(III) and nickel(II) complex compounds permitted the determination of ligand field parameters for these complexes.

EXPERIMENTAL

Synthesis of the hydrazone:

To a solution of 5-formylvanyline in ethanol (3,6g/25 ml ethanol) was added 4 ml dimethylhydrazine dissolved in 30 ml ethanol. The resulting mixture was heated under reflux for 1 hr and concentred under vacuum. To the residue was added 3-4 ml petroleum ether. The solid product that resulted was recrystallised from diethyl ether and precipited with petroleum ether, m.p.=87°C. Its formula was confirmed by elemental analysis (exp. C: 58,97, H: 7,62, N: 21,03; calc. C:59,09, H:7,57, N: 21,21%) and IR spectrum (Table 2).

Preparation and analysis of metal complexes

All the metal chelates were prepared by the following general method: a solution of the ligand (0,001 mol) in ethanol was added to a solution of metal salt (0,0005 mol)-$CrCl_3 \cdot 6H_2O$, $NiCl_2 \cdot 6H_2O$ and $Cu(CH_3COO)_2 \cdot 2H_2O$, respectively, in water. The resulting solution was heated under reflux for 2hr. After cooling at room temperature, the resulting precipitate was filtered, washed successively with ethanol and ether and dried in air.

Carbon, hydrogen and nitrogen were determined by micro-analysis. Metals were analysed employing standard procedures[13], after destroying the organic part with sulfuric acid and 30%H_2O_2. The analytical data are given in Table 1.

Physical measurements.

Molar conductance of the complexes in dimethylformamide was measured with a Phillips PR 9500 conductivity meter. Magnetic susceptibility measurements were carried out at room temperature, by the Faraday method, using $Hg[Co(SCN)_4]$ as calibrant.

IR spectra (in KBr pellets) were recorded on a Spekord M-80 Carl Zeiss Jena spectrophotometer, in the range 4000-400 cm^{-1}. Diffuse reflectance spectra were recorded on Spekord M-40 spectrophotometer, in the range 200-900 nm. The near infrared spectra were registered on Nirs Sisten Pharma-5100 (USA) spectrophotometer, with monofascicle, in reflection.

Thermogravimetric analysis was carried out with a MOM Q-1500 derivatograph, in static air atmosphere, at a heating rate of 10°C/min, from room temperature to 1000°C.

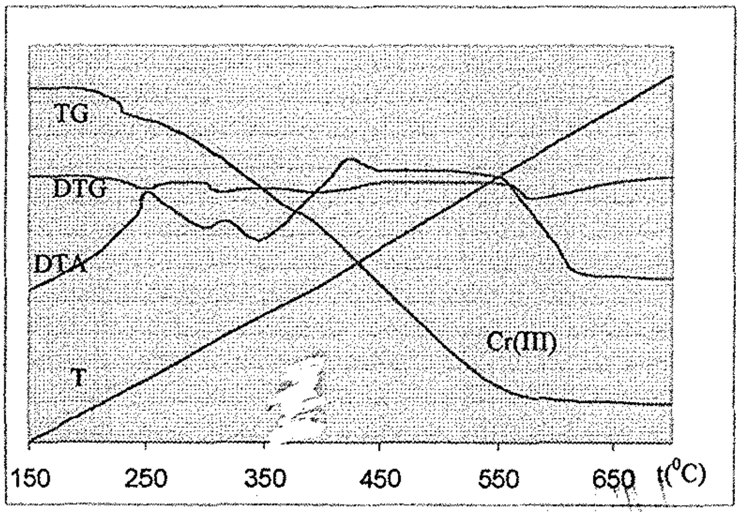
RESULTS AND DISCUSSION

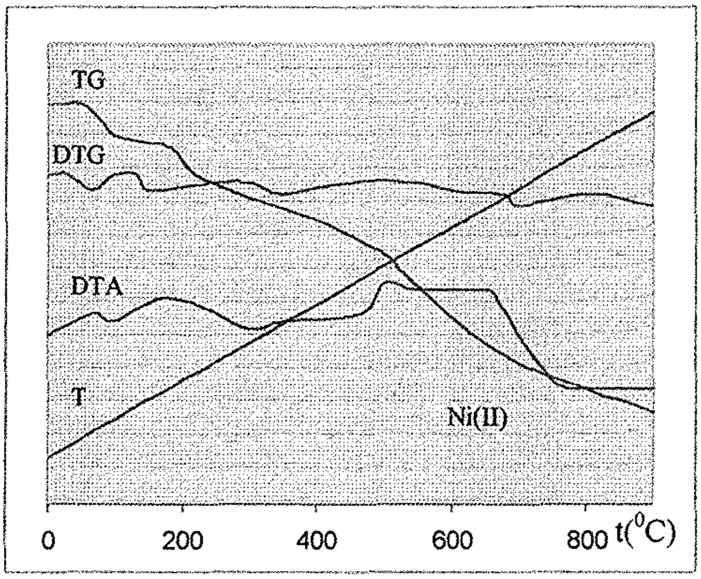
The analytical data (Table 1) show 1:2 metal:ligand composition for the complexes. Molar conductance values of the complexes in DMF show that the Ni(II) and Cu(II) complexes are non-electrolytes, while the Cr(III) complex is 1:1: electrolyte (Table 1).

Table 1. Analytical Data and Molar Conductibility for the Complexes Studied

Complex	C% Calc/exp	N% Calc/exp	Metal% Calc/exp	λ_M(in DMF) $\Omega^{-1}cm^2mol^{-1}$
$[CrL_2(H_2O)_2]Cl$ (I)	48,07 47,90	17,20 16,87	8,47 8,02	70
$[NiL_2(H_2O)_2]\cdot 3H_2O$ (II)	46,22 45,86	16,59 16,30	8,74 8,04	27
$[CuL_2(H_2O)_2]$ (III)	49,84 49,31	17,89 17,13	10,22 9,93	30

The presence of coordinated water is supported by TG and IR spectral data. Thus, the thermogravimetric curves for all the three complexes show an exothermic peak in the range of 180-240°C, which corresponds to the loss of coordinated water[14]. For the Ni(II) complex, the first exothermic process, with a maximum at 120°C, corresponds to the loss of crystalline water. The exothermic process, with a maximum at 290°C, observed on the thermal diagram of the Cr(III) complex, is due to the loss of anionic chlorine. All the complexes lose the organic ligand in a large exothermic process, in the range of 300-800°C. Figure 1. gives the characteristic thermal diagrams of the complex compounds.





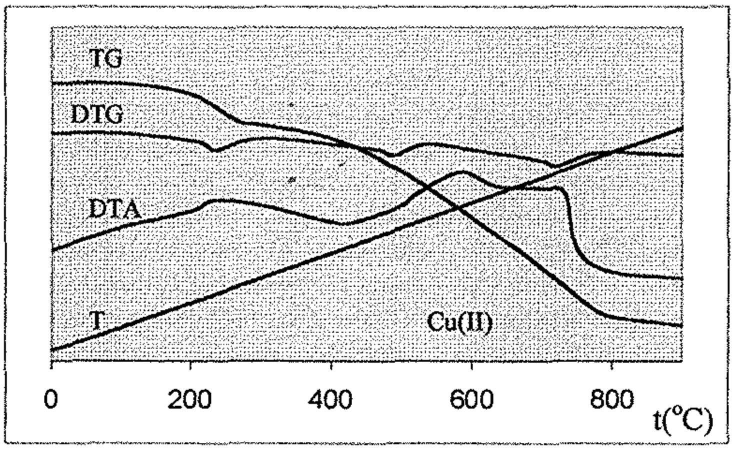


Figure 1. Thermogravimetric curves of the hydrazone complexes

Infrared spectra. IR spectrum of the ligand shows a broad band at 2890 cm^{-1}, due to the OH stretching frequency (Table 2). The metal complexes do not absorb in this region, but show a broad medium band at 3420-3460 cm^{-1}, that may be due to ν_{OH} of coordinated water[1,2,7,15]. The new bands at 770-780 cm^{-1} and 900 cm^{-1} appearing in IR spectra of the complexes are attributed to ρ_w(OH) and ρ_r(OH) respectively, of coordinated water[16].

Table 2. Characteristic Bands in the IR Spectra of the Ligand and its Complexes (ν_{max}, cm^{-1})

Assignments	HL	I	II	III
$\nu_{C=N}$	1566 s	1590 s (C=N coord.) 1610 s (C=N noncoord.)	1590 s 1620 s	1590 s 1600 s
$\nu_{C=N}+\nu_{C=C}$ conj.	1566 s	1560 s	1550 s	1550 s
ν_{C-O} phen.	1280 s	1295 s	1295 s	1290 s
ν_{NN}	965 m	980 m	975 m	980 m
ν_{OH}	2952 m (OH chelated)	3420 ms (H_2O)	3450 s	3460 s
$\rho_w(H_2O)$	-	780 w	770 w	770 w
$\rho_r(H_2O)$	-	900 w	910 w	910 w
ν_{M-O}	-	660 (H_2O) 540 w (O phen.)	590 w 570 w	580 w 565 w
ν_{M-N}	-	485 w	450 w	460 w

The strong band which occurs at 1566 cm^{-1} is assigned to the stretching vibration of >C=N group, $\nu_{C=N}$[1-4,6-8]. In the IR spectra of the complexes, this band splits in two components, due to the difference in environment of the azomethine groups: one is coordinated and the other non-coordinated. The coordination of one azomethine group (the >C=N group in *ortho* to the hydroxy group) is also supported by a upward shift in the position of the band assigned to ν_{NN} (965 cm^{-1} in IR spectrum of the ligand)[2,3,17].

The strong band at 1280 cm^{-1} assignable to C-O bonding of phenolic group shifts to higher frequencies, indicating the coordination of the ligand through phenolic oxygen[4,6,14,17].

New weak bands appearing in the far-infrared region for all the complexes may be assigned to ν_{M-O} and ν_{M-N} vibrations, respectively[1,2,6,7].

The analysis of the IR spectra of 4-hydroxy-5-methoxy isophthalaldehyde bis-dimethylhydrazone and its Cr(III), Ni(II) and Cu(II) complexes shows that the ligand is coordinated to the phenolic oxygen atom and the nitrogen atom of azomethine group, in the *ortho* position to the phenolic group.

Magnetic and electronic spectral data. The hexacoordination of the metal ions in all the complexes, established by analytical, thermogravimetrical and IR spectral data, is confirmed by electronic spectra, characteristic for distorted octahedral complexes.

The μ_{eff} value of the chromium(III) complex is 3,82 MB, according to an octahedral stereochemistry[3]. The six-coordinated Cr(III) complexes having octahedral symmetry generally show three spin-allowed bands, due to $^4A_{2g}\rightarrow{}^4T_{2g}$, $^4A_{2g}\rightarrow{}^4T_{1g}$ and $^4A_{2g}\rightarrow{}^4T_{1g}(P)$ [18]. For the present complex, the observation of six spin-allowed transitions can be accounted for by a low symmetry ligand field, due to the existence of the chromophore CrO_4N_2. In such a situation, the UV-VIS absorption bands (Figure 2) can be assigned to the transitions shown in Table 3, assuming an octahedral symmetry with tetragonal distortion, by compression along z axis (direction of azomethine nitrogens, while xOy plane is limited by the oxygen atoms of water and the phenolic groups of hydrazone). The transitions with low intensity at 14084cm^{-1} and 19723 cm^{-1} are spin-forbidden transitions.

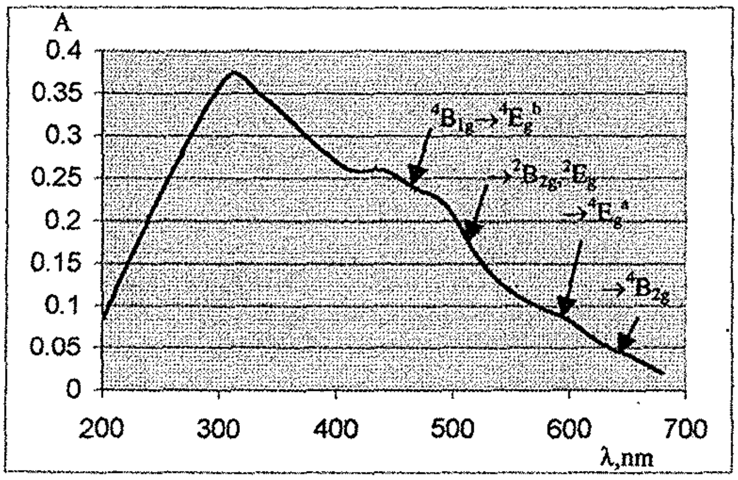


Figure 2. Diffuse-reflection electronic spectrum of the Cr(III) complex

Table 3. Electronic transitions for $[CrL_2(H_2O)_2]Cl$

Observed bands (ν_{max}, cm^{-1})	Assignments
14084	$^4B_{1g}\rightarrow{}^2A_{1g}, {}^2B_{1g}; {}^2A_{2g}, {}^2E_g$
16666	$^4B_{1g}\rightarrow{}^4B_{2g}$
18518	$^4B_{1g}\rightarrow{}^4E_g{}^a$
19723	$^4B_{1g}\rightarrow{}^2B_{2g}, {}^2E_g$
20283	$^4B_{1g}\rightarrow{}^4E_g{}^b$
21459	$^4B_{1g}\rightarrow{}^4A_{2g}{}^a$
25839	$^4B_{1g}\rightarrow{}^4E_g{}^c$
26809	$^4B_{1g}\rightarrow{}^4A_{2g}{}^b$

The splitting of the excited states for this complex permitted the calculation of ligand field parameters.

Using the weak field approach, the positions of the various transitions are expressed in terms of crystal field parameters Dq, D_t and D_s [18,19].

Because the splitting of the first "octahedral" band is equal to 35/4 Dq, we obtained D_t = -211 cm^{-1}, which corresponds to an octahedron compressed along the z axis (the axis of azomethine nitrogens). The splitting of the second "octahedral" band permitted the calculation of D_s=152 cm^{-1}.

Molecular orbital parameters $d\sigma$ and $d\pi$ and AOM parameters are calculated, using the relations proposed by Lever [18]. We thus obtained $d\sigma = 167$ cm^{-1} and $d\pi = -755$ cm^{-1}, showing that the azomethinic nitrogen is a better σ-donor but a poorer π-donor than oxygen (phenolic and oxygen of water)[20].

In the case of this complex, none of the AOM parameters can be written as zero, so we arbitrarily chose $e'_\sigma = 7459$ cm^{-1}, which represents the value determined by Lever [18] for water, in the complex $[Cren_2(H_2O)_2]^{3+}$. Applying the relations [18]:

$$Dq_L = 1/10\,[e'_\sigma(L) - 4e'_\pi(L)]$$
$$D_s = 2/7\,[e'_\sigma(L) + e'_\pi(L) - e'_\sigma(Z) - e'_\pi(Z)]$$
$$D_t = 2/35\,[3\,e'_\sigma(L) - 4e'_\pi(L) - 3e'_\sigma(Z) + 4\,e'_\pi(Z)]$$

we obtained the values of AOM parameters given in Table 4 (column 2). If we assume for $e'_\sigma(L)$ the value indicated above, we must find $e'_\pi(L) = 1370$ cm^{-1}. In fact, we found a greater value for $e'_\sigma(L)$ which indicates that the phenolic oxygen is a better π-donor than the oxygen of water.

Using the relations:

$\Delta_1 = 3D_s - 5D_t$ and $\Delta_3 = 4D_s + 5D_t$, we have also obtained the values of strong field parameters.

The separation energy between the ground state and the first excited doublet state, equal to $9B_{55} + 3C$, provides the value of B_{55}, assuming $C = 4B_{55}$. The large value obtained, $B_{55} = 670$ cm^{-1} ($\beta = 0{,}72$), indicates that the ligands form weak π bonds, so that the interelectronic repulsions into the t_{2g} level are strong. The transitions from $^4T_{1g}(F)$ to $^4T_{1g}$ (equal to $12B_{35}$) is a measure of interelectronic repulsions between the t_{2g} and e_g orbitals, so that we can calculate the value $B_{35} = 400$ cm^{-1} ($\beta = 0{,}43$). The relation $\beta_{35}^2 = \beta_{33}\cdot\beta_{55}$ gives the value of $\beta_{33} = 0{,}26$, which is in accordance with strong σ metal-ligand bonds [21].

Using a strong field approach, expressing the energy of the states in terms of Δ_1, Δ_2, Δ_3 and B parameters [18], we obtained the values given in the third column of Table 4.

Table 4. Ligand Field Parameters for $[CrL_2(H_2O)_2]Cl$ (cm^{-1})

Parameter	Weak Field Approach	Strong Field Approach
Dq_L	1666	1666
Dq_Z	2037	1797
D_t	-211	-75
D_s	152	-12
$d\sigma$	167	159
$d\pi$	-755	-170
$e'_\sigma(L)$	7459	7459
$e'_\pi(L)$	1427	1427
$e'_\sigma(Z)$	7682	7671
$e'_\pi(Z)$	672	1257
Δ_1	1511	341

Δ_3	-447	-425
B_{55}	670	670
B_{35}	400	364
B_{33}	239	195

A comparison between the two sets of data leads to the following conclusions:

-dσ is positive, according to a better σ-donor capability of azomethinic nitrogen than oxygen (phenolic and oxygen of water);

-dπ is negative, signifying that the azomethinic nitrogen is a poorer π-donor than oxygen.

-the agreement between the data obtained by the two approaches is good, except for the values of $e'_\pi(Z)$. This parameter has a greater value in the strong field approach, indicating a more important contribution of the azomethinic nitrogen to the π interactions with metal orbitals. As a consequence of the value of $e'_\pi(Z)$, the values of dπ and Δ_1 are smaller.

Expressing the distortion from the octahedral symmetry in terms of DS, DT and DQ parameters [22], we obtained :

Weak Field Approach	Strong Field Approach
DS = -1064 cm^{-1}	DS = 84 cm^{-1}
DT = -2860 cm^{-1}	DT = -1016 cm^{-1}
DQ = 49191 cm^{-1}	DQ = 47009 cm^{-1}
DT/DQ = -0,058	DT/DQ = -0,022

The value of the ratio DT/DQ corresponds to an octahedron compressed along z axis (at the limit, the value for this ratio is –0,45).

The magnetic moment for the *nickel(II)* complex is equal to 3,04 MB, in accordance with an octahedral stereochemistry [4]. This conclusion is supported by the electronic spectrum, characteristic for a tetragonally distorted nickel(II) complex [18,23-25].

A comparison with the position of the first octahedral band for the complex compounds of the type ML_6 (10800 cm^{-1} for $[Ni(NH_3)_6]^{2+}$ and 8500 cm^{-1} for $[Ni(H_2O)_6]^{2+}$), permitted the assignment of the observed bands (Figure 3) to the transitions written in Table 5. The first transition is observed in near-infrared region; the third "octahedral" band is unresolved.

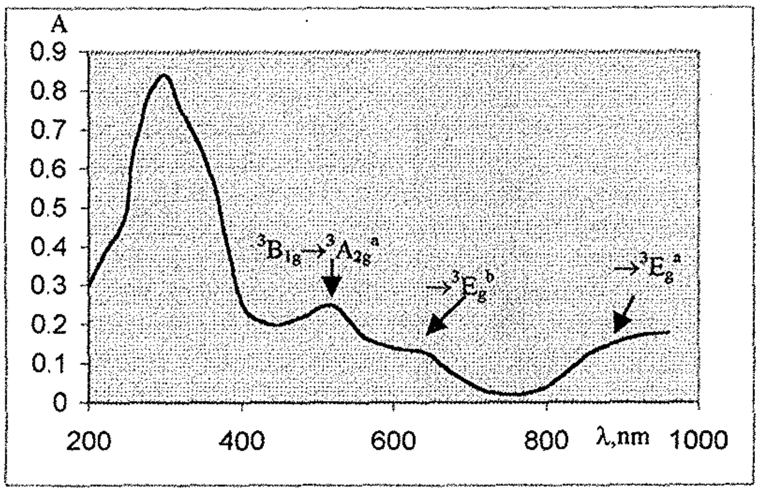


Figure 3. Diffuse-reflection electronic spectrum of the Ni(II) complex

Table 5. Electronic Transitions for $[NiL_2(H_2O)_2]\cdot 3H_2O$

Observed bands (ν_{max}, cm^{-1})	Assignments
9090	$^3B_{1g}\rightarrow{}^3B_{2g}$
10000	$^3B_{1g}\rightarrow{}^3E_g{}^a$
17021	$^3B_{1g}\rightarrow{}^3E_g{}^b$
18760	$^3B_{1g}\rightarrow{}^3A_{2g}{}^a$
22700	$^3B_{1g}\rightarrow{}^3E_g{}^c,{}^3A_{2g}{}^b$

Using the weak field approach and the strong field approach, as we showed above [18,19,22,23], we have obtained the values of the ligand field parameters, reported in Table 6.

Table 6. Ligand Field Parameters for $[NiL_2(H_2O)_2]\cdot 3H_2O$

Parameters	Weak Field Approach	Strong Field Approach
Dq_L	909	909
Dq_z	1091	1046
D_t	-104	-78
D_s	-311	-267
$d\sigma$	661	547
$d\pi$	206	206
$e'_\sigma(L)$	4118	4271
$e'_\pi(L)$	816	930
$e'_\sigma(Z)$	5000	5000
$e'_\pi(Z)$	1022	1290
Δ_1	-413	-412
Δ_3	-1764	-1459
B	835	927

Because D_t is negative, the sign of the tetragonal distortion is such that the octahedron is compressed along the z-axis. The value of DT/DQ ratio, indicated below, is also in accordance with a weak compression of the octahedral ligand field along the z-axis [22].

Weak Field Approach	Strong Field Approach
DS = 2177 cm^{-1}	DS = 1869 cm^{-1}
DT = -1410 cm^{-1}	DT = -1057 cm^{-1}
DQ = 26661 cm^{-1}	DQ = 26244 cm^{-1}
DT/DQ = -0,052	DT/DQ = -0,040

The agreement between the values of the ligand field parameters calculated with the two approaches is very good. The negative values of Δ_1 and Δ_3 are in accordance with the following sequence of *d* metal orbitals energy:

$$d_{xy} < d_{xz}, d_{yz} < d_{x^2-y^2} < d_{z^2}$$

It is interesting to observe that the π-donor power of the azomethinic nitrogen of the hydrazone ligand is greater than that of oxygen in the case of Ni(II) complex, contrary to the results obtained for Cr(III) complex.

The magnetic moment for the *copper*(II) complex is equal to 1,76 MB, corresponding to the spin-only value for the unpaired electrons [26]. The electronic spectrum (Figure 4) shows a broad band in the visible region, with a maximum at 15200 cm^{-1} and another, at 21500cm^{-1}, characteristic for distorted octahedral stereochemistry [4,27].

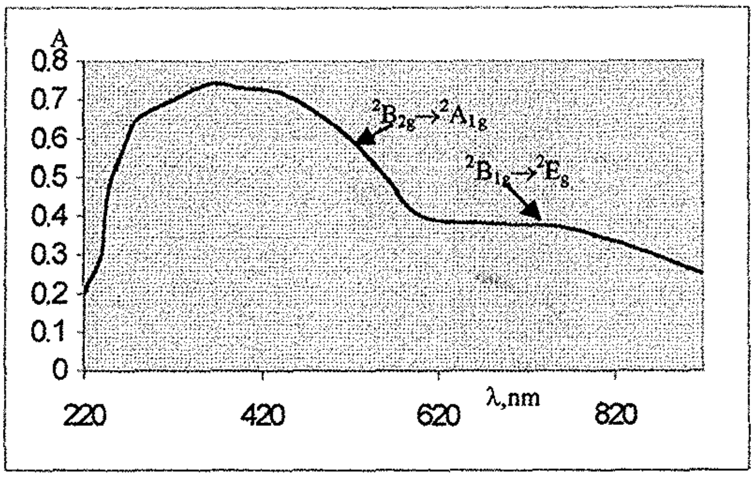


Figure 4. Diffuse-reflection electronic spectrum of the Cu(II) complex

The sign of the octahedron distortion would be established by the Electronic Paramagnetic Resonance(EPR) spectrum, but its anisotropy is not clearly resolved (Figure 5).

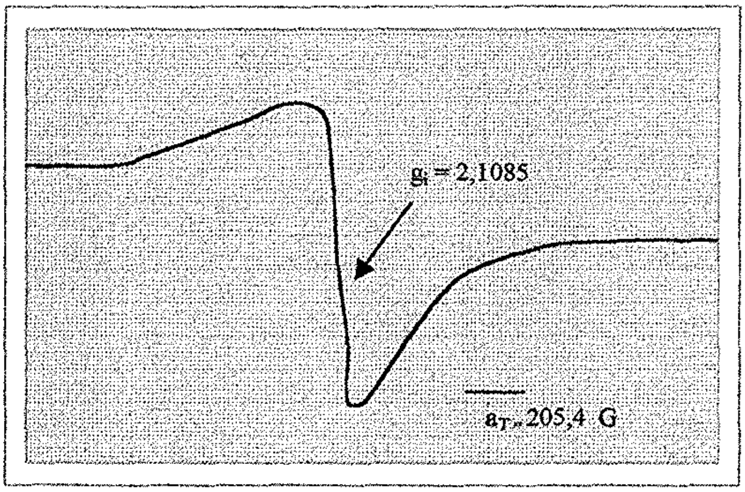


Figure 5. Electronic Paramagnetic Resonance powdered spectrum (X band) of the Cu(II) complex

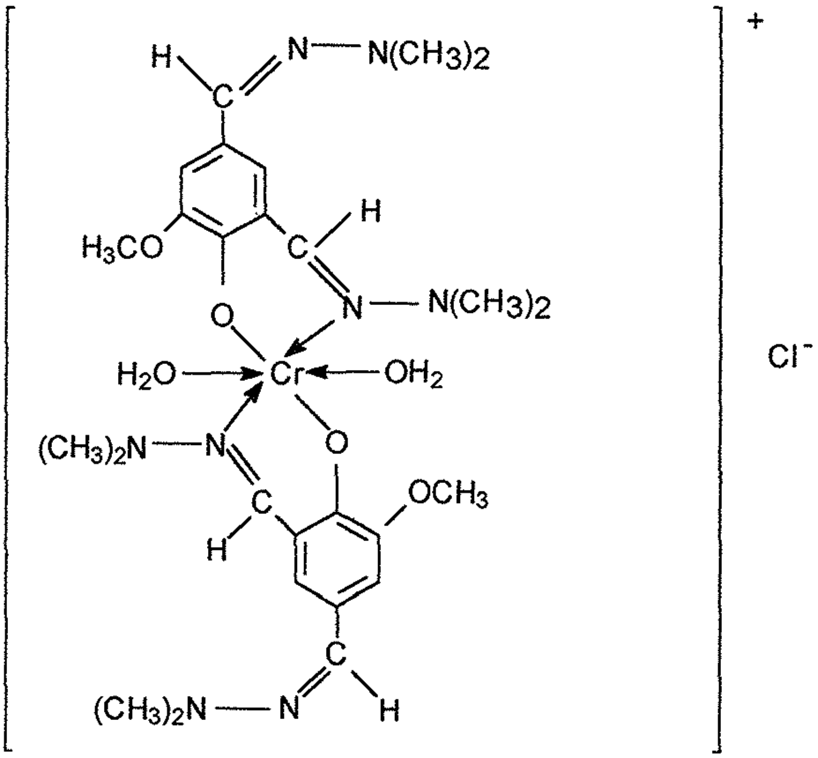
Based on an analogy with the Cr(III) and Ni(II) complexes, we proposed an octahedral symmetry, distorted by compression along the z-axis. Under this assumption, the observed bands may be assigned to the following transitions: $^2B_{2g} \rightarrow {}^2E_g$ (15200 cm^{-1}); $^2B_{2g} \rightarrow {}^2A_{1g}$ (21500 cm^{-1}); CT (23972 cm^{-1}).

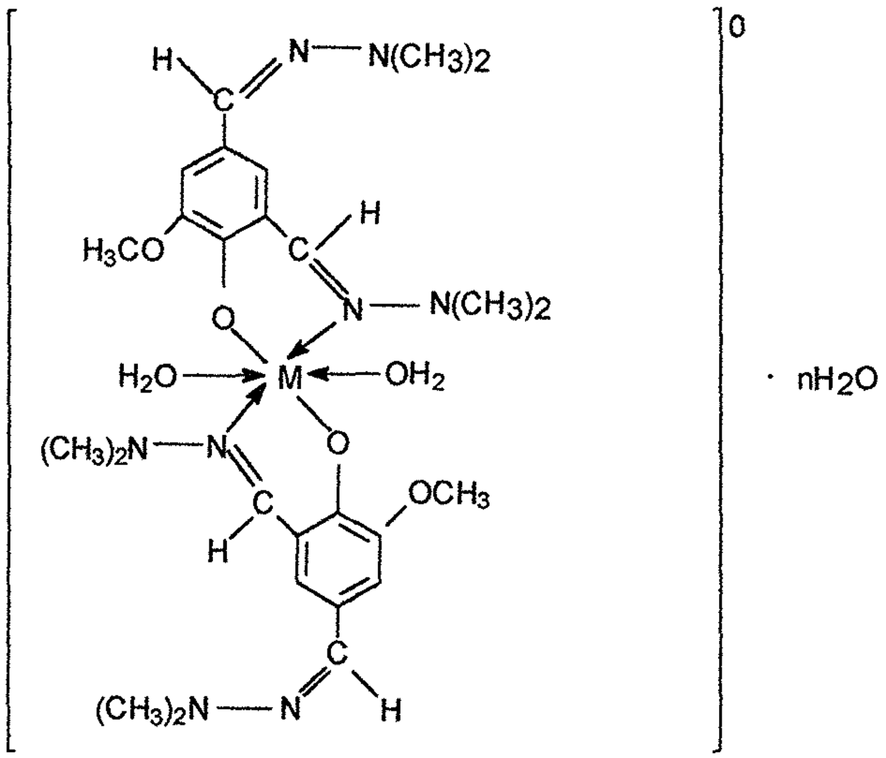
Assuming a compressed octahedral symmetry, $g_{\parallel}$ = 2,0023 and $g_{\perp}$ can be calculated with the relation[28]: g_i=1/3 ($g_{\parallel}$ + 2 $g_{\perp}$). We thus obtained $g_{\perp}$ = 2,170, a value that can be used for the calculation of the spin-orbit coupling constant, λ:

$$g_{\perp} = 2{,}0023 - 6\lambda/\Delta_2 \qquad \Delta_2 = \Delta E(d_{z^2} - d_{yz,xz})$$

The value obtained for the spin-orbit constant, λ = -410 cm^{-1}, gives λ/λ_0 = 0,49 (λ_0 = -830 cm^{-1}), indicating an important covalent character of the metal-ligand bond.

On the basis of the results of analytical and physico-chemical analyses, the following structures may be assigned to the complexes:





M = Ni; n=3
M = Cu; n=0

Figure 6. Structures of the complexes studied

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