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Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry

Publication details, including instructions for authors and subscription information: <u>http://www.tandfonline.com/loi/lsrt19</u>

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To cite this article: R. M. Issa, S. A. Abdel-Latif & H. A. Abdel-Salam (2001): SYNTHESIS AND CHARACTERIZATION OF NEW Cu(II) COMPLEXES DERIVED FROM BENZILIC AND MANDELIC HYDRAZONES, Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry, 31:1, 95-105

To link to this article: http://dx.doi.org/10.1081/SIM-100001935

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SYNTH. REACT. INORG. MET.-ORG. CHEM., 31(1), 95–105 (2001)

SYNTHESIS AND CHARACTERIZATION OF NEW Cu(II) COMPLEXES DERIVED FROM BENZILIC AND MANDELIC HYDRAZONES

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ABSTRACT

Two new sets of Cu(II) complexes with newly synthesised benzilic and mandelic hydrazone derivatives were prepared in the mole ratios 1:1 and 1:2 (Cu:L). The structure of the complexes was identified on the basis of the data obtained from elemental and thermal analyses as well as IR, UV-visible, ESR spectra, and X-ray diffraction. The results indicate that the ligands are tightly bound to the metal ion through the phenolic OH oxygen, the azomethine nitrogen, and the enolic OH oxygen in case of the 1:1 complexes while for the 1:2 complexes the enolic OH group did not participate in bonding. The complexes have elongated octahedral as well as square-planar symmetries.

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Figure 1. Ketonic (I) and enolic (II) structures of the hydrazone part of the complexes with copper ions.

INTRODUCTION

The interest in studying the hydrazides of organic acids and their derivatives arises from the fact that most of these compounds have relevant biological properties such as virus inhibitors (1), analgesics (2), antitumor agents (3), antiradiation drugs (4), and other pharmacological activities. The biological activity of hydrazides was ascribed to their ability to form chelates with transition metal ions (5). Transition metal ion complexes with arylhydrazones have found many applications as catalysts in various chemical and photochemical reactions as well as in some biological systems (6). The use of arylhydrazone complexes in a given reaction depends to a great extent on their molecular structure. The structure of metal chelates with arylhydrazones drew the attention of many investigators, since the ligand can retain its ketonic structure (I) or react with the metal ion in the enolic form (II) as shown in Figure 1 for copper ions.

The reactivity of the metal complex in biological systems was found to depend on its structure. The structure of the ligand in the complex was also found to be dependent on the nature of the anion of the metal salt used and the alkalinity of the medium (7-10). Also, arylhydrazones have been used for the spectrophotometric determination of some metal ions (11,12).

The present work is concerned with the preparation of some new Cu(II) complexes of benzilic and mandelic hydrazones (Fig. 2)



Figure 2. Structure of benzilic and mandelic hydrazones. $X = C_6H_5$ for benzilic hydrazones and H for mandelic hydrazones; $Y = H (H_3L^1)$, 4-OH (H_4L^2) or $C_6H_4 (H_3L^3)$ for benzilic hydrazones; $Y = H (H_3L^4)$, 4-OH (H_4L^5) or $C_6H_4 (H_3L^6)$ for mandelic hydrazones.

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The complexes were identified on the basis of the data obtained from elemental and thermal analyses, IR, UV-visible, ESR spectra, and X-ray diffraction.

EXPERIMENTAL

All chemicals used in the present work were chemically pure from BDH Chemicals. The ligands utilised were prepared by the condensation of benzilic or mandelic hydrazide with salicylaldehyde, 2,4-dihydroxybenzaldehyde, or 2-hydroxy-1-naphthaldehyde following a known procedure using ethanol as solvent (13,14). After refluxing the reaction mixture for 2 h on a water bath, the respective hydrazones separated on cooling. The compounds were filtered, recrystallized from ethanol and then dried in a desiccator over silica gel. The purity of the ligands under investigation has been confirmed by a sharp melting point, elemental analyses (Table 1), IR, electronic, and ¹H NMR spectra.

The Cu(II) complexes were prepared by mixing the ligands (0.01 or 0.02 mol in 100 mL ethanol) with anhydrous Cu(II) acetate (0.01 mol, 1.8164 g) in 100 mL ethanol. The mixture was refluxed for 8 h on a water bath, then cooled to a room temperature. The solid complexes which separated were filtered, washed with ethanol, and dried in a desiccator over silica gel. The apparatus and working procedures were the same as described previously (15).

RESULTS AND DISCUSSION

The 1:1 and 1:2 (Cu:L) complexes were prepared as shown in the experimental part according to the following equations:

					Melting	%C	%H	%N
Cpd.	Empirical Formula	Formula Weight	% Yield	Color ^a	Point (°C)	Found (Calc.)	Found (Calc.)	Found (Calc.)
			Ben	zilic hyd	lrazone der	ivatives		
H_3L^1	$C_{21}H_{18}N_2O_3$	346	75	p.y.	211-212	72.39 (72.83)	5.09 (5.20)	7.57 (8.09)
H_4L^2	$C_{21}H_{18}N_2O_4$	362	80	p.y.	261-262	69.45 (69.61)	5.09 (4.97)	7.66 (7.73)
H_3L^3	$C_{25}H_{20}N_{2}O_{3} \\$	396	65	у.	265-267	75.42 (75.76)	5.70 (5.05)	7.16 (7.07)
			Man	delic hyd	drazone de	rivatives		
H_3L^4	$C_{15}H_{14}N_2O_3$	270	70	w.	180-181	66.53 (66.67)	5.30 (5.19)	10.55 (10.37)
H_4L^5	$C_{15}H_{14}N_2O_4$	286	65	p.o.	215-216	62.84 (62.94)	5.01 (4.90)	9.72 (9.79)
H ₃ L ⁶	$C_{19}H_{16}N_{2}O_{3} \\$	320	75	у.	242-243	70.99 (71.25)	5.49 (5.00)	8.73 (8.75)

Table 1. Analytical Data of Benzilic and Mandelic Hydrazone Derivatives

^{*a*}p.y.: pale yellow, Y.: yellow, w.: white, p.o.: pale orange.



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For benzilic hydrazone (1:1) complexes:

$$\begin{split} H_{3}L^{1} + Cu^{2+} &\rightarrow [Cu(HL^{1})] + 2H^{+} \\ H_{4}L^{2} + Cu^{2+} &\rightarrow [Cu(H_{2}L^{2})] + 2H^{+} \\ H_{3}L^{3} + Cu^{2+} &\rightarrow [Cu(HL^{3})] + 2H^{+} \end{split}$$

for 1:2 complexes as follows:

 $\begin{array}{l} 2H_{3}L^{1}+Cu^{2+}\rightarrow [Cu(H_{2}L^{1})_{2}]+2H^{+}\\ 2H_{4}L^{2}+Cu^{2+}\rightarrow [Cu(H_{3}L^{2})_{2}]+2H^{+}\\ 2H_{3}L^{3}+Cu^{2+}\rightarrow [Cu(H_{2}L^{3})_{2}]+2H^{+} \end{array}$

while for mandelic hydrazone (1:1) complexes:

$$\begin{split} H_{3}L^{4} + Cu^{2+} &\rightarrow [Cu(HL^{4})] + 2H^{+} \\ H_{4}L^{5} + Cu^{2+} &\rightarrow [Cu(H_{2}L^{5}] + 2H^{+} \\ H_{3}L^{6} + Cu^{2+} &\rightarrow [Cu(HL^{6})] + 2H^{+} \end{split}$$

for 1:2 complexes as follows:

 $\begin{array}{l} 2H_{3}L^{4}+Cu^{2+}\rightarrow [Cu(H_{2}L^{4})_{2}]+2H^{+}\\ 2H_{4}L^{5}+Cu^{2+}\rightarrow [Cu(H_{3}L^{5})_{2}]+2H^{+}\\ 2H_{3}L^{6}+Cu^{2+}\rightarrow [Cu(H_{2}L^{6})_{2}+2H^{+}\end{array}$

The physico-chemical data of the ligands are summarized in Table 1. The results of elemental analyses of the complexes (Table 2) show that they include one or more water or ethanol molecules. These solvent molecules are not removed in vacuo for three hours at room temperature.

Thermogravimetric and Differential Thermal Analyses

Thermogravimetric(TG) and differential thermal (DTA) analyses of the $[Cu(H_2L^6)_2(EtOH)_2]$ complex (Table 3) indicated that the ethanol molecules associated with the complex are removed within the 100–240 and 240–260°C temperature ranges. Hence, these solvent molecules are involved in the coordination sphere. Decomposition of the nonsolvated complex started at 375°C, the DTA curve of the complex exhibits two exothermic peaks at 220 and 250°C. The first is due to the removal of the ethanol molecules leading to the desolvated complex which underwent some lattice rearrangement at the second step. Finally, thermal decomposition of the complex gave a broad exothermic peak in the 330–375°C range leading to CuO as the final product.



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	Table 2.	Ana]	lytical a	und ES	R Data	of Cu(II) Be	enzilic or N	Aandelic Hy	drazone Coi	mplex			
	Empirical			20		%C Found	%H Found	%N Found	%Cu Found		ESR	- 4	
Complex	Formula	Cu:L	F.W.	Yield	Color ^a	(Calc.)	(Calc.)	(Calc.)	(Calc.)	$g_z = g_{//}$	$g_{\mathbf{x}} = g_{\perp}$	g_{y}	$g_{\rm iso}$
[Cu(HL ¹)EtOH	$C_{23}H_{22}CuN_2O_4$	1:1	453.5	70	y.g	60.50	5.36	6.64	13.96	I	I	Ι	I
						(60.85)	(4.85)	(6.17)	(14.00)				
[Cu(H ₂ L ¹) ₂]EtOH	$C_{44}H_{40}CuN_4O_7$	1:2	799.5	80	1.g	66.27	5.13	68.9	8.03	I	I	I	2.0955
						(66.04)	(5.00)	(00)	(7.94)				
[Cu(H ₂ L ²)EtOH]-	$C_{23}H_{24}CuN_2O_6$	1:1	487.5	75	si.	56.45	5.39	5.31	13.21	I	I	I	I
H_2O						(56.61)	(4.92)	(5.74)	(13.02)				
$[Cu(H_3L^2)_2-$	$C_{42}H_{42}CuN_4O_{12}$	1:2	857.5	65	1.g	58.62	4.98	6.96	7.06	2.109	2.006	2.058	2.0581
$(H_2O)_2]2H_2O$						(58.77)	(4.89)	(6.53)	(7.40)				
[Cu(H ₂ L ³) ₂]EtOH	$C_{52}H_{44}CuN_4O_7$	1:2	899.5	65	à	69.64	4.92	6.06	6.81	I	I	I	I
						(69.37)	(4.89)	(6.22)	(1.06)				
$[Cu(HL^4)H_2O]$	$C_{15}H_{14}CuN_2O_4$	1:1	349.5	70	à	51.11	4.06	7.93	18.20	I	I	I	2.0158
						(51.50)	(4.01)	(8.01)	(18.17)				
$[Cu(H_2L^4)_2]H_2O$	$C_{30}H_{28}CuN_4O_6$	1:2	603.5	75	50	59.45	4.59	9.16	10.40	I	Ι	I	I
						(59.65)	(4.63)	(9.27)	(10.52)				
$[Cu(H_2L^5)H_2O-$	$C_{19}H_{26}CuN_2O_7$	1:1	457.5	80	d.g.	50.13	5.29	6.48	13.75	2.085	2.003	2.02	2.0345
$(EtOH)_2$]						(49.84)	(5.68)	(6.12)	(13.88)				
$[Cu(H_3L^5)_2-$	C ₃₄ H ₃₈ CuN ₄ O ₁₀	1:2	725.5	65	50	56.19	5.61	7.70	8.86	2.032	2.007	2.023	2.0205
$(EtOH)_2$]						(56.24)	(5.24)	(7.72)	(8.75)				
[Cu(HL ⁶)EtOH]	$C_{21}H_{20}CuN_2O_4$	1:1	427.5	75	d.g.	58.54	4.31	6.85	14.99	I	I	Ι	I
						(58.95)	(4.68)	(6.55)	(14.85)				
$[Cu(H_2L^6)_2-$	$\mathrm{C}_{42}\mathrm{H}_{42}\mathrm{CuN}_4\mathrm{O}_8$	1:2	793.5	70	ы	63.56	5.04	6.96	8.07	Ι	Ι	Ι	I
$(EtOH)_2$]						(63.51)	(5.29)	(7.05)	(8.00)				
^a y.g.: yellowish g	green, 1.g.: light g	green,	g.: gree	en, d.g	:: dark g	green.							



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		Temperate	ure, °C		%l	055	
	Decomp.	Temp.	TG	DTA	,01	000	
Complex	Init	Cu-lig. Core	Thermal step	Exo	Found	Calc.	Assignment
$\begin{array}{c} [Cu(H_2L^6)_2\\ (EtOH)_2] \end{array}$	50	280	100–240	220	5.84	5.81	Coordinated EtOH
$\begin{array}{c} [Cu(H_2L^6)_2\\ EtOH] \end{array}$	-	-	240-260	250	11.67	11.62	Coordinated EtOH
$[\operatorname{Cu}(\operatorname{H}_2\operatorname{L}^6)_2]$	-	-	260–280	-	-	-	Thermal stability
$[Cu(H_2L^6)^+$	-	-	280–375	-	51.56	51.80	One molecule of ligand
	_	_	375	_	_	_	Decomp. and formation of Cu_2O

Table 3. TG-DTA of Mandelic Hydrazone Cu(II) 1:2 Complexes

100

Infrared Spectra

The IR spectra (Table 4) of all of the complexes exhibit a broad band within the range 3550–3300 cm⁻¹, which is assigned to ν (OH) of solvent (ethanol or water) molecules associated with the Cu(II) ions in the complexes; the ν (NH) band at 3301–3260 cm⁻¹ and the ν (C=O) band at 1674–1650 cm⁻¹ in the spectra of the free ligands are absent in the spectra of the (1:1) complexes with HL¹, H_2L^2 , HL^4 , H_2L^5 , HL^6 indicating that they are bound to Cu(II) ions in the enolic form. The ν (OH) bands of the phenolic (3230–3180 cm⁻¹) and enolic groups $(3070-3050 \text{ cm}^{-1})$ in the spectra of the free ligands are absent in those of the Cu(II) complexes. This denotes that Cu(II) ions are bound to the ligands through displacement of protons of these two OH groups. This assumption finds support in the disappearance of the ν (C–H) ligand bands at 1060–1050 cm⁻¹ and δ (OH) at 1350–1270 cm⁻¹ on complex formation. The ν (C=N) band at 1640 cm⁻¹ in the spectra of the ligands is split into two bands, one at 1640 cm^{-1} while the second is located at 1617–1609 cm⁻¹; this shift denotes the coordination of Cu(II) through one azomethine nitrogen, most probably that of the hydrazone. The ν (OH) of the alcoholic group at $3270-3220 \text{ cm}^{-1}$ in the spectra of the free ligands is still observed at the same position for the Cu(II) complexes, indicating that this group does not participate in the bonding between the ligands and Cu(II) ions.

According to the results of the IR spectral study of the 1:1 complexes, the bonding of Cu(II) to the ligands takes place through two covalent bonds, with the oxygen of the phenolic and enolic OH groups and a coordinative bond to the azomethine nitrogen atom of the hydrazone. Thus, the hydrazones behave as dibasic tridentate ligands. The suggested mode of bonding is further supported by

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Tabl	e 4. IR and	Electronic	c Spectral	Data of C	u(II) Benz	zilic or Man	delic Hyd	razone Co	mplexes		
				IR (cn	n ⁻¹)				UV-V	isible λ _{max} -	(um)
Complex	ν(OH) H ₂ O/EtOH	ν (OH) alc.	ν (C=N) firee	ν (C=N) coord.	β(OH)	γ(C-OH)	ν(M-O)	ν(M-N)	DMF	$\varepsilon \times 10^{-2}$	Nujol
[Cu(HL ¹)EtOH]	3530 m	3420 m	1640 sh	1613 s	1276 m	1049 m	476 m	356 m	800,610	sat.sol.	820,620
$[Cu(H_2L^2)EtOH]H_2O$	$3550 \mathrm{m}$	3378 m	1640 sh	1611 s	1349 m	1050 m	465 m	357 m	800,600	sat.sol.	840,650
$[Cu(HL^4)H_2O]$	3480 m	3370 m	1640 m	1609 s	1251 s	1057 m	523 m	359 m	635	1.17	815,630
$[Cu(H_2L^5)H_2O(EtOH)_2]$	3600 m	3480 m	1640 sh	1610 s	1278 s	1054 m	465 m	357 w	625	1.79	875,675
[Cu(HL ⁶)EtOH]	3510 m	$3400 \mathrm{m}$	1640 sh	1617 s	1301 s	1056 m	476 m	377 m	630	1.62	850,650
$[Cu(H_2L^1)_2]EtOH^a$	3536 m	3380 s	1675 m	1619 s	$1274 \mathrm{~s}$	1050 m	461 m	331 w	800,620	sat. sol.	820,635
$[Cu(H_3L^2)_2(H_2O)_2]2H_2O^{a}$	3540 m	$3400 \mathrm{m}$	1669 m	1611 s	1352 m	1050 m	450 w	330 w	800,600	2.55	830,660
$[Cu(H_2L^3)_2]EtOH^a$	3540 m	3390 s	1675 s	1618 s	1320 m	1061 m	475 m	375 w	800,625	1.69	845,625
$[Cu(H_2L^4)_2]H_2O^a$	3600 m	3360 s	1677 s	1612 s	1274 m	1095 m	477 m	331 m	630	1.63	775,630
$[Cu(H_3L^5)_2(EtOH)_2]^d$	$3500 \mathrm{m}$	3400 s	1650 s	1608 s	1357 m	1056 s	466 m	341 m	625	1.25	850,650
$[Cu(H_2L^6)_2(EtOH)_2]^d$	$3540 \mathrm{m}$	3360 s	1675 s	1618 m	1323 m	1056 m	479 m	380 m	625	1.78	870,650

 $^a\,\nu(\rm NH)$ 3276–3303; s: strong, m: medium, w: weak, sh: shoulder.



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the appearance of two new bands at 532–465 and 377–329 cm⁻¹ in the spectra of the complexes, which are assigned to ν (Cu–O) and ν (Cu–N), respectively (16). The spectra of the 1:2 complexes display a behaviour that is somewhat different from that of the 1:1 complexes. The bands due to ν (OH) alcohol, ν (OH) enol, ν (NH) (3300–3260 cm⁻¹), and ν (C=O) display only small changes in their positions, indicating that these groups do not coordinate to Cu(II). Also, the ligand is coordinated to the central metal ion in the ketonic form.

The $\nu(OH)_{phenolic}$ is absent in the spectra of the complexes and the $\nu(C=N)$ band is shifted to lower wave numbers by 32–21 cm⁻¹ which reveals that Cu(II) is bonded to the ligands through coordination bonds with the azomethine nitrogen of the hydrazone part and the oxygen of the deprotonated phenolic group. This is confirmed by the appearance of two new bands at 479–450 and 380–330 cm⁻¹ in the spectra of the complexes which are assigned to $\nu(Cu-O)$ and $\nu(Cu-N)$ modes (16). Thus, in the 1:2 complexes the hydrazones behave as monobasic bidentate ligands.

Electronic Absorption Spectra

The electronic absorption spectra of the complexes under study were measured as Nujol mulls and in DMF solution. The results (Table 4) are very similar, indicating that no changes in their geometry occur on going from the solid state to solution. The spectra exhibit one or two broad bands at 600–650 nm $(2B_{1g} \rightarrow 2B_{1g})$ and 800–850 nm $(2B_{1g} \rightarrow 2E_g)$. The spectral patterns indicate a tetragonally elongated octahedral geometry.

Electron Spin Resonance

The X-band ESR spectra of the Cu(II) hydrazone complexes at room temperature generally show one broad signal which is split in some cases into two or three signals (Table 2). These complexes are not magnetically dilute, therefore, exchange and/or dipolar forces are expected to operate in such a case, the anisotropy is likely to be reduced. This appears to be the case for $[Cu(H_2L^1)_2]EtOH$ and $[Cu(HL^4)H_2O$ complexes, where only a single EPR line is observed. The apparent broadening of the EPR signals may be due to an interaction between Cu(II) ions which are probably present in nonequivalent lattice positions. The g_{iso} value for complexes ($[Cu(H_2L^5)H_2O(EtOH)_2]$, $[Cu(H_3L^5)_2(EtOH)_2]$, $[Cu(H_2L^1)_2EtOH$, and $[Cu(H_3L^2)_2(H_2O)_2]2H_2O$, relative to the other complexes, $[Cu(HL^1)EtOH]$, $[Cu(H_2L^2)EtOH]H_2O$, $[Cu(H_2L^3)_2]$ -EtOH, $[Cu(H_2L^4)_2]H_2O$, $[Cu(HL^6)]EtOH$, and $[Cu(H_2L^6)(EtOH)_2]$, may be attributed to a varied electron donating effect of the group attached to the hydrazone molecule (17,18). The positive shift of g_{iso}





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from that of the free electron (2.0023) indicates a high covalent character of the bonding between the Cu(II) ions and the hydrazone ligands (19).

X-Ray Diffraction

On comparing the X-ray diffraction pattern of the complexes under study with those of the free ligands, the following may be pointed out: The diffraction patterns of the Cu(II) complexes are totally different in terms of the interplanar distances d, Åand relative intensities I/I from those of the corresponding ligands. This proves complex formation between Cu(II) ions and the hydrazone ligands. The change in d, Å, and I/\tilde{I} is attributed to differences in the structures, such as the contribution of solvent molecules to the lattice structure and the different orientation of the ligands around the metal ion (20), the various spherical rotations around the lattice axes, diffraction planes, and transition group operations (21,22). The relatively high background and the broadening of the diffraction lines in the patterns of the complexes reflect a crystalline structure and strain occurring in the crystal lattice. This results from the orientation of the ligand molecules around the central Cu(II) ions or the distortion of the coordination polyhedron around the central metal ion (16). The diffraction patterns of the 1:1 complexes $[Cu(HL^1)EtOH]$ and $[Cu(HL^4)H_2O]$ differ completely from the corresponding 1:2 complexes $[Cu(H_2L^1)_2]$ EtOH and $[Cu(H_2L^4)_2]H_2O$.

CONCLUSION

Based on the knowledge gained from the results of the various methods used, the structures of the Cu(II) complexes with Benzilic and Mandelic hydrazones for the 1:1 complexes are square-planar for [Cu(HL¹)-EtOH], [Cu(H₂L²)EtOH]H₂O, [Cu(HL⁴)H₂O] and [Cu(HL⁶)EtOH] as shown in Figure 3 and for the 1:2 complexes [Cu(H₂L¹)₂]EtOH, [Cu(H₂L⁴)₂]H₂O and [Cu(H₂L³)₂]EtOH as shown in



Figure 3. The structural formula of the 1:1 complexes formed between Cu(II) ions and the benzilic or mandelic hydrazones.



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 $S = solvent (H_2O or EtOH)$

Figure 4. The 1:2 complexes of Cu(II) ions with benzilic or mandelic hydrazones.

Figure 4, while for the 1:1 complex $[Cu(H_2L^5) H_2O(EtOH)_2]$ and the 1:2 complex $[Cu(H_3L^2)_2(H_2O)_2]2H_2O$, $[Cu(H_3L^5)_2(EtOH)_2]$ and $[Cu(H_2L^6)_2(EtOH)_2]$ an elongated octahedral structure is assigned as shown in Figures 3 and 4.

Axial solvent molecules are present in case of the 1:1 complexes $[Cu(H_2L^5)-H_2O(EtOH)_2]$ and $[Cu(H_3L^2)_2(H_2O)_2]2H_2O$, $[Cu(H_3L^5)_2(EtOH)_2]$ and $[Cu(H_2L^6)_2(EtOH)_2]$ but no solvent molecules are present in the other complexes.

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Received June 16, 2000 Accepted October 27, 2000

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