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Copper(II) Complexes of Schiff Bases Derived of 2-Hydroxy-3-naphthaldehyde. The Crystal and Molecular Structures of Bis-{(phenyl)[(2-oxo-3*H*-naphth-3-ylidene)methyl]aminato}Copper(II) and Bis-{(benzene-4-trifluoromethyl)[(2-oxo-3*H*-naphth-3-ylidene)-methyl]aminato}copper(II)

J. M. Fernández-G.*, **J. J. Lembrino-Canales**, and **R. Villena-I.**

Instituto de Química, U.N.A.M., Circuito Exterior, Cd. Universitaria, Coyoacán 04510, México, D.F., México

Summary. The Schiff base ligands, 3-[(Phenyl)-2-hydroxy-3*H*-Naphth-3-ylidene)methyl]aldamine (**1**) and 3-[(benzene-4-trifluoromethyl)-2-hydroxy-3*H*-naphth-3-ylidene)methyl]aldamine (**2**), and their corresponding Cu(II) complexes (**I** and **II** were synthesized. The crystal and molecular structures of **I** and **II** were determined. Compound **I** crystallizes in the triclinic crystal system $a = 10.804(5)$, $b = 12.589(5)$, and $c = 10.369(3)$ (Å), $\alpha = 107.72(3)$, $\beta = 95.75(3)$, and $\gamma = 76.32(4)$(°), in the space group $P\bar{1}$ with $Z = 2$. Compound **II** crystallizes in the triclinic crystal system $a = 10.718(2)$, $b = 13.861(4)$, and $c = 10.110(9)$ (Å), $\alpha = 95.99(2)$, $\beta = 90.16(2)$, and $\gamma = 93.90(2)$(°), in the space group $P\bar{1}$ with $Z = 2$. The geometry around the metal atom in both complexes **I** and **II** is square planar.

Keywords. Hydroxy-naphthalenic Schiff bases; Schiff base copper(II) complexes; Copper(II) complexes; Crystal structure.

Kupfer(II)-Komplexe von Schiffbasen von 2-Hydroxy-3-naphthaldehyd. Die Kristall- und Molekülstrukturen von Bis{(phenyl)[(2-oxo-3*H*-naphth-3-yliden)methyl]aminato}kupfer(II) und Bis{(benzen-4-trifluoromethyl)[(2-oxo-3*H*-naphth-3-yliden)methyl]aminato}kupfer(II)

Zusammenfassung. Es wurden die Schiffbasen-Liganden 3-[(Phenyl)-2-hydroxy-3*H*-naphth-3-yliden)-methyl]aldamin (**1**) und 3-[(Benzen-4-trifluoromethyl)-2-hydroxy-3*H*-naphth-3-yliden)-methyl]aldamin (**2**) inklusive der entsprechenden Kupfer(II)-Komplexe **I** und **II** dargestellt. Von **I** und **II** wurden die Kristallstrukturen ermittelt. Komplex **I** kristallisiert im triklinen System mit $a = 10.804(5)$, $b = 12.589(5)$, $c = 10.369(3)$ Å, $\alpha = 107.72(3)$, $\beta = 95.75(3)$ und $\gamma = 76.32(4)$° in der Raumgruppe $P\bar{1}$ mit $Z = 2$. Verbindung **II** kristallisiert ebenfalls im triklinen System mit $a = 10.718(2)$, $b = 13.861(4)$, $c = 10.110(9)$ Å, $\alpha = 95.99(2)$, $\beta = 90.16(2)$ und $\gamma = 93.90(2)$° in der Raumgruppe $P\bar{1}$ mit $Z = 2$. Die Geometrie rund um Cu ist in beiden Komplexen quadratisch-planar.

Introduction

Although metal Schiff base complexes derived of salicylaldehyde have been extensively studied [1–3], there are few X-ray structural studies of the copper(II) complexes of the 1,2-naphthalenic analogs [1–9], and as far as we are informed there is none X-ray single crystal structural study reported in literature of a copper(II) complex of any bis bidentate Schiff base derived of 2-hydroxy-3-naphthaldehyde. Therefore, it is important to develop the available structural data of closely related systems as coordination compounds of Schiff bases derived of salicylaldehyde, 2-hydroxy-1-naphthaldehyde and 2-hydroxy-3-naphthaldehyde in order to compare the effect of aromaticity and the different bond orders of the ligand, in the conformation and structure of the resulting copper(II) complexes. In this paper, we report the synthesis and characterization of two 2,3-hydroxynaphthaldimines where phenyl or benzene-4-trifluoromethyl radicals are bonded to the imine nitrogen: N(phenyl)-2-hydroxy-3-naphthaldimine (**1**) which has been described 1931 [10] and N(benzene-4-trifluoromethyl)-2-hydroxy-3-naphthaldimine (**2**), and their corresponding copper(II) complexes bis-{(phenyl)[(2-oxo-3*H*-naphth-3-ylidene)methyl]-aminato} copper(II) (**I**) and bis-{(benzen-4-trifluoromethyl)[(2-oxo-3*H*-naphth-3-ylidene)methyl]aminato} copper(II) (**II**). Single crystal X-ray analyses of the copper complexes **I** and **II** were undertaken in order to elucidate the conformation and structure of both complexes, to examine the conformational differences between them and to compare these results with the available structural data for similar copper complexes derived of salicylaldehyde.

Materials and Methods

Melting points were determined in a Fisher–Jones melting point apparatus and are uncorrected; infrared spectra (KBr disks) were recorded on a Perkin–Elmer Mod. 203-B spectrometer, uv-visible spectra on a Perkin–Elmer Mod. 552 spectrophotometer using chloroform as solvent, mass spectra were obtained using a Hewlett-Packard instrument Mod. 5985, at 70 eV ionizing potential; nmr spectra in a Varian Gemini 200 using $CDCl_3$ as solvent and *TMS* as internal standard; magnetic moment measurements were made with a Johnson Matthey magnetic susceptibility balance at 24°C. Elemental analyses were performed by Galbraith Laboratories, Inc. Knoxville, Tenn. 37921-1750 U.S.A.

The chemicals aniline, benzenamine-4-trifluoromethyl, copper(II) acetate monohydrate, phosphorus pentachloride, naphthol AS, and tin(II) chloride anhydrous were obtained from Aldrich Chemical Co. Inc., and were used without further purification, 2-hydroxy-3-naphthaldehyde was prepared as described in literature [11] using Naphthol AS as starting material. Ligands and copper complexes were prepared by methods described elsewhere [12, 13].

Crystals of **I** and **II** were grown by slow diffussion of MeOH into a CH_2Cl_2 solution. The data for the two compounds were collected on a Nicolet R3m four-circle diffractometer with graphite monochromated CuKα radiation ($\lambda = 1.54178$ Å) using an ω-scan mode with variable scan rate from 4 to 30 deg/min and a scan width of 1.0°. Crystal data and additional data collection parameters are given in Table 1. Cell dimensions for **I** and **II** were determined by least squares from 25 machine centered reflections. Corrections were applied for Lorentz and polarization effects, absorption corrections ignored.

The structures were solved by direct methods and subsequent use of Fourier difference maps. They were refined by the full-matrix least-squares method using anisotropic thermal parameters for all non-hydrogen atoms. The H atoms of the CH_2 and CH_3 groups were allowed to ride on bonded C atoms with a fixed isotropic temperature factor, $U = 0.06$ Å^2. The the function minimized was

Table 1. Summary of crystal and intensity collection data for I and II

Compound	I	II
Formula	$C_{34}H_{24}N_2O_2Cu$	$C_{36}H_{22}N_2O_2F_6Cu$
Molecular Weight	556.102	692.106
Crystal System	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Crystal colour	Brown-red	Brown-red
T	298 K	298 K
Crystal dimensions (mm)	0.2 × 0.5 × 0.2	0.26 × 0.1 × 0.3
a (Å)	10.804(5)	10.718(2)
b (Å)	12.589(5)	13.861(4)
c (Å)	10.369(3)	10.110(3)
α (°)	107.72(3)	95.99(2)
β (°)	95.75(3)	90.16(2)
γ (°)	76.32(4)	93.90(2)
V(Å^3)	1298.44(0.94)	1490.21(0.65)
$\rho_{\text{calc.}}$ (g cm^{-3})	1.423	1.54
Z	2	2
F(000)	573.89	701.88
μ cm^{-1}	14.22	16.67
Total reflections measrd	3268	3756
Unique reflections [I − 3σ(I)]	2973	3283
R	0.0513	0.054
R_w	0.0616	0.0677
σ limit [$F > n\sigma(F)$], n	3	3
$2\Theta_{\text{min}}$,°	3	3
$2\Theta_{\text{max}}$,°	110.0	110.0

$\Sigma W|\Delta F|^2$ with a weighting scheme $W = [\sigma^2(F_0) + G(F_0)^2]^{-1}$ was used where σ is the esd, based on counting statistics and G an adjustable variable, final G for I and II, $G = 0.004$ and $G = 0.0012$ respectively. The final R and R_w for I and II were 0.051, 0.068 and 0.054, 0.062, respectively [where $R_w = \Sigma w^{1/2} \| F_0| - |F_c\| / \Sigma w^{1/2}(|F_0|)$].

The largest peaks in the final difference map for I and II were 0.246 to −0.382 and 0.397 to −0.415 e/Å^3, respectively, and s values were 1.369 and 1.792. The atomic scattering factors were taken from International Tables for X-ray Crystallography [14]. Solution and refinement of I and II were carried out by using SHELXTL [15] on a Data General NOVA 4S computer and plots drawn on a Tektronix Plotter.

Results and Discussion

Some physical properties of the synthesized compounds are listed in Table 2. All compounds are soluble in dichloromethane or chloroform, but the complexes are less soluble than the ligands in other solvents such as alcohols, tetrahydrofuran, benzene, and ethyl acetate. The elemental analyses were satisfactory and show that the complexes have a ligand-to-metal ratio of 2:1. Mass spectra of the ligands show the expected molecular ion, the molecular ion in the mass spectrum of complex II agrees with the 2:1 ligand to metal stoichiometric ratio and shows the

Table 2. Color, melting points, molecular ion, magnetic moments, NMR and UV. Visible bands of compounds **1**, **2** and **I**, **II**

Compound	Color	m.p. (°C)	M^+ (m/e)	μ_{eff} (B.M.)	NMR (in ppm)	UV.Vis (in cm^{-1}) [ε]
1	yellow	160–62[a]	247	—	7.25–8.0 m (arom. H); 8.8 s (imine H); 12.75 s (O–H)	25 575[2 148]sh, 30 675[24 081], 37 736[34 495]
I	brown-red	>300	Desc.	1.85	—	21 645[4 482], 31 250[47 039], 37 736[62 718]
2	yellow	209–10	315	—	7.26–8.16 m (arom. H); 8.8 s (imine H); 12.3 s (O–H)	25 641[1 980]sh, 30 675[23 712], 37 175[30 120]
II	brown-red	279–80	692	1.84	—	22 522[2.247]sh, 25 510[2 069]sh, 30 769[49 153], 37 313[68, 656

[a] Lit. 158–59 °C

Table 3. Atom coordinates (× 10^4) and temperature factors ($Å^2 \times 10^3$) for compounds **I** and **II**

Atom	x	y	z	U^a
	Compound **I**			
Cu(1)	0	0	0	63(1)
O(1a)	1646(2)	−901(2)	26(3)	79(1)
N(1a)	670(3)	1258(2)	−353(3)	60(1)
C(1a)	3691(3)	−1808(3)	−722(4)	65(1)
C(2a)	2676(3)	−892(3)	−548(4)	61(1)
C(3a)	2778(3)	54(3)	−1006(3)	55(1)
C(4a)	3904(3)	35(3)	−1557(3)	58(1)
C(5a)	6128(3)	−893(3)	−2223(3)	63(1)
C(6a)	7114(4)	−1805(4)	−2346(4)	73(2)
C(7a)	7015(4)	−2739(3)	−1947(4)	74(2)
C(8a)	5912(4)	−2755(3)	−1411(4)	76(2)
C(9a)	4841(3)	−1833(3)	−1269(3)	61(1)
C(10a)	4948(3)	−884(3)	−1697(3)	56(1)
C(11a)	1786(3)	1082(3)	−835(3)	58(1)
C(12a)	−114(3)	2385(3)	−162(4)	59(1)
C(13a)	−405(4)	2876(4)	−1197(5)	81(2)
C(14a)	−1168(5)	3978(4)	−958(6)	107(3)
C(15a)	−1629(5)	4569(4)	329(5)	103(3)
C(16a)	−1349(4)	4071(4)	1329(5)	90(2)
C(17a)	−607(4)	2986(4)	1101(4)	75(2)
Cu(2)	5000	0	5000	52(1)
O(1b)	3651(2)	1149(2)	5876(2)	58(1)
N(1b)	3826(2)	−1088(2)	4276(3)	54(1)
C(1b)	1706(3)	2433(3)	5789(3)	58(1)
C(2b)	2465(3)	1339(3)	5431(3)	53(1)
C(3b)	1904(3)	440(3)	4547(3)	54(1)
C(4b)	653(3)	675(3)	4101(4)	61(1)
C(5b)	−1399(3)	2053(3)	3994(4)	70(2)
C(6b)	−2093(4)	3145(4)	4357(4)	82(2)
C(7b)	−1541(4)	4027(4)	5182(5)	92(2)
C(8b)	−316(4)	3811(4)	5651(4)	82(2)
C(9b)	448(3)	2675(3)	5312(3)	58(1)
C(10b)	−104(3)	1784(3)	4456(3)	57(1)
C(11b)	2607(3)	−735(3)	4102(3)	59(1)
C(12b)	4319(3)	−2292(3)	3759(3)	57(1)
C(13b)	4084(5)	−2878(4)	2452(4)	93(2)
C(14b)	4596(6)	−4044(4)	1981(5)	124(3)
C(15b)	5339(4)	−4609(4)	2844(6)	105(2)
C(16b)	5514(4)	−4028(3)	4154(5)	85(2)
C(17b)	5041(3)	−2873(3)	4620(4)	67(1)
	Compound **II**			
Cu(1)	5000	0	10000	54(1)
O(1a)	5229(2)	1288(2)	10755(3)	66(1)
C(1a)	4737(4)	2760(3)	11907(4)	59(1)
C(2a)	4389(3)	1914(3)	11093(4)	53(1)

(*continued on* p. 280)

Table 3. (*Continued*)

Atom	x	y	z	U^a
C(3a)	3131(4)	1780(3)	10616(4)	53(1)
C(4a)	2286(4)	2460(3)	11015(4)	60(1)
C(5a)	1765(4)	4004(3)	12270(4)	73(2)
C(6a)	2135(5)	4832(3)	13044(5)	85(2)
C(7a)	3377(5)	4986(3)	13474(5)	88(2)
C(8a)	4238(5)	4341(3)	13112(4)	75(2)
C(9a)	3890(4)	3455(3)	12295(4)	61(1)
C(10a)	2622(4)	3298(3)	11866(4)	58(1)
C(11a)	2702(4)	965(3)	9689(4)	57(1)
N(1a)	3328(3)	250(2)	9241(3)	54(1)
C(12a)	2714(3)	−471(3)	8281(4)	53(1)
C(13a)	3224(4)	−646(3)	7035(4)	67(2)
C(14a)	2698(4)	−1361(3)	6134(4)	66(2)
C(15a)	1651(4)	−1924(3)	6478(4)	57(1)
C(16a)	1145(4)	−1731(3)	7708(4)	67(1)
C(17a)	1658(4)	−1014(3)	8620(4)	61(1)
C(18a)	1098(4)	−2735(3)	5532(4)	80(2)
F(1a)	1700(4)	−2873(3)	4403(3)	99(2)
F(2a)	−63(4)	−2578(4)	5165(5)	132(2)
F(3a)	966(27)	−3556(10)	6036(16)	98(5)
Cu(2)	0	5000	0	54
O(1b)	−259(2)	6268(2)	698(3)	67(1)
C(1b)	161(4)	7820(3)	1818(4)	59(1)
C(2b)	546(3)	7000(3)	1087(4)	55(1)
C(3b)	1845(3)	6984(3)	756(3)	51(1)
C(4b)	2661(4)	7763(3)	1203(4)	60(1)
C(5b)	3112(4)	9387(3)	2448(4)	70(2)
C(6b)	2699(5)	10175(3)	3170(4)	80(2)
C(7b)	1453(5)	10210(3)	3452(4)	87(2)
C(8b)	599(4)	9460(3)	3036(4)	75(2)
C(9b)	994(4)	8809(3)	2279(4)	58(1)
C(10b)	2270(4)	8581(3)	1984(3)	56(1)
C(11b)	2338(3)	6210(3)	−98(4)	56(1)
N(1b)	1725(3)	5414(2)	−595(3)	54(1)
C(12b)	2396(3)	4741(3)	−1437(3)	50(1)
C(13b)	3422(3)	4337(3)	−942(4)	59(1)
C(14b)	3996(4)	3620(3)	−1725(4)	64(1)
C(15b)	3551(4)	3314(3)	−3004(4)	61(1)
C(16b)	2536	3751	−3489	67
C(17b)	1966	4453	−2706	64
C(18b)	4127	2490	−3809	86
F(1b)	3412(23)	2231(13)	−4975(17)	126(6)
F(2b)	5362(20)	2430(18)	−3400(12)	90(6)
F(3b)	4928(21)	2792(10)	−4704(12)	95(5)

[a] Equivalente isotropic U defined as one third of the trace of the of the orthogonalized U_{ij} tensor

characteristic copper isotopic distribution. The nmr spectra for the ligands show signals characteristic of the naphthalene rings and the phenyl and benzene-4-trifluoromethyl radicals. There are two signals at low field, the one at lower field (around 12 ppm) disappears with D_2O and can be attributed to a phenolic proton. The other one (around 8.8 ppm) is assigned to the proton bonded to the carbon atom of the imine group. Finally, for both ligands **1** and **2** there are two additional small signals around 10 ppm which could be an indication of an equilibrium, details of this behaviour will be subject of a further study [16].

Magnetic moments for the complexes **1** and **II** are 1.85 and 1.84 B.M., respectively, (Table 2) which are in agreement with the values described for this kind of compounds [17]. The infrared spectra of ligands show the characteristic O–H absortion bands around 3400 cm^{-1} which disappears after complexation, the absortion corresponding to ν-C=N– vibration is around 1640 cm^{-1} in the ligands, in the complexes it is splitted and shifted approximately 20 cm^{-1} to lower wave number as it has been described in similar complexes [18–19]. The electronic spectra of the complexes show only intraligand or charge transfer bands (see Table 2).

Final positional parameters for compounds **I** and **II** are given in Table 3 while Table 4 contains a selection of bond lengths and angles. The molecular structures of the compounds **I** and **II**, including atom-numbering schemes, are illustrated in Figs. 1 and 2.

Complexes **I** and **II** have similar structures. In both cases there are two crystallographic independent but structurally similar centrosymmetric molecules in the unit cell. The copper ion is four-coordinate and is bonded to the oxygen and nitrogen donor atoms of the two ligand molecules in a *trans* stepped square planar structure, with a step for the two independent molecules of **1** (**1a** and **1b**) and the two independent molecules of **II** (**IIa** and **IIb**) of 0.86, 1.18 and 0.86, 0.84 Å, respectively.

Table 4. Selected bond lengths (Å) for **I** and **II** with esd's in the least significant figure given in parentheses

Compound **I**		Compound **II**	
Cu(1)–O(1a)	1.870(2)	Cu(1)–O(1a)	1.868(3)
Cu(1)–N(1a)	2.022(3)	Cu(1)–N(1a)	2.013(3)
Cu(2)–O(1b)	1.871(2)	Cu(2)–O(1b)	1.863(2)
Cu(2)–N(1b)	2.009(3)	Cu(2)–N(1b)	2.010(3)
O(1a)–C(2a)	1.318(5)	O(1a)–C(2a)	1.314(5)
O(1b)–C(2b)	1.309(4)	O(1b)–C(2b)	1.313(4)
C(11a)–N(1a)	1.296(4)	C(11a)–N(1a)	1.279(5)
C(11b)–N(1b)	1.299(4)	C(11b)–N(1b)	1.299(4)
N(1a)–C(12a)	1.431(4)	N(1a)–C(12a)	1.441(4)
N(1b)–C(12b)	1.425(4)	N(1b)–C(12b)	1.429(4)
C(2a)–C(3a)	1.434(6)	C(2a)–C(3a)	1.424(5)
C(2b)–C(3b)	1.435(4)	C(2b)–C(3b)	1.433(5)
C(3a)–C(11a)	1.443(4)	C(3a)–C(11a)	1.439(5)
C(3b)–C(11b)	1.444(4)	C(3b)–C(11b)	1.437(5)

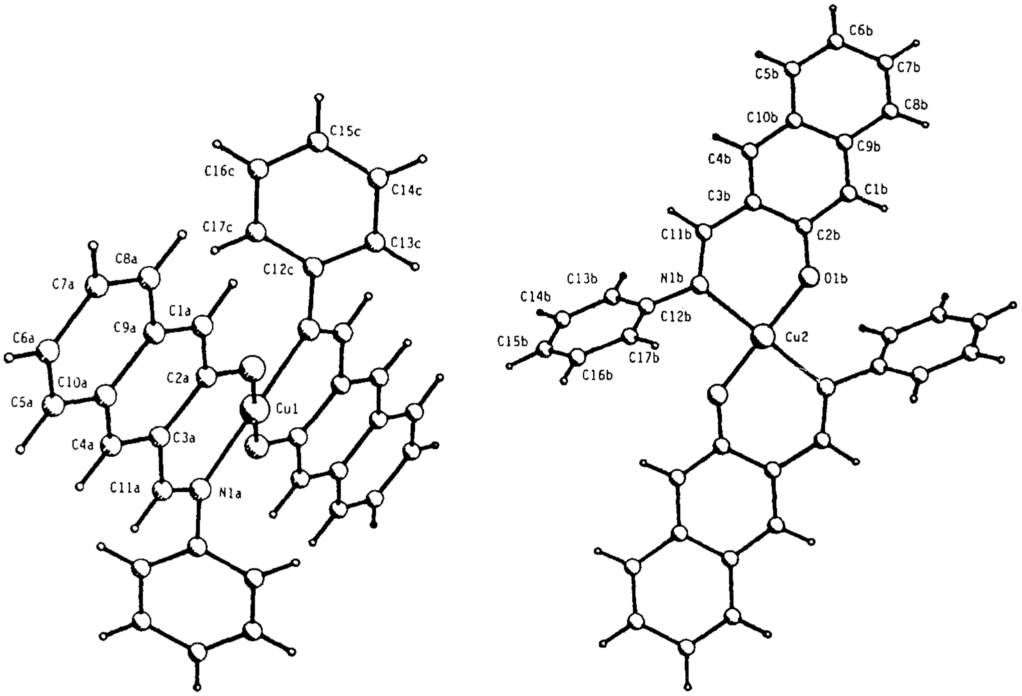

Fig. 1. Complex I

In both complexes, **I** and **II**, the corresponding Cu–O and Cu–N bonds have similar lengths and are in the same range to those reported for bis(1,2-naphthaldaminato) copper complexes [4–9] and two related Schiff base copper complexes derived of salicylialdehyde [20, 21], i.e. Cu–O distances are in the range 1.87 to 1.92 Å while Cu–N distances have values between 1.971 and 2.013 Å. The C(11)–N(1) and N(1)–C(12) bond lengths (see Table 4) are in agreement with the values found in those related salicylideneaminato copper complexes [20, 21]. The trifluoromethyl groups in **II** show some amount of disorder which limited the precision of the structure determination at this site.

Bond lengths and bond angles in the naphthalene rings are very similar in complexes and both show some differences from naphthalene itself as expected for a chelate derivative [22]. Bond angles in complexes **I** (**Ia** and **Ib**) and **II** (**IIa** and **IIb**) show also that the coordination geometry about the copper atoms is square planar with O–Cu–N angles of 91.6(1), 90.7(1) and 91.1(1), 91.2(1°), respectively. Other angles show the expected values.

The mean planes through naphthalene rings, the chelate rings, the coordination planes and the benzene rings were calculated*. In both complexes **I** and **II**, we can

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* Additional material to the structure determination for compounds I and II such as packing arrangement, atom coordinates, complete bond lenghts and angles, least-squares mean planes in I and II and angles between the least-squares mean planes, anisotropic temperature factors and hydrogen coordinates, and observed and calculated structure factors can be ordered from Fachinformationszentrum Energie-Physik-Mathemetik, D-76344 Eggenstein-Leopoldshafen 2, Germany, referring to the deposition no. CSD-57990, the names of the authors and the citation of the paper

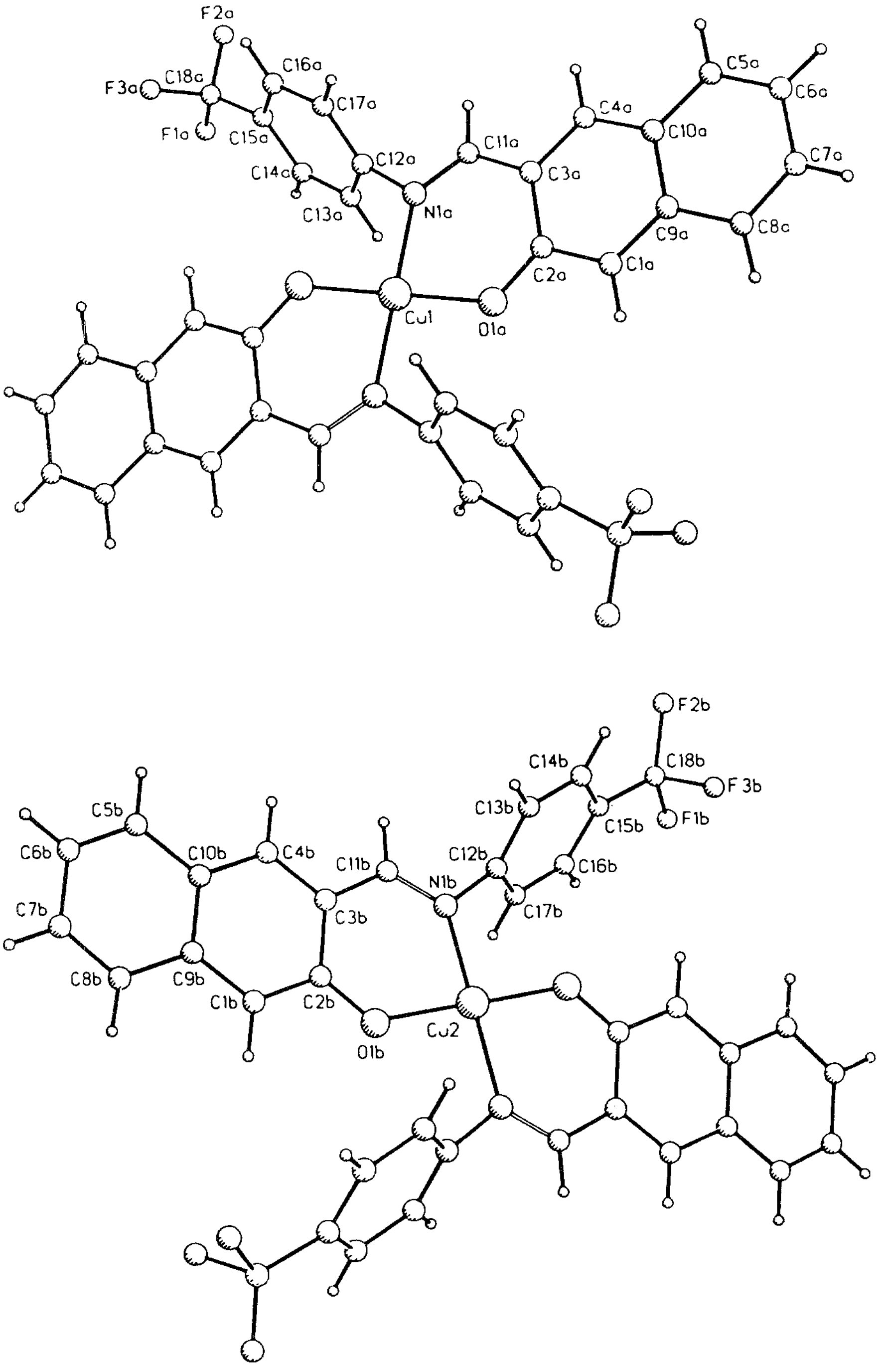

Fig. 2. Complex **II**

observe that the naphthalene and chelate ring planes of each ligand are nearly coplanar while the phenyl and benzen-4-trifluoromethyl groups are making an angle with the coordination and naphthaldimine groups.

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