

Table 3. Mean Sn—Cl bond distances (Å) in hexacoordinated chlorostannate(IV) compounds

Compound	All Sn—Cl bonds	cis Sn—Cl bonds	trans Sn—Cl bonds	Reference
[(CH ₃) ₄ N] ₂ SnCl ₆	2.424 (3)	—	—	(a)
[C ₂ H ₅ NH] ₂ SnCl ₆	2.431 (1)	—	—	(b)
[4-ClC ₂ H ₄ NH] ₂ SnCl ₆	2.432 (4)	—	—	(c)
[(CH ₃) ₂ NH(C ₂ H ₅)SnCl ₅	2.495 (4)	2.516 (3)	2.410 (4)	(d)
[C ₂ H ₅ NH] ₂ (C ₂ H ₅)SnCl ₅	2.495 (2)	2.519 (1)	2.400 (2)	(d)
[CH ₃ NH] ₂ (C ₂ H ₅)SnCl ₅	2.502 (1)	2.522 (1)	2.426 (1)	(d)
[4-CH ₃ C ₅ H ₄ NH] ₂ (C ₂ H ₅)SnCl ₅	2.500 (1)	2.526 (1)	2.397 (1)	(d)
[4-CH ₃ C ₅ H ₄ NH] ₂ (C ₆ H ₅)SnCl ₅	2.488 (1)	2.492 (1)	2.473 (1)	(e)

References: (a) Nielsen & Berg (1980); (b) Borchers & Weiss (1987); (c) Gearhart, Brill, Welsh & Wood (1973); (d) Storck & Weiss (1989); (e) this work.

are weakened through the influence of the C₂H₅⁻ group, while the *trans* Sn—Cl bonds are slightly shorter than in SnCl₆²⁻ compounds ('*trans*-strengthening and '*cis*-weakening'). Within the (C₆H₅)SnCl₅²⁻ anion of (4-CH₃C₅H₄NH)₂[(C₆H₅)SnCl₅], *cis* and *trans* bonds are weakened. This may be a result of hydrogen bonding. In none of the four (C₂H₅)SnCl₅²⁻ compounds of Table 3 were short N...Cl_{trans} distances observed (only the Cl atoms, *cis* to the ethyl group, are hydrogen bonded), while in (4-CH₃C₅H₄NH)₂[(C₆H₅)SnCl₅], the *trans* Cl atom has N...Cl distances of 3.295 and 3.598 Å. This can be compared with Cl(5), where N...Cl(5) distances of 3.216 and 3.355 Å are accompanied by a lengthening of 0.078 Å over the mean *cis* Sn—Cl bond length. Therefore, hydrogen bonds may lengthen the *trans* Sn—Cl bond by up to 0.06 Å — enough to compensate for the *trans* influence of the C₆H₅⁻ group.

The ³⁵Cl NQR signals observed for (4-CH₃C₅H₄NH)₂[(C₆H₅)SnCl₅] are weak. Only two of the expected five signals showed a sufficient intensity to be recognized unambiguously as NQR resonances.

The ³⁵Cl NQR frequencies are 15.92 and 14.66 MHz at room temperature.

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Structure of Bis{*N*-[2-(2-pyridyl)ethyl]-3-ethoxysalicylideneaminato}copper(II)

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Abstract. [Cu(C₁₆H₁₇N₂O₂)₂], *M_r* = 602.19, monoclinic, *P*2₁/*n*, *a* = 13.246 (5), *b* = 7.155 (3), *c* = 15.123 (4) Å, β = 92.25 (4)°, *V* = 1432.2 Å³, *Z* = 2, *D_x* = 1.397 g cm⁻³, λ(Cu Kα) = 1.54184 Å, μ =

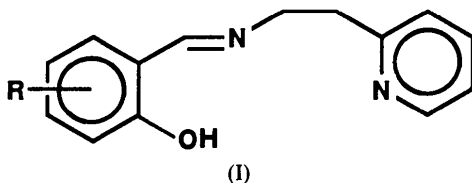
13.950 cm⁻¹, *F*(000) = 630, *T* = 293 K. The final *R* value is 0.045 for 1896 significant [*I* > 3σ(*I*)] reflections. In the centrosymmetric compound the Cu atom is bonded to an imine N atom and a phenolate

Table 1. *Data-collection and structure-refinement parameters*

Crystal shape, dimensions	Prismatic, 0.10 × 0.10 × 0.35 mm
Diffractometer used	CAD-4, Enraf-Nonius
Method of intensity measurement	θ -2 θ
No. and θ range of reflections for lattice parameters	25; 17–30°
Method used for absorption correction	DIFABS (Walker & Stuart, 1983)
Minimum absorption correction	0.732
Maximum absorption correction	1.514
Average absorption correction	0.987
Maximum value of $(\sin \theta)/\lambda$ reached in intensity measurement	0.588 Å ⁻¹
Range h, k, l	0→15, -8→8, -17→17
Standard reflections	323; 406; 440
Interval standard reflections measured	2 h, no intensity variation
Total No. of reflections measured; θ range	5409; 65° (940 systematic absences included)
No. of unique reflections; R_{int}	2462; 0.029
No. of observed reflections	1896
Criterion for observed reflections	$I > 3\sigma(I)$
Methods used to solve structure	Patterson and subsequent difference Fourier map
Use of F or F^2 in LS refinement	F
Method of locating H atoms	ΔF map
Method of refining H atoms	Positional parameters with fixed isotropic temperature factors
Weighting scheme	$1/\sigma^2$
Parameters refined	239
Value of R	0.045
Value of wR	0.049
Ratio of max. LS shift to e.s.d. (Δ/σ)	0.05
Max. height in final ΔF map	0.445 e Å ⁻³
Error in an observation of unit weight	1.363
Secondary-extinction coefficient	2.8516 (1) × 10 ⁻⁶ (Zachariasen, 1963)
Source of atomic scattering factors	<i>International Tables for X-ray Crystallography</i> (1974)
Computer used	DEC MicroVAXII
Programs used	VAX/SDP (B. A. Frenz & Associates Inc., 1985); SHELXS86 (Sheldrick, 1986)

O atom of two symmetry-related Schiff bases in a square-planar geometry. The pyridyl N atom is not coordinated to the metal atom.

Introduction. In a previous paper (Atherton, Fenton, Hewson, McLean, Bastida, Romero, Sousa & Castellano, 1988), the multidentate ligand (I) has been studied. In the case of the 4,6-dimethoxy derivative, X-ray diffraction data have shown that the copper(II) complex exhibits a square-planar coordination with the metal coordinated to the phenolate O and imine N atoms. However when R is 3-ethoxy, electronic spectra and EPR data suggested that the copper(II) complex has a different structure. For this reason it was considered worthwhile to determine the structure of this complex using X-ray diffraction techniques.

Table 2. *Positional and equivalent isotropic thermal parameters U_{eq} (Å²)*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.				
	x	y	z	U_{eq}
Cu	0.000	0.000	0.000	0.0344 (3)
O(1)	0.0943 (2)	0.1110 (4)	-0.0732 (2)	0.0453 (1)
O(2)	0.2411 (2)	0.3083 (4)	-0.1301 (2)	0.0500 (2)
N(1)	-0.0892 (2)	-0.0604 (4)	-0.1056 (2)	0.0340 (1)
N(2)	-0.2557 (2)	-0.5946 (5)	-0.1443 (2)	0.0467 (2)
C(1)	-0.3209 (3)	-0.7326 (6)	-0.1298 (3)	0.0537 (2)
C(2)	-0.3210 (3)	-0.8361 (6)	-0.0535 (3)	0.0527 (2)
C(3)	-0.2505 (3)	-0.7970 (6)	0.0115 (3)	0.0520 (2)
C(4)	-0.1818 (3)	-0.6542 (6)	-0.0017 (3)	0.0450 (2)
C(5)	-0.1870 (3)	-0.5553 (5)	-0.0805 (3)	0.0377 (2)
C(6)	-0.1149 (3)	-0.3995 (6)	-0.0983 (3)	0.0460 (2)
C(7)	-0.1662 (3)	-0.2087 (6)	-0.1004 (3)	0.0383 (2)
C(8)	-0.0888 (3)	0.0377 (5)	-0.1775 (2)	0.0363 (2)
C(9)	-0.0138 (3)	0.1718 (5)	-0.2002 (2)	0.0353 (2)
C(10)	-0.0271 (3)	0.2744 (6)	-0.2802 (3)	0.0503 (2)
C(11)	0.0462 (4)	0.3907 (7)	-0.3077 (3)	0.0587 (3)
C(12)	0.1369 (3)	0.4087 (6)	-0.2593 (3)	0.0517 (2)
C(13)	0.1523 (3)	0.3098 (6)	-0.1830 (2)	0.0403 (2)
C(14)	0.0756 (3)	0.1935 (5)	-0.1488 (2)	0.0337 (2)
C(21)	0.3252 (3)	0.4168 (7)	-0.1570 (3)	0.0590 (2)
C(22)	0.4003 (4)	0.3939 (8)	-0.0888 (4)	0.0900 (4)

Table 3. *Selected bond distances (Å) and angles (°)*

Cu—O(1)	1.878 (2)	N(1)—C(7)	1.475 (5)
Cu—N(1)	1.996 (3)	N(1)—C(8)	1.294 (5)
O(1)—C(14)	1.303 (4)	N(2)—C(1)	1.336 (5)
O(2)—C(13)	1.396 (5)	N(2)—C(5)	1.330 (5)
O(2)—C(21)	1.431 (5)		
O(1)—Cu—O(1)	180.1 (6)	Cu—N(1)—C(8)	122.2 (3)
O(1)—Cu—N(1)	90.4 (1)	Cu—O(1)—C(14)	126.9 (2)
O(1)—Cu—N(1)	89.6 (1)	C(8)—C(9)—C(14)	121.4 (3)
N(1)—Cu—N(1)	180.1 (4)	O(1)—C(14)—C(9)	124.5 (3)

Symmetry operator: (i) $-x, -y, -z$.

Experimental. The complex was prepared by an electrochemical procedure similar to that described in the literature (Habeeb, Tuck & Walters, 1978). The parameters for intensity-data collection, structure solution and refinement are listed in Table 1. The final atomic coordinates are given in Table 2, selected bond distances and angles in Table 3.* Figs. 1 and 2 show the numbering scheme and a stereoview, respectively.

Discussion. The Cu^{II} atom is located on a crystallographic symmetry center and is coordinated to the phenolate O and imine N atoms of two symmetry-related ligands. The coordination polyhedron is square-planar within experimental accuracy with Cu—O(1) and Cu—N(1) distances of 1.878 (2) and

* Lists of structure factors, anisotropic thermal parameters, bond distances and angles, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52502 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

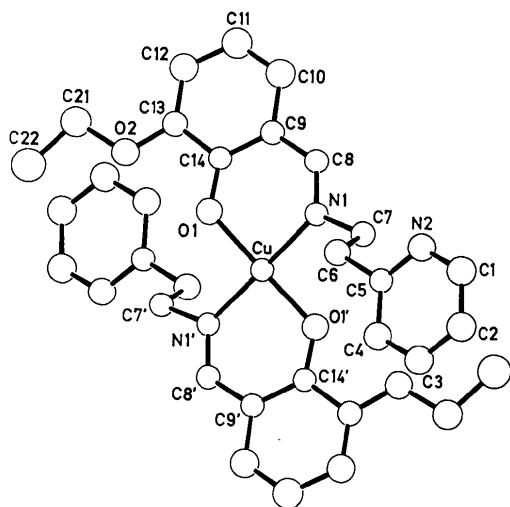


Fig. 1. Plot of the molecule showing the numbering scheme.

1.996 (3) Å, respectively. These bond distances are similar to those found in related salicylidiminato complexes (Lingafelter & Braun, 1966) and also found in bis{*N*-[2-(2-pyridyl)ethyl]-4,6-dimethoxy-salicylideneaminato}copper(II) (Atherton, Fenton, Hewson, McLean, Bastida, Romero, Sousa & Castellano, 1988). In the Schiff base, the N(1)—C(8) bond length of 1.294 (5) Å is in agreement with the value of 1.30 Å proposed for a C—N double bond (Brown, Towns & Trefonas, 1970). The pyridyl N atom is not coordinated to the metal. The bond lengths and angles in this ring are close to the expected values for a heteroaromatic ring.

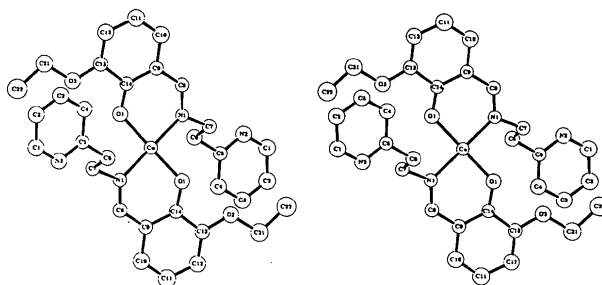


Fig. 2. Stereoscopic view.

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Bis(*N*-isopropyl-5-bromosalicylidiminato)nickel(II)

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Abstract. Bis[4-bromo-2-(isopropyliminomethyl)-phenolato]nickel(II), [Ni(C₁₀H₁₁BrNO)₂], *M*_r = 540.90, triclinic, *P*1̄, *a* = 8.789 (6), *b* = 10.203 (7), *c* =

6.075 (4) Å, $\alpha = 103.92 (5)$, $\beta = 80.23 (5)$, $\gamma = 110.21 (5)^\circ$, *V* = 493.9 (6) Å³, *Z* = 1, *D*_x = 1.818 Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$, $\mu(\text{Mo } K\alpha) = 5.01 \text{ mm}^{-1}$, *F*(000) = 272, *T* = 293 K, *R* = 0.055 for 1189 unique observed reflections [*F* > 3σ(*F*)]. The Ni atom at the symmetry centre is *trans*-planar coor-

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