

coordinations arise from the chelate with the pyridine moiety occupying the sixth coordination position. In the chelate the absolute configuration at C11 and the planarity of the salicylaldimine units give rise to a geometry in which the two phenoxide O ligating atoms and the two azomethine N atoms are *cis* to one another. To the best of our knowledge [Co(sal<sub>2</sub>pen)py] is the first Schiff base complex derived from a terdentate amino acid to be definitely characterized by X-ray analysis.

The geometry of the Co octahedron given in Table 2 suggests that the strain induced by the pentadentate ligand results both in several angular distortions and extension of some bond lengths. The five-membered chelate ring (Co, N2, C11, C12 and O3) lies almost in the plane of the salicylaldimine ring involving N2 and O4, thereby satisfying the steric and electronic requirements of this system. The six-membered chelate rings assume envelope conformations with different degrees of puckering depending on the Co—N and Co—O bond lengths. Thus, the ring defined by Co, N2, C13, C14, C15 and O4 with the shorter bond lengths, Co—N2 = 1.870 (3) and Co—O4 = 1.877 (2) Å, is more puckered than that defined by Co, N1, C7, C6, C1 and O1 with Co—N1 = 1.937 (4) and Co—O1 = 1.894 (2) Å. The conformation of the seven-membered ring (Co, N2, C11, C10, C9, C8 and N1) can be best described as a twist-boat (Hendrickson, 1961), whilst that of the eight-membered ring (Co, N1, C8, C9, C10, C11, C12 and O3) approximately as that of a deformed boat (Borgen & Dale, 1970). The crystal packing is shown in Fig. 2.

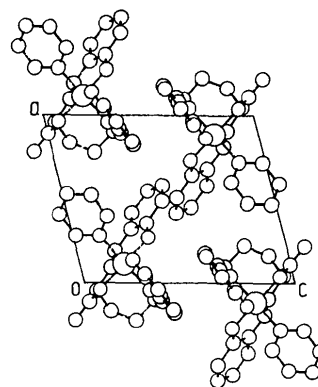


Fig. 2. Packing scheme.

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### Bis(3,5-dibromo-*N*-*o*-tolylsalicylaldiminato)copper(II)\*

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**Abstract.** [Cu(C<sub>14</sub>H<sub>10</sub>Br<sub>2</sub>NO)<sub>2</sub>], *M<sub>r</sub>* = 799.6, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 17.866 (5), *b* = 21.963 (6), *c* =

\* IUPAC name: bis(3,5-dibromo-*N*-*o*-tolylsalicylideneaminato)-copper(II).

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14.128 (4) Å, β = 93.16 (2)° *V* = 5535 (2) Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.919 Mg m<sup>-3</sup>, μ = 6.54 mm<sup>-1</sup>, λ(Mo *K*α) = 0.71069 Å, *F*(000) = 3096, *T* = 293 K, final *R* = 0.076 for 5411 unique observed reflections with *F* > 3σ(*F*). The two crystallographically independent molecules have a nearly identical distorted planar coordination

about the Cu atoms. The angles between the two coordination planes defined by O1—Cu—N1 and O2—Cu—N2 are 33.4 and 33.7° for molecules 1 and 2, respectively. The mean N—Cu—N and N—Cu—O bond angles are 155.0 (4) and 92.5 (12)°, the mean Cu—N and Cu—O bond lengths are 1.963 (12) and 1.885 (3) Å, respectively. No interaction is possible between Cu atoms.

**Introduction.** Schiff-base complexes containing different metal atoms such as Cu, Ni, Co and Pd have been investigated in great detail. Copper(II) Schiff-base complexes are of particular interest in bio-inorganic chemistry because these complexes have been suggested as inorganic models for copper proteins (Osterberg, 1974; Patterson & Holm, 1975).

Structural studies have shown that the variation of the size and nature of the substituent of the salicylaldehyde ligand could be responsible for changes in the coordination geometry about the metal atoms. It has been reported that copper complexes with an *N*-substituted salicylaldehyde ligand can occur in a planar or a tetrahedral configuration. The *N*-substituted salicylaldehyde complex of copper(II) which has a perfectly planar molecular structure is the  $\alpha$ -form of bis(*N*-methylsalicylaldehyde)copper(II) (Lingafelter, Simmons, Morosin, Scheringer & Freiburg, 1961). Bis(*N*-*tert*-butylsalicylaldehyde)copper(II) is markedly nonplanar and the stereochemistry of the Cu atom is a flattened tetrahedron (Cheeseman, Hall & Waters, 1966). This paper describes the structure of the related bis(3,5-dibromo-*N*-*o*-tolylsalicylaldehyde)copper(II).

**Experimental.** A shapeless and faceless green crystal of dimensions 0.5 × 0.5 × 0.3 mm was used on a Nicolet *R3m/V* diffractometer, graphite-monochromatized Mo  $K\alpha$  radiation, unit-cell dimensions from 25 centred reflections  $8 < \theta < 14^\circ$ . Wyckoff scan used for data collection of 9805 unique reflections of which 5411 were observed with  $F > 3\sigma(F)$ . According to the prescan intensity, the Wyckoff scan speed ranged from 1.5 to 19.5° min<sup>-1</sup>. Absorption correction based on  $\psi$  scan of 11 reflections. Diffraction intensities were measured up to 0.59 Å<sup>-1</sup> in the index range  $h = 0 \rightarrow 21$ ,  $k = 0 \rightarrow 26$  and  $l = -16 \rightarrow 16$ . Three standard reflections (800, 656 and 4,10,3) varied less than 3.1% over 214.9 h of data collection. Structure solved by direct phase determination,  $E_{\min} = 1.6$ . Full-matrix least squares minimized  $w(\Delta F)^2$ ; H-atom positions calculated geometrically and considered isotropically with  $U = 1.2U_C$  of bonded C. All other atoms refined anisotropically for 667 variables.  $R = 0.076$ ,  $wR = 0.052$ ,  $S = 1.50$ , where  $w^{-1} = \sigma^2(F)$ . Final  $(\Delta/\sigma)_{\max} = 0.065$ ,  $\Delta\rho_{\max} = 0.8$  and  $\Delta\rho_{\min} = -0.9$  e Å<sup>-3</sup> on final difference Fourier map.

Atomic scattering factors taken from *SHELXTL-Plus* (Sheldrick, 1988).

**Discussion.** The final positional and equivalent isotropic thermal parameters of all non-H atoms are listed in Table 1. Selected interatomic distances and bond angles are given in Table 2.\*

The crystal can be described as consisting of discrete molecules with two symmetry-independent molecules in the unit cell. The two independent molecules have essentially identical structures with each Cu atom fourfold coordinated. Fig. 1 shows the atomic arrangement in the molecule together with the atom-labelling sequence.

The environment around the Cu atom is best visualized as a distorted-planar to tetrahedral coordination. The distortion involves the twisting of the ligand and planes in opposite directions. The angles between the two coordination planes defined by O1—Cu—N1 and O2—Cu—N2 are 33.4 and 33.7° for molecules 1 and 2, respectively. These angles, in addition to the bond angles at the Cu atoms (Table 2), show the coordination configuration to be closer to planar than to tetrahedral. As expected, the introduction of a bulky *o*-tolyl substituent at the *N* atom distorts the coordination configuration from planar to tetrahedral. However, the bromine substituents in the benzene rings restore some planarity, as expected from the comparison with the related isomorphous nickel complex bis(*N*-isopropyl-5-bromosalicylaldehyde)nickel(II) (Manriquez, Vargas, Costamagna, von Schnering & Peters, 1990).

The mean planes through the benzene rings, chelate ring and coordination plane were calculated and are given in Table 3. It is apparent that the ligands are all very nearly planar in each independent molecule. The N—C bonds were found to have the largest deviations from planarity (Table 2).

The bond lengths and angles are, where applicable, very similar to those observed in the bis(*N*-substituted-salicylaldehyde)copper(II) (Lingafelter & Braun, 1966). The mean values of 1.963 (12) and 1.885 (5) Å for the Cu—N and Cu—O bond lengths, respectively, are similar to those of 1.942 (34) and 1.890 (22) Å in bis(*N*-ethylsalicylaldehyde)copper(II) (Baker, Clark, Hall & Waters, 1967), and to 1.980 and 1.880 Å in bis(*N*-isopropylsalicylaldehyde)copper(II) (Orioli & Sacconi, 1966), both of which were found to be distorted tetrahedra. The steric effect of the tolyl group causes an increase in

\* Lists of bond lengths and angles, atomic coordinates of H atoms, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53001 (44 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Refined atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters with *e.s.d.*'s in parentheses

Molecule 1	x	y	z	$U_{eq}^*(\text{\AA}^2)$
Cu(1)	8125 (1)	2591 (1)	7273 (1)	51 (1)
Br(3)	5564 (1)	2069 (1)	7864 (1)	66 (1)
Br(5)	4710 (1)	4492 (1)	7124 (1)	65 (1)
Br(10)	10703 (1)	3152 (1)	7912 (1)	83 (1)
Br(12)	11561 (1)	741 (1)	7093 (1)	70 (1)
O(1)	7097 (4)	2564 (3)	7513 (5)	54 (3)
N(1)	8103 (5)	3457 (4)	6945 (7)	51 (4)
C(1)	6776 (6)	3606 (5)	7210 (8)	43 (4)
O(2)	9166 (4)	2621 (3)	7565 (5)	57 (3)
N(2)	8137 (5)	1713 (4)	6992 (7)	53 (4)
C(2)	6614 (6)	2986 (5)	7432 (8)	44 (4)
C(3)	5832 (6)	2873 (5)	7554 (8)	44 (4)
C(4)	5297 (6)	3300 (5)	7466 (8)	47 (4)
C(5)	5474 (6)	3895 (5)	7251 (8)	46 (4)
C(6)	6195 (6)	4044 (5)	7108 (7)	50 (5)
C(7)	7515 (6)	3789 (5)	6989 (8)	48 (4)
C(8)	9491 (6)	1588 (5)	7227 (8)	44 (4)
C(9)	9658 (6)	2204 (6)	7452 (8)	47 (5)
C(10)	10433 (6)	2338 (5)	7603 (8)	52 (5)
C(11)	10986 (6)	1915 (6)	7491 (8)	57 (5)
C(12)	10801 (7)	1325 (6)	7266 (8)	50 (5)
C(13)	10062 (6)	1158 (5)	7137 (8)	50 (5)
C(14)	8734 (6)	1392 (5)	7032 (7)	45 (4)
C(15)	8770 (7)	3779 (6)	6687 (11)	60 (6)
C(16)	9060 (6)	4210 (6)	7292 (13)	96 (8)
C(17)	9690 (7)	4533 (7)	7050 (15)	110 (9)
C(18)	10034 (9)	4414 (8)	6240 (15)	113 (10)
C(19)	9748 (8)	3971 (8)	5672 (12)	100 (8)
C(20)	9118 (8)	3634 (7)	5862 (12)	81 (7)
C(21)	8834 (7)	3160 (7)	5227 (10)	99 (7)
C(22)	7440 (6)	1386 (5)	6788 (10)	52 (5)
C(23)	7197 (7)	1008 (6)	7475 (11)	70 (6)
C(24)	6525 (8)	717 (6)	7314 (12)	87 (7)
C(25)	6109 (9)	788 (7)	6469 (13)	92 (8)
C(26)	6375 (8)	1155 (6)	5803 (11)	78 (7)
C(27)	7056 (8)	1479 (6)	5953 (10)	60 (6)
C(28)	7326 (7)	1890 (6)	5205 (10)	96 (7)

Molecule 2	x	y	z	$U_{eq}^*(\text{\AA}^2)$
Cu(2)	-3001 (1)	7556 (1)	4874 (1)	52 (1)
Br(3)	-2241 (1)	9614 (1)	5689 (1)	73 (1)
Br(5)	766 (1)	8946 (1)	5321 (1)	77 (1)
Br(10)	-3756 (1)	5518 (1)	5587 (1)	71 (1)
Br(12)	-6775 (1)	6261 (1)	5316 (1)	73 (1)
O(1)	-2504 (4)	8299 (3)	5156 (6)	59 (3)
N(1)	-2065 (5)	7119 (4)	4630 (6)	47 (4)
C(1)	-1250 (6)	7979 (5)	4963 (7)	44 (4)
O(2)	-3500 (4)	6829 (3)	5148 (6)	56 (3)
N(2)	-3927 (5)	7991 (4)	4524 (7)	52 (4)
C(2)	-1799 (6)	8415 (5)	5182 (7)	43 (4)
C(3)	-1536 (7)	9012 (5)	5428 (8)	57 (5)
C(4)	-793 (6)	9157 (5)	5467 (8)	53 (5)
C(5)	-276 (6)	8718 (6)	5252 (8)	52 (5)
C(6)	-477 (6)	8145 (5)	5006 (8)	49 (5)
C(7)	-1415 (6)	7368 (5)	4718 (8)	51 (5)
C(8)	-4768 (6)	7160 (6)	4930 (8)	50 (5)
C(9)	-4215 (7)	6721 (6)	5149 (8)	49 (5)
C(10)	-4474 (7)	6138 (5)	5377 (8)	52 (5)
C(11)	-5224 (7)	6002 (6)	5411 (8)	54 (5)
C(12)	-5744 (6)	6453 (6)	5218 (8)	53 (5)
C(13)	-5535 (6)	7016 (6)	4970 (8)	53 (5)
C(14)	-4583 (7)	7773 (5)	4611 (8)	52 (5)
C(15)	-2079 (7)	6466 (6)	4425 (10)	58 (5)
C(16)	-1723 (7)	6080 (6)	5085 (10)	65 (6)
C(17)	-1759 (8)	5456 (6)	4908 (12)	80 (7)
C(18)	-2121 (9)	5227 (8)	4118 (14)	99 (9)
C(19)	-2471 (9)	5621 (7)	3493 (13)	96 (8)
C(20)	-2457 (7)	6239 (7)	3632 (11)	69 (6)
C(21)	-2849 (8)	6651 (6)	2952 (10)	99 (7)
C(22)	-3942 (7)	8622 (7)	4242 (11)	63 (6)
C(23)	-4163 (8)	9073 (6)	4871 (14)	103 (8)
C(24)	-4152 (11)	9653 (10)	4539 (19)	148 (14)
C(25)	-3842 (10)	9804 (10)	3699 (21)	144 (15)
C(26)	-3626 (9)	9376 (9)	3127 (17)	135 (11)
C(27)	-3653 (8)	8783 (7)	3412 (16)	94 (8)
C(28)	-3392 (9)	8343 (8)	2794 (11)	126 (9)

\* Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

Table 2. Selected interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

	Mol. 1	Mol. 2	Mol. 1	Mol. 2	
Cu1—N1	1.958 (9)	1.974 (9)	N2—C22	1.454 (14)	1.441 (17)
Cu1—N2	1.969 (9)	1.951 (9)	O1—C2	1.268 (13)	1.283 (14)
Cu1—O1	1.886 (7)	1.889 (7)	O2—C9	1.285 (14)	1.299 (14)
Cu1—O2	1.885 (7)	1.881 (7)	Br3—C3	1.886 (11)	1.877 (12)
N1—C7	1.283 (14)	1.284 (14)	Br5—C5	1.894 (11)	1.924 (11)
N1—C15	1.448 (15)	1.462 (16)	Br10—C10	1.896 (11)	1.883 (12)
N2—C14	1.278 (14)	1.278 (15)	Br12—C12	1.894 (12)	1.901 (12)
N1—Cu1—N2	154.7 (4)	155.3 (4)	N2—Cu1—O1	91.5 (3)	90.7 (3)
N1—Cu1—O1	93.8 (3)	93.7 (3)	N2—Cu1—O2	93.2 (3)	93.6 (3)
N1—Cu1—O2	91.4 (3)	92.2 (3)	O1—Cu1—O2	157.1 (3)	155.9 (4)

Table 3. Least-squares mean planes

(a) Coefficients of equations $Ax + By + Cz = d$					
Plane	A	B	C	d	
Coordination					
1 O1—Cu—N1	Mol. 1	2.979	5.149	13.381	13.4868
2	Mol. 2	0.857	-5.717	13.556	2.0349
3 O2—Cu—N2	Mol. 1	-3.696	-4.432	13.666	5.7880
4	Mol. 2	-2.039	6.477	13.472	12.0721
Chelate rings					
5 O1—C1—C2—C7—N1	Mol. 1	3.031	4.664	13.444	13.4216
6	Mol. 2	0.558	-5.842	13.567	2.0089
7 O2—C8—C9—C14—N2	Mol. 1	-2.599	-5.021	13.693	6.6276
8	Mol. 2	-0.561	6.848	13.421	11.7707
Benzene rings					
9 C1—C2—C3—C4—C5—C6	Mol. 1	1.461	4.653	13.675	12.5206
10	Mol. 2	0.548	-5.968	13.545	1.8951
11 C8—C9—C10—C11—C12—C13	Mol. 1	-1.413	-4.895	13.768	7.8308
12	Mol. 2	-0.415	5.820	13.617	11.0848

(b) Angles ( $^\circ$ ) between the least-squares mean planes

	Mol. 1	Mol. 2	
1-3	33.4	2-4	33.7
5-7	31.4	6-8	33.8
1-5	1.3	2-6	1.1
3-7	4.0	4-8	4.9
9-11	26.8	10-12	31.3
1-9	5.1	2-10	1.2
3-11	7.4	4-12	5.6
5-9	5.1	6-10	0.3
7-11	3.9	8-12	2.8

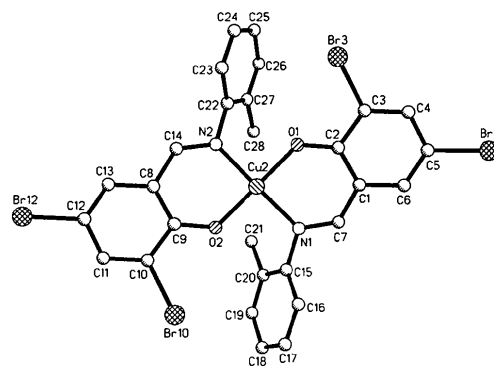


Fig. 1. A perspective drawing of the complex showing the atom labelling.

the length of the metal–nitrogen bond by 0.078  $\text{\AA}$  over that of the metal–oxygen bond. In bis(salicylal-dimino)copper(II) both  $M-N$  and  $M-O$  bond lengths are 1.908  $\text{\AA}$  (Baker, Hall & Waters, 1966).

There are no unusual intermolecular contacts within the crystal lattice. The closest intermolecular contact between Cu atoms is 4.049 Å.

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## Structure of the Copper(II) Complex of the Macrocycle 1-Thia-4,7-diazacyclononane with Two Pendant Pyridine Arms

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**Abstract.** [4,7-Bis(2-pyridyl- $\kappa N$ -methyl)-1-thia-4,7-diazacyclononane- $\kappa^2 N, N', \kappa S$ ]copper(II) diperchlorate, [Cu(C<sub>18</sub>H<sub>24</sub>N<sub>4</sub>S)](ClO<sub>4</sub>)<sub>2</sub>,  $M_r = 590.9$ , orthorhombic, *Pnma*,  $a = 17.30$  (1),  $b = 11.937$  (7),  $c = 11.179$  (3) Å,  $V = 2308.8$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.68$ ,  $D_x = 1.73$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 13.2$  cm<sup>-1</sup>,  $F(000) = 1212$ ,  $T = 294$  K,  $R = 0.068$  for 1568 observed reflections. The copper coordination is square pyramidal with the S atom in an axial site. The Cu—S bond [2.496 (8) Å] is rather short. The basal plane is trapezoidally distorted. The ligand adopts a strained conformation to match the coordination properties of Cu<sup>II</sup>.

**Introduction.** Recently we reported the structure of several complexes of the mixed donor atom macrocyclic ligand 1-thia-4,7-diazacyclononane (L) (Hoffmann, Steinhoff & Mattes, 1987; Hoffmann & Mattes, 1988; Hoffman, Hermes & Mattes, 1988; Hoffmann & Mattes, 1989). In the course of our studies derivatives of this ligand with pendant amine and pyridyl arms were prepared and their complex chemistry investigated. During the progress of our work the preparation of 4,7-bis(3-aminopropyl)-1-thia-4,7-diazacyclononane and its copper(II) complex (Fortier & McAuley, 1989), and the structure of the copper(II) complex of dimethyl(1-thia-4,7-diaza-4,7-cyclononane)diacetate (Lac<sub>2</sub>) (Craig, Parker &

Ferguson, 1989) have been reported. In this paper we describe the structure of the copper(II) complex of the macrocycle 1-thia-4,7-diazacyclononane with two pendant pyridine arms.

**Experimental.** The ligand 4,7-bis(2-methylpyridyl)-1-thia-4,7-diaza-cyclononane (Lpy<sub>2</sub>) was prepared by reaction of 1-thia-4,7-diazacyclononane dihydrobromide (Hoffmann, Steinhoff & Mattes, 1987) and 2-picoly chloride in sodium hydroxide solution, evaporation of the solvent under reduced pressure and extraction with chloroform and ligroin. Dark-blue prismatic crystals of [4,7-bis(2-methylpyridyl)-1-thia-4,7-diazacyclononane]copper(II) diperchlorate were grown by slow evaporation of an aqueous solution containing the ligand, CuCl<sub>2</sub> and NaClO<sub>4</sub>.

Crystal sealed in Lindemann capillary and centered on an Enraf-Nonius CAD-4 diffractometer. Crystal size 0.8 × 0.1 × 0.1 mm; graphite-monochromated Mo  $K\alpha$  radiation. Cell dimensions and orientation matrix determined from 25 reflections ( $19.6 \leq 2\theta \leq 28.5^\circ$ ); intensities measured with indices  $h$  0 to 21,  $k$  0 to 14 and  $l$  0 to 4  $\leq 2\theta \leq 54^\circ$ ;  $\omega$ - $2\theta$  scans, three standard reflections monitored every four hours showed no decay. 2921 reflections measured, 2640 were unique and 1570 with  $I \geq 1.96\sigma(I)$  were labelled observed and used in structure solution and refinement. Data were corrected for Lorentz,