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## Structures of Two Complexes of Copper(II) Chloride and 1,4-Oxathiane. The Two-Dimensional Polymers Poly[di- $\mu$ -chloro- $\mu$ -(1,4-oxathiane-*O,S*)-copper(II)], $[\text{Cu}_3(\text{C}_4\text{H}_8\text{OS})_2\text{Cl}_6]_n$ , and Poly[dichlorobis- $\mu$ -(1,4-oxathiane-*O,S*)-copper(II)], $[\text{Cu}(\text{C}_4\text{H}_8\text{OS})_2\text{Cl}_2]_n$

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**Abstract.** (I)  $[\text{CuCl}_2]_3[\text{C}_4\text{H}_8\text{OS}]_2$ ,  $M_r = 611.68$ , triclinic,  $P\bar{1}$ ,  $a = 9.375$  (5),  $b = 7.370$  (5),  $c = 7.821$  (4) Å,  $\alpha = 112.17$  (12),  $\beta = 112.56$  (11),  $\gamma = 76.18$  (5)°,  $U = 459.3$  Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 2.212$  Mg m<sup>-3</sup>; Cu  $K\alpha$  radiation,  $\lambda = 1.5418$  Å,  $\mu = 13.6$  mm<sup>-1</sup>,  $F(000) = 301$ ,  $T = 293$  K;  $R = 0.060$  for 1077 reflexions. (II)  $[\text{CuCl}_2][\text{C}_4\text{H}_8\text{OS}]_2$ ,  $M_r = 342.80$ , orthorhombic, *Abma* (a non-standard setting of *Cmca*, No. 64),  $a = 10.86$  (1),  $b = 12.63$  (1),  $c = 9.64$  (1) Å,  $U = 1322.2$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.723$  Mg m<sup>-3</sup>; Cu  $K\alpha$  radiation,  $\lambda = 1.5418$  Å,  $\mu = 8.5$  mm<sup>-1</sup>,  $F(000) = 700$ ,  $T = 293$  K;  $R = 0.047$  for 390 reflexions. In (I) stepped chains  $(\text{CuCl}_2)_n$  are crosslinked by (S...O)-bridging 1,4-oxathiane ligands to give five-coordinate  $(\text{CuCl}_4\text{S})$  and six-coordinate  $(\text{CuCl}_4\text{O}_2)$  environments. In (II) linear  $\text{CuCl}_2$  units are joined into layers normal to **b** by (S...O)-bridges to give a six-coordinate environment with no chloride bridges.

**Introduction.** There has been renewed interest in the structures of metal complexes of thioether ligands (Murray & Hartley, 1981; Walton, 1980). Until recently, 1,4-oxathiane (tx) was known only as a monodentate ligand giving monomeric complexes (McEwen & Sim, 1967; Barnes, Hunter & Lown, 1977). This ligand is now known to give polymeric complexes with Cu<sup>I</sup> and Ag<sup>I</sup> using S—, O— and

S...O—bridges (Barnes & Paton, 1982, 1983; Barnes, Blyth & Paton, 1982). The reaction of CuCl with tx has been reinvestigated. In addition to green  $\text{CuCl}_2 \cdot 2\text{tx}$  (II) (Walton, 1966), red  $(\text{CuCl}_2)_3 \cdot 2\text{tx}$  (I) and white  $\text{CuCl} \cdot \text{tx}$  (III) have been obtained. (III) is not isomorphous with  $\text{CuBr} \cdot \text{tx}$  (Barnes & Paton, 1982) but no crystals suitable for structure determination have been obtained.

**Experimental.** 1,4-Oxathiane vapour was allowed to diffuse into anhydrous  $\text{CuCl}_2$  in ethanol at room temperature. The usual product was (II) but microscopic examination of some batches revealed red plates (I) and occasionally a few white plates (III). The compounds were separated under the microscope. They deteriorate in air but could be stored indefinitely in sealed vials when dry.

The structures of (I) and (II) were determined using intensities from equi-inclination Weissenberg photographs scanned by the SERC Microdensitometer Service, Daresbury Laboratory. All calculations were performed on the Dundee University DEC 10 computer using the *SHELX* 76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978) program packages. Atomic scattering parameters were from *International Tables for X-ray Crystallography* (1974). All crystals were mounted in Lindemann-glass capillaries.

*Compound (I).* Data were collected for layers (0–8)*kl* and *h*(0–5)*l*. The  $\gamma$  angle was measured by

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comparison of upper-level Weissenberg photographs on the *a* and *b* axes with computer-simulated patterns produced by the local program *WEISS* (Barnes, Low & Young, 1980) since the crystal habit precluded mounting a crystal in the *c* direction in a capillary.

The structure was solved by the direct-methods routine *TANG*. The fourth most likely *E* map in a calculation in *P1* showed a recognizable Cu<sub>2</sub>Cl<sub>2</sub> ring. After some difficulty due to false symmetry one Cu atom was placed at the origin with the second unique Cu on a general position close to the *a* axis in space group *P1*. A model with Cu, Cl and S atoms then refined to *R* 0.24 at which point an electron density map revealed the remaining non-hydrogen atoms. Absorption corrections were applied (crystal dimensions 0.15 × 0.15 × 0.09 mm). H atoms were included on calculated positions with individual isotropic thermal parameters; anisotropic thermal parameters were used for all non-hydrogen atoms. Final refinement (minimizing  $\sum w|F_o - |F_c||^2$ ): 107 refined parameters, *wR* = 0.066, *w* = 1/[ $\sigma^2(F) + 0.1000F^2$ ], mean shift/e.s.d. = 0.029, max. shift/e.s.d. = 0.247, max. difference peak = 1.1 e Å<sup>-3</sup> (close to Cu).

**Compound (II).** Data were collected for layers *h*(0–2)*l* and for layers 0–12 of a crystal mounted on the *ac* diagonal. Interpretation of the Patterson function located the Cu atom at the origin with two unique Cl atoms along the *b* axis and the S and O atoms on general positions close to the *ac* plane. A structure factor calculation on this model gave *R* 0.21 and an electron density map revealed the four unique C atoms. The structure refined to *R* 0.13 with all atoms isotropic. An attempt to convert to anisotropic thermal parameters gave no improvement in *R* and unsatisfactory dimensions for the oxathiane molecule. Anisotropic refinement continued normally when the bond lengths in the oxathiane molecule were constrained to C–C 1.54, C–O 1.45 and C–S 1.81 Å. This procedure has been used in several recent structures of tx complexes (Barnes, Blyth & Paton, 1982; Barnes & Paton, 1982). The constraints were released at *R* 0.07 and isotropic H atoms were introduced on calculated positions using a single refined thermal parameter for all the H atoms. Final refinement (minimizing  $\sum w|F_o - |F_c||^2$ ): 48 refined parameters, *wR* = 0.053, *w* = 2.4264/[ $\sigma^2(F) + 0.001273F^2$ ], mean shift/e.s.d. = 0.078, max. shift/e.s.d. = 0.28, max. difference peak = 0.58 e Å<sup>-3</sup> (close to Cu).\*

**Discussion.** Atomic coordinates for (I) are given in Table 1 with interatomic distances and angles in Table 2. There are chains of Cl-bridged Cu atoms in the *a*

Table 1. *Atomic coordinates* (× 10<sup>4</sup>) *and equivalent isotropic thermal parameters* (× 10<sup>3</sup>) *with e.s.d.'s in parentheses for the non-hydrogen atoms of (I)*

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub> (Å <sup>2</sup> )
Cu(1)	6519 (1)	1338 (2)	810 (2)	30 (1)
Cu(2)	0	0	0	36 (1)
Cl(1)	5227 (2)	341 (3)	2208 (2)	33 (1)
Cl(2)	7757 (2)	2191 (3)	-272 (2)	32 (1)
Cl(3)	8976 (2)	-828 (3)	1758 (3)	37 (1)
S(11)	6454 (2)	4379 (3)	3255 (2)	31 (1)
O(14)	8857 (7)	7017 (8)	6832 (8)	36 (2)
C(12)	7596 (10)	6036 (12)	3285 (11)	37 (2)
C(13)	7921 (10)	7673 (12)	5202 (11)	37 (2)
C(15)	8153 (11)	5718 (14)	7158 (12)	39 (3)
C(16)	7785 (10)	3827 (13)	5447 (11)	36 (2)

Table 2. *Interatomic distances* (Å) *and angles* (°), *with e.s.d.'s in parentheses in compounds (I), (II) and (IV)*

	(I)	(IV)	(II)
<i>(a) Bonds to copper</i>			
Cu(1)–Cl(1)	2.292 (2)	2.302	2.254 (3)
Cu(1)–Cl(2)	2.275 (2)	2.302	
Cu(1)–Cl(3)	2.496 (2)	2.302	
Cu(1)–S(11)	2.339 (2)	2.341, 2.845*	2.372 (2)
Cu(1)–Cl(1)'	2.363 (2)	—	
Cu(1)–O(14)'			2.618 (6)
Cu(2)–Cl(2)	2.347 (2)	2.296	
Cu(2)–Cl(3)	2.261 (2)	2.296	
Cu(2)–O(14)	2.664 (6)	2.436	
Cl(1)–Cu(1)–Cl(2)	176.9 (1)	170.5	180.0
Cl(1)–Cu(1)–Cl(3)	95.1 (1)	92.9	
Cl(2)–Cu(1)–Cl(3)	84.8 (1)	86.3	
Cl(1)–Cu(1)–S(11)	85.7 (1)	94.6, 95.5*	90.0
Cl(2)–Cu(1)–S(11)	97.2 (1)		
Cl(3)–Cu(1)–S(11)	112.1 (1)	94.9	
Cl(1)–Cu(1)–Cl(1)'	85.7 (1)		
Cl(2)–Cu(1)–Cl(1)'	91.4 (1)		
Cl(3)–Cu(1)–Cl(1)'	111.1 (1)	92.9	
S(11)–Cu(1)–Cl(1)'	136.5 (1)	94.6	
S(11)–Cu(1)–O(14)'			90.1 (4)
Cl(1)–Cu(1)–O(14)'			90.0
Cl(2)–Cu(2)–Cl(3)	88.7 (1)	86.6	
Cl(2)–Cu(2)–O(14)	93.6 (1)	83.9	
Cl(3)–Cu(2)–O(14)	91.5 (1)	96.1	
Cl(2)–Cu(2)–Cl(3)'	91.3 (1)		
O(11)–Cu(1)–O(14)	—	166.1	
C(12)–S(11)–Cu(1)	112.2 (3)		110.4 (2)
C(13)–O(14)–Cu(2)	111.8 (4)		
C(16)–S(11)–Cu(1)	102.7 (3)		
C(15)–O(14)–Cu(2)	114.3 (4)		
C(13)–O(14)–Cu(1)'			123.0 (5)
<i>(b) Oxathiane</i>			
S(11)–C(12)	1.795 (8)		1.820 (8)
C(12)–C(13)	1.502 (11)		1.516 (10)
C(13)–O(14)	1.426 (9)		1.427 (8)
O(14)–C(15)	1.436 (10)		
C(15)–C(16)	1.523 (12)		
C(16)–S(11)	1.818 (8)		
S(11)–C(12)–C(13)	109.8 (5)		110.3 (5)
C(13)–O(14)–C(15)	114.1 (6)		114.1 (6)
O(14)–C(15)–C(16)	113.5 (6)		
C(15)–C(16)–S(11)	110.7 (6)		
C(16)–S(11)–C(12)	97.3 (4)		97.3 (5)

\* Lists of structure factors, anisotropic thermal parameters and H atom coordinates for both compounds have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38334 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

\* Distances and angles to O(11) and O(14) respectively.

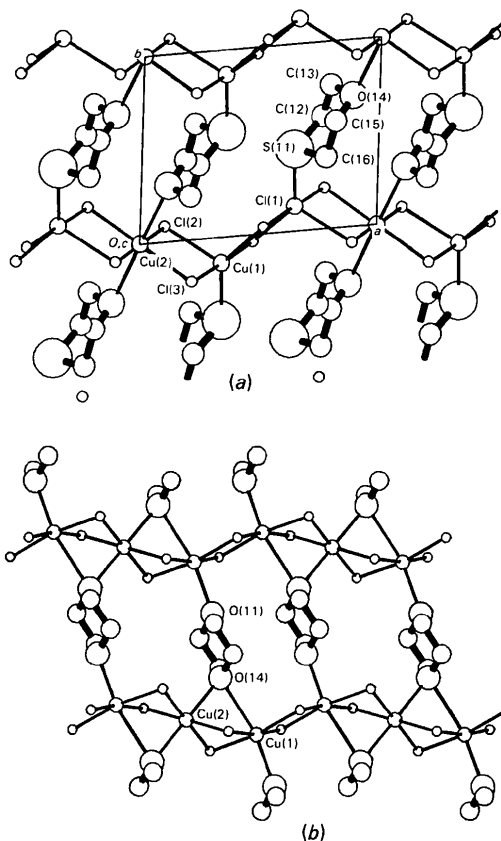
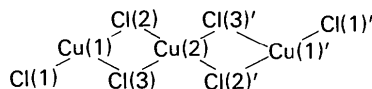


Fig. 1. (a) Perspective view of  $(\text{CuCl}_2)_3 \cdot 2(1,4\text{-oxathiane})$ , seen perpendicular to the  $\text{CuCl}_2$  chain. (b) Perspective view of  $(\text{CuCl}_2)_3 \cdot 2(1,4\text{-dioxane})$ , seen perpendicular to the  $\text{CuCl}_2$  chain.

direction (Fig. 1a). Cu(2) is octahedrally coordinated with four short Cu—Cl bonds and two long Cu—O bonds. Cu(1) has trigonal bipyramidal coordination, with Cl(1) and Cl(2) axial, and Cl(1)', Cl(3) and S(11) equatorial. As in  $\text{CuCl}_3^{2-}$  (Schlueter, Jacobsen & Rundle, 1966) (values in parentheses) the axial Cu—Cl bonds at Cu(1) are shorter [2.275 (2), 2.292 (2) (2.30) Å] than the equatorial bonds [2.363 (2), 2.496 (2) (2.39) Å]. The alternation of the Cu—Cl distances at Cu(2) is also seen in the planar  $(\text{CuCl}_4)$  fragments in  $\text{CsCuCl}_3$  (2.28, 2.36 Å) (Raymond, Meek & Ibers, 1968).

The arrangement of  $(\text{CuCl}_2)_n$  ribbons linked into sheets by the organic ligands is generally similar to that in  $(\text{CuCl}_2)_3 \cdot 2(1,4\text{-dioxane})$  (IV) (Fig. 1b) (Barnes & Weakley, 1977). The axial length along the  $\text{CuCl}$  chain is 9.375 (5) Å in (I) compared with 9.208 Å in (IV). In the direction of the interchain bonds the  $(\text{Cu} \cdots \text{Cu})$  distance is 7.821 Å (II) compared with 7.880 Å (IV).

In (I) the whole fragment:



is planar with the independent  $(\text{Cu} \cdots \text{Cu})$  distances [ $\text{Cu}(1) \cdots \text{Cu}(2)$  and  $\text{Cu}(1) \cdots \text{Cu}(1')$ ] essentially equal [3.406 (2) and 3.412 (2) Å]. The  $\text{Cu}(2) \cdots \text{Cu}(1) \cdots \text{Cu}(1')$  angle is  $130.0 (1)^\circ$ . In (IV) the  $\text{CuCl}_2$  chain is twisted between each pair of Cu atoms giving an angle of  $49.4^\circ$  between the unique  $\text{CuCl}_4$  planes and two distinct  $(\text{Cu} \cdots \text{Cu})$  distances (3.120, 3.358 Å).

Bond lengths and angles for (IV) are included in Table 2 for comparison. Cu(1) adopts a good approximation to trigonal bipyramidal geometry in (II), with no possible contact to another ligand atom whereas in (IV) the twisted chain brings O(14) into contact with Cu(1) to give a very distorted octahedral geometry.

Atomic coordinates for (II) are given in Table 3 with bond lengths and angles in Table 2. Fig. 2 shows that each Cu atom has *trans* terminal Cl ligands. The polymer is formed by four oxathiane ligands, each bridging to a different Cu atom to give a layer structure. These ligands are arranged in *trans* pairs, two S-bonded and two O-bonded to each Cu atom. The Cu—S bond in (II) is 0.03 Å longer than in (I) whereas the Cu—O bond is 0.05 Å shorter. The Cu—S bond lies in the direction of the equatorial lone pair of the S atom in both (I) and (II). The Cu—O vector in (II) approximately bisects the axial and equatorial directions at O, as in many complexes of metal ions with ethers. In (I) the Cu—O vector is close to the direction of the equatorial lone pair.

Table 3. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\times 10^3$ ) with *e.s.d.*'s in parentheses for the non-hydrogen atoms of (II)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$ (Å <sup>2</sup> )
Cu(1)	0	0	0	20 (1)
Cl(1)	0	1785 (2)	0	25 (1)
S(11)	417 (2)	0	2415 (2)	23 (1)
C(12)	1453 (8)	-1081 (5)	2857 (7)	30 (2)
C(13)	1933 (7)	-948 (6)	4324 (8)	32 (2)
O(14)	2634 (6)	0	4477 (7)	27 (1)

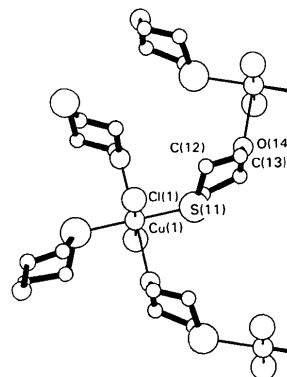


Fig. 2. Perspective view of  $(\text{CuCl}_2)_2 \cdot 2(1,4\text{-oxathiane})$ .

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(*N*-{2-[(2-Aminoethyl)amino]ethyl}salicylideneaminato-*O,N,N',N''*)aquacopper(II) Hexafluorophosphate, [Cu(C<sub>11</sub>H<sub>16</sub>N<sub>3</sub>O)(H<sub>2</sub>O)]PF<sub>6</sub>

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**Abstract.**  $M_r = 432.8$ , monoclinic,  $P2_1/c$ ,  $a = 10.182$  (2),  $b = 14.863$  (3),  $c = 11.188$  (2) Å,  $\beta = 98.6$  (1)°,  $U = 1674.1$  (7) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.717$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $F(000) = 876$ , final  $R = 0.085$  and  $R_w = 0.089$  for 1406 reflections. The coordination polyhedron around Cu is a square pyramid with the saldien ligand coordinated in the basal plane through one O and three N atoms; the apical position is occupied by the O of the water molecule; the Cu atom is displaced 0.174 (2) Å from the equatorial plane toward the water molecule.

**Introduction.** In recent years a great deal of attention has been given to salicylideneaminato derivatives of transition metals and their structural, electronic and magnetic properties have been measured and explained in depth. This interest can be connected both to the electronic and structural similarity of salicylaldehyde and pyridoxal (vitamin B<sub>6</sub> coenzyme) derivatives (Snell, Fasella, Bramestein & Rossi Fanelli, 1963) and to the oxygenation ability of some 'oxygen-carrier' salicylideneaminato complexes (Floriani & Calderazzo, 1969; Crumblis & Basolo, 1970).

Schiff-base complexes, therefore, have been suggested as models to describe energy transfer in naturally occurring systems and the role of the coordination sphere about the metal ion in determining the nature of the model system has been greatly emphasized (Wilkins, 1971). We have recently described the kinetic template effect of Cu<sup>II</sup> in the reaction of the salicylaldehydato

ion with diethylenetriamine (Rotondo & Cusmano Priolo, 1982): as a consequence of the presence of Cu<sup>II</sup>, the reaction between the ligands leads to the monoimine derivative through a first-order kinetic process occurring within the coordination sphere of the metal. The peculiarity of the proposed mechanism together with the nature of the title compound, which is, to our knowledge, the first example of a diethylenetriamine with only one of its NH<sub>2</sub> groups condensed with the carbonyl, suggested the undertaking of the crystal structure determination of this compound.

Crystal structure determinations of analogous Ni<sup>II</sup> and Co<sup>II</sup> derivatives are in progress.

**Experimental.** A methanolic solution of dien added to 241 mg of Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O in water; the mixture allowed to stand for several days at room temperature, a saturated solution of KPF<sub>6</sub> in water then added; slow evaporation of the solvent leads to the formation of dark-violet crystals of the monoimino derivative. The analytical data (wt %) with the calculated values in parentheses and the characteristic IR bands (cm<sup>-1</sup>) are: C 30.60 (30.52), H 4.22 (4.16), N 9.55 (9.71),  $\nu(\text{OH})$  3640,  $\nu(\text{NH})$  3358, 3315, 3150 (*br*),  $\nu(\text{C}=\text{N})$  imino 1640, (PF) 850 *vs* (*br*). The  $\nu(\text{OH})$  stretching frequency of 3640 cm<sup>-1</sup> is sharp and supports the absence of hydrogen-bonding interactions. Prismatic crystal 0.10 × 0.12 × 0.14 mm; 25 reflections with  $8 < \theta < 12^\circ$  used for measuring lattice parameters; two standard reflections measured every 120 min, no significant