other and to the anions to complete a three-dimensional array.

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Structure of Di-μ-chloro-bis[chloro(2-ethyl-2-hydroxymethyl-1,3-propanediol) copper(II)]

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Abstract. $[Cu(C_6H_{14}O_3)Cl_2]_2$, monoclinic, $P2_1/c$, a = 6·188 (2), b = 11·404 (2), c = 15·161 (4) Å, β = 109·60 (2)° (at 295 K), V = 1007·8 Å^3, Z = 4, M_r = 268·5, $F(000)$ = 548, D_c = 1·77 g cm^{-3}, λ(Mo K) = 0·7107 Å, μ = 27·4 cm^{-1}. Least-squares refinement based on 2456 reflections gave a conventional R value of 0·033. The complex crystallizes as discrete centrosymmetric dichloro-bridged dimers containing two five-coordinated Cu^{II} ions each bound by a bidentately coordinated triol molecule and three chloride ions. The dimers are held together by hydrogen bonds. Cu^{II} has coordination geometry intermediate between a trigonal bipyramid and a square pyramid. Cu—O bond lengths are 1·976 (1) and 1·991 (1) Å; the terminal Cu—Cl distance is 2·228 (1) Å and the bridging Cu—Cl distances are 2·271 (1) and 2·690 (1) Å. The six-membered chelate ring adopts the chair conformation.

Introduction. There has been considerable research activity recently on the coordination chemistry of weak neutral ligands (Poonia & Bajaj, 1979). Our principal interest is in coordination compounds with neutral polyol ligands. In this work we have prepared the $CuCl_2$ adduct of 2-ethyl-2-hydroxymethyl-1,3-propanediol (trimethylolpropane, abbreviated here as tmp) in a molar ratio of 1:1.

The first trimethylolpropane complexes in which tmp acts as a neutral ligand were reported by Hein & Buckhardt (1957), who prepared $NiSO_4$ and $CoSO_4$ adducts of the ligand in a metal-to-ligand molar ratio of 1:2. They suggested that the ligand is coordinated to the metal ion by all three neutral alcoholic O atoms. Kilivnik, Lipatova & Veselovskii (1975) found that alkaline-earth-metal chlorides inhibit the reaction of isocyanates with tmp, probably as a result of reaction of these salts with tmp. They succeeded in synthesizing $MgCl_2$, $CaCl_2$. $SrCl_2$ and $Mg(ClO_4)_2$ adducts of tmp in a molar ratio of 1:2.

In order to obtain precise information about the coordination chemistry of trimethylolpropane we have synthesized $CuCl_2$ (tmp) and determined its structure.

The complex was prepared by the addition of 3·82 g (28·4 mmol) of ligand to a stirred solution of 1·829 g (13·6 mmol) of anhydrous copper(II) chloride in 70 cm^3 of tetrahydrofuran. 15 cm^3 hexane was added to the solution and the container stoppered and set aside at room temperature. Large green crystals were obtained after about one month and were filtered off and washed with ether.

The X-ray diffraction measurements were made on a crystal 0·2 × 0·2 × 0·4 mm, with a Syntex $P2_1$ (Fortran version) automatic four-circle diffractometer employing graphite-monochromatized Mo $K\alpha$ radiation. The unit-cell parameters were calculated by least squares from the setting angles for 25 reflections. The intensities were recorded using the ω-scanning mode with a variable scan speed (3·0–29·3° min^{-1}), depending on the peak intensity of the reflection. 3093 intensities were measured for $2\theta \leq 55°$, and the 2456 reflections for which $I > 3\sigma(I)$ were used in the refinement. One check reflection was recorded after every 100 measurements. The net intensities were corrected for Lorentz and polarization effects; no absorption correction was made. The systematic absences were consistent with space group $P2_1/c$.

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Table 1. *Atomic coordinates and isotropic temperature coefficients with their estimated standard deviations*

The temperature coefficients are of the form $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta)$ and have been multiplied by 10^2.

	x	y	z	U_{eq} (Å^2)
Cu	0·5877 (1)	0·3929 (1)	0·5861 (1)	2·19 (1)
Cl(1)	0·5589 (1)	0·3835 (1)	0·4330 (1)	2·78 (1)
Cl(2)	0·2785 (1)	0·2837 (1)	0·5708 (1)	3·59 (1)
O(1)	0·6540 (1)	0·4032 (1)	0·7237 (1)	3·07 (3)
O(2)	0·9156 (1)	0·4319 (1)	0·6162 (1)	3·47 (3)
O(3)	1·3762 (1)	0·3537 (1)	0·8214 (1)	3·26 (3)
C(1)	0·7993 (1)	0·4903 (1)	0·7864 (1)	2·78 (4)
C(2)	1·0475 (2)	0·5141 (1)	0·6866 (1)	2·56 (3)
C(3)	1·1399 (1)	0·3614 (1)	0·8141 (1)	2·49 (3)
C(4)	1·0452 (1)	0·4835 (1)	0·7840 (1)	2·05 (3)
C(5)	1·1928 (2)	0·5791 (1)	0·8499 (1)	2·98 (4)
C(6)	1·2120 (2)	0·5709 (1)	0·9526 (1)	3·94 (5)
H(1)	0·4988 (44)	0·0665 (22)	0·2137 (18)	39·7 (23)
H(2)	0·0213 (27)	0·1057 (11)	0·0870 (9)	7·1 (9)
H(3)	0·4652 (30)	0·2041 (9)	0·3550 (9)	6·2 (8)
H(4)	0·1934 (17)	0·5077 (9)	0·1492 (7)	2·6 (5)
H(5)	0·2602 (18)	0·4341 (9)	0·2250 (8)	3·3 (6)
H(6)	0·8182 (21)	0·4907 (11)	0·3153 (7)	3·9 (6)
H(7)	0·0311 (17)	0·4091 (12)	0·3211 (7)	3·8 (6)
H(8)	0·1310 (18)	0·1543 (8)	0·3763 (7)	2·7 (5)
H(9)	0·0651 (26)	0·2015 (10)	0·2720 (11)	5·9 (8)
H(10)	0·6282 (19)	0·4288 (12)	0·1490 (8)	5·8 (7)
H(11)	0·8916 (28)	0·3440 (17)	0·1667 (9)	11·8 (12)
H(12)	0·9503 (24)	0·4039 (13)	0·0360 (10)	6·9 (9)
H(13)	0·3194 (29)	0·8552 (12)	0·4893 (7)	6·9 (9)
H(14)	0·7333 (23)	0·5167 (9)	0·0220 (9)	5·2 (7)

The structure was solved by direct methods using the program *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). This gave the positional parameters for the Cu and Cl atoms. Refinement of positional and thermal parameters was carried out with the XRAY 76 system (Stewart, 1976). The scattering factors were those of Cromer & Mann (1968) for the neutral atoms. After refinement of positional and isotropic thermal parameters for Cu and Cl, the coordinates of the O and C atoms could be obtained from a difference Fourier map. Positional parameters for all non-hydrogen atoms were refined with isotropic temperature factors to $R = 0{\cdot}098$ and with anisotropic temperature factors to $R = 0{\cdot}043$. All 14 H atoms could then be located from a Fourier map. When the H atoms were taken into account with isotropic temperature factors $R = 0{\cdot}034$ and with anisotropic thermal parameters for the H atoms $R = 0{\cdot}033$. The atomic fractional coordinates and isotropic thermal parameters of all atoms are presented in Table 1.*

Discussion. The complex crystallizes as discrete centrosymmetric dichloro-bridged $\mathrm{Cu^{II}}$ dimers containing five-coordinated $\mathrm{Cu^{II}}$ ions each bound by a bidentately coordinated triol molecule and three chloride ions. Bond lengths and angles around Cu are given in Table 2. An *ORTEP* (Johnson, 1965) plot of the dimer is shown in Fig. 1. It can be concluded from the values in Table 2 that the coordination geometry around the $\mathrm{Cu^{II}}$ ion is not perfectly square pyramidal but is distorted towards a trigonal bipyramid; *e.g.* the Cl(2)—Cu—O(2) and Cl(2)—Cu—Cl(1^{i}) bond angles are 158·3 (1) and 105·8 (1)°, respectively, whereas in a square pyramid they should be 180 and 90° and in a trigonal bipyramid both 120°.

Bond distances and angles within the organic ligand are presented in Table 3. The observed values are normal and the average C—O and C—C distances are 1·446 (1) and 1·531 (1) Å, respectively. The corresponding bond lengths in solid pinacol are 1·435 and 1·530 Å (Jeffrey & Robbins, 1978). Two of the angles, *viz* C(2)—C(4)—C(5) (105·5°) and C(4)—C(5)—C(6) (116·2°), deviate significantly from the tetrahedral value (109·5°). The six-membered chelate ring adopts the chair conformation (Fig. 1). The average O—H and C—H distances are also normal, 0·96 (2) and 1·01 (1) Å, respectively, but the C—H bonds, in particular, differ considerably (Table 3).

The dimers are held together by hydrogen bonds (Table 4). The bond parameters involving H(2) and H(3) are normal; H(2) lies between O(2) and Cl(2^{iii}) and H(3) between O(3) and the bridging chloride ion Cl(1^{iv}). The situation is not unambiguous with respect to H(1) (Fig. 2). There exist two possible acceptors,

Table 2. *Distances* (Å) *and angles* (°) *around the* Cu *atom*

Cu—Cl(1)	2·271 (1)	Cu—O(1)	1·991 (1)
Cu—Cl(2)	2·228 (1)	Cu—O(2)	1·976 (1)
Cu—Cl(1^{i})	2·690 (1)	Cu—$\mathrm{Cu^{(i)}}$	3·474 (1)
Cl(1)—Cu—Cl(2)	95·6 (1)	Cl(2)—Cu—O(1)	90·6 (1)
Cl(1)—Cu—O(1)	173·0 (1)	Cl(2)—Cu—O(2)	158·3 (1)
Cl(1)—Cu—O(2)	88·2 (1)	Cl(2)—Cu—Cl(1^{i})	105·8 (1)
Cl(1)—Cu—Cl(1^{i})	91·5 (1)		
O(1)—Cu—O(2)	84·8 (1)	O(2)—Cu—Cl(1^{i})	95·4 (1)
O(1)—Cu—Cl(1^{i})	89·9 (1)		

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

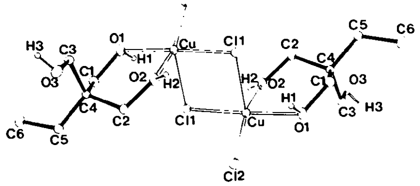

Fig. 1. A perspective view of $\mathrm{CuCl_2(tmp)}$ dimer.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36608 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Distances* (Å) *and angles* (°) *in the trimethylolpropane molecule*

C(1)–O(1)	1·458 (1)	C(1)–H(4)	0·962 (11)
C(1)–C(4)	1·536 (1)	C(1)–H(5)	0·932 (10)
C(2)–O(2)	1·449 (1)	C(2)–H(6)	0·843 (14)
C(2)–C(4)	1·523 (1)	C(2)–H(7)	0·989 (13)
C(3)–O(3)	1·431 (1)	C(3)–H(8)	0·980 (12)
C(3)–C(4)	1·520 (1)	C(3)–H(9)	0·969 (13)
C(4)–C(5)	1·553 (1)	C(5)–H(10)	1·106 (13)
C(5)–C(6)	1·524 (2)	C(5)–H(11)	1·009 (18)
O(1)–H(1)	0·983 (27)	C(6)–H(12)	1·112 (16)
O(2)–H(2)	1·001 (17)	C(6)–H(13)	1·102 (13)
O(3)–H(3)	0·899 (12)	C(6)–H(14)	1·083 (11)
O(1)–C(1)–C(4)	110·5 (1)	C(1)–C(4)–C(5)	108·9 (1)
O(2)–C(2)–C(4)	112·3 (1)	C(2)–C(4)–C(3)	111·9 (1)
O(3)–C(3)–C(4)	111·2 (1)	C(2)–C(4)–C(5)	105·5 (1)
C(1)–C(4)–C(2)	110·1 (1)	C(3)–C(4)–C(5)	111·5 (1)
C(1)–C(4)–C(3)	108·9 (1)	C(4)–C(5)–C(6)	116·2 (1)

Table 4. *Distances* (Å) *and angles* (°) *associated with the possible hydrogen bonds*

X–H···Y	X···Y	X–H	H···Y	∠X–H···Y
O(1)–H(1)···O(3^{ii})	2·679 (1)	0·98 (3)	2·21 (3)	107 (2)
O(1)–H(1)···Cl(2)	3·002 (1)	0·98 (3)	2·74 (3)	96 (2)
O(2)–H(2)···Cl(2^{iii})	3·067 (1)	1·00 (2)	2·11 (2)	160 (1)
O(3)–H(3)···Cl(1^{iv})	3·189 (1)	0·90 (1)	2·34 (1)	158 (2)

Symmetry code: (ii) $x-1, y, z$; (iii) $x+1, y, z$; (iv) $x+1, \frac{1}{2}-y, \frac{1}{2}+z$.

Cl(2) and O(3^{ii}). On the other hand, bifurcated hydrogen bonds are a common phenomenon in molecular crystals (Ceccarelli, Jeffrey & Taylor, 1981). The observed angles around H(1) are small for a bifurcated hydrogen bond and the sum of the angles around H(1) is 300° instead of 360° as would be expected for an ideal bifurcated hydrogen bond. It is clear that O(1), H(1), Cl(2) and O(3^{ii}) are not exactly in the same plane. However, the large temperature factor of H(1) and the observed distances around H(1) indicate the existence of at least a weak hydrogen bond and possibly a bifurcated hydrogen bond.

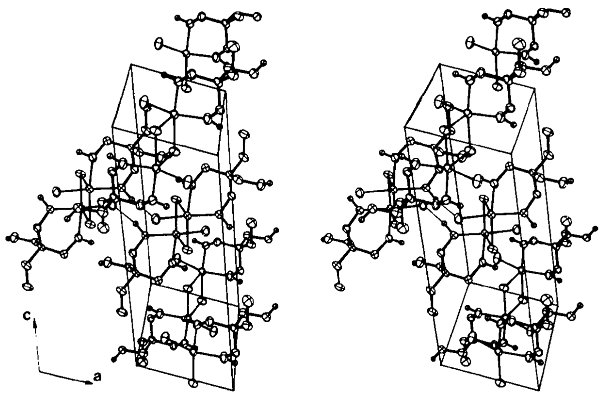

Fig. 2. A stereoscopic drawing of the molecular packing in the unit cell.

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Structure of Bis(trifluoromethyl)mercury(II)–2,2′:6′,2″-Terpyridyl

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Abstract. $[Hg(CF_3)_2].C_{15}H_{11}N_3$, triclinic, $P\bar{1}$, a = 11·821 (5), b = 9·141 (3), c = 8·955 (4) Å, α = 110·12 (3), β = 95·04 (3), γ = 95·66 (3)°, V = 896·5 (7) Å^3, μ(Mo $K\alpha$) = 8·83 mm^{-1}, Z = 2, D_m = 2·22, D_x = 2·118 Mg m^{-3}. The structure was refined by least-squares procedure to R = 0·069 for 2561 reflexions with $I > 3\sigma(I)$. The crystal structure is built up of F_3C–Hg–CF_3 and 2,2′:6′,2″-terpyridyl (tpy)

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