

Crystal and Molecular Structures of Two Di- μ -chlorobis[chloro-(1,2-cyclohexanediol)copper(II)] Dicyclohexanediol Complexes

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Copper(II) chloride forms with both *cis*- and *trans*-forms of 1,2-cyclohexanediol (chd) a complex having the composition $\text{CuCl}_2(\text{C}_6\text{H}_{12}\text{O}_2)_2$. Both complexes crystallize in the monoclinic system with four molecules in the unit cell. The unit cell parameters for $\text{CuCl}_2 \cdot 2\text{cis-chd}$ are: $a=12.542(5)$, $b=8.830(3)$, $c=14.265$ Å, $\beta=90.24(2)^\circ$ and $V=1579.8$ Å³; the space group is $P2_1/n$. For $\text{CuCl}_2 \cdot 2\text{trans-chd}$ the unit cell is slightly smaller: $a=10.282(2)$, $b=14.675(2)$, $c=10.379(1)$ Å, $\beta=96.79(1)^\circ$ and $V=1555.1$ Å³; the space group is $P2_1/c$.

The crystal structures of the complexes were determined from single crystal data and refinements resulted in *R*-values of 0.042 and 0.047 for $\text{CuCl}_2 \cdot 2\text{cis-chd}$ and $\text{CuCl}_2 \cdot 2\text{trans-chd}$, respectively. In both structures there is a 4+1 coordination around the copper atom. In the compound of *cis*-form, two oxygen atoms and two chlorine atoms are situated on plane at an average distance of 2.01 and 2.25 Å from the copper atom, respectively, while one chlorine atom is 2.76 Å above the copper atom and the plane. In the complex of *trans*-form the corresponding distances are 2.01 (Cu-O), 2.26 (Cu-Cl) and 2.65 Å (Cu-Cl'). The $\text{CuCl}_2(\text{chd})$ units are joined into dimers *via* chloro bridges and only one chd molecule is coordinated to the copper(II) ion. The two uncoordinated diol molecules per dimer are held in the structure by hydrogen bonding to the $[\text{CuCl}_2(\text{chd})]_2$ complexes.

preparative studies by Grün *et al.* who studied the ethylene glycol complexes with light transition metals.^{1,2} Later structural information has also become available for the solid ethylene glycol complexes; the most comprehensive X-ray study has been performed by Antti and co-workers.³ Recently, there has been considerable research interest involving also other weak neutral ligands.^{4,5}

As part of a larger investigation into the coordination chemistry of neutral diol and polyol ligands,⁶ the present study dealing with 1,2-cyclohexanediol and copper(II) ions was initiated. The diols used were *cis*-1,2-cyclohexanediol (abbreviated *cis-chd*) and (\pm)-*trans*-1,2-cyclohexanediol (*trans-chd*). A discussion of the structures of these diols in the solid state has been given by Furberg and Hassel⁷ who showed that the cyclohexane ring of both diols is in the chair form. Hydroxyl groups of (\pm)-*trans*-1,2-cyclohexanediol are equatorial but in the *cis-chd* one group is axial while the other is equatorial. In the gas phase, however, *trans-chd* exists also as a conformer with both OH-substituents axial.⁸ These diols are often used as model compounds in carbohydrate chemistry.⁹

EXPERIMENTAL

Syntheses. Ligands were prepared according to existing methods.^{10,11} $\text{CuCl}_2 \cdot 2\text{cis-chd}$ was prepared by dissolving 5.0 mmol of CuCl_2 and 12.5 mmol of *cis-chd* in 25 cm³ of 1,2-dimethoxyethane. The solution was heated gently until

The great stability of metal complexes formed by diols was recognized already during the early

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Table 1. Crystal data for $[\text{CuCl}_2(\text{C}_6\text{H}_{12}\text{O}_2)] \cdot \text{C}_6\text{H}_{12}\text{O}_2$

	$\text{CuCl}_2 \cdot 2\text{cis-chd}$	$\text{CuCl}_2 \cdot 2\text{trans-chd}$
Crystal system	monoclinic	monoclinic
Space group	$P2_1/n$	$P2_1/c$
a (Å)	12.542(5)	10.282(2)
b (Å)	8.830(3)	14.675(2)
c (Å)	14.265(7)	10.379(1)
β (°)	90.24(2)	96.79(1)
V (Å ³)	1579.8	1555.1
Formula weight	366.77	366.77
Z	4	4
D_x (g cm ⁻³)	1.54	1.57
Crystal size (mm ³)	0.3×0.3×0.6	0.2×0.4×0.4
μ (cm ⁻¹)	17.75	17.98
Number of reflections collected	3709	3731
Number of reflections used in refinement	3096	3022

complete dissolution and then kept in a closed vessel at room temperature for a few weeks. The green crystals formed were separated by decantation and washed twice with 10 cm³ of ether. $\text{CuCl}_2 \cdot 2\text{trans-chd}$ crystals were obtained by the same method. Crystals are stable in dry air but are destroyed by moisture.

Structure determination. The X-ray diffraction measurements were carried out on a Syntex P2₁ (Fortran version) automatic four-circle diffractometer employing graphite monochromatized MoK α -radiation ($\lambda=0.71069$ Å). The unit cell parameters at 298 K were calculated by least-

squares refinements of 25 and 22 reflections for the *cis*- and *trans*-compounds, respectively. The intensities were recorded using the θ - 2θ scanning mode with varying scan speed (3–29.3° min⁻¹) depending on the peak intensity of the reflection. All reflections $2\theta < 55^\circ$ were measured and those with $I > 3\sigma(I)$ were used in the subsequent calculations. The intensity of one check reflection was recorded after every 100 measurements. The net intensities were corrected for Lorentz and polarization effects but not for absorption. The systematic absences indicated the space group 14 ($P2_1/n$ or $P2_1/c$); crystal data are summarized in Table 1.

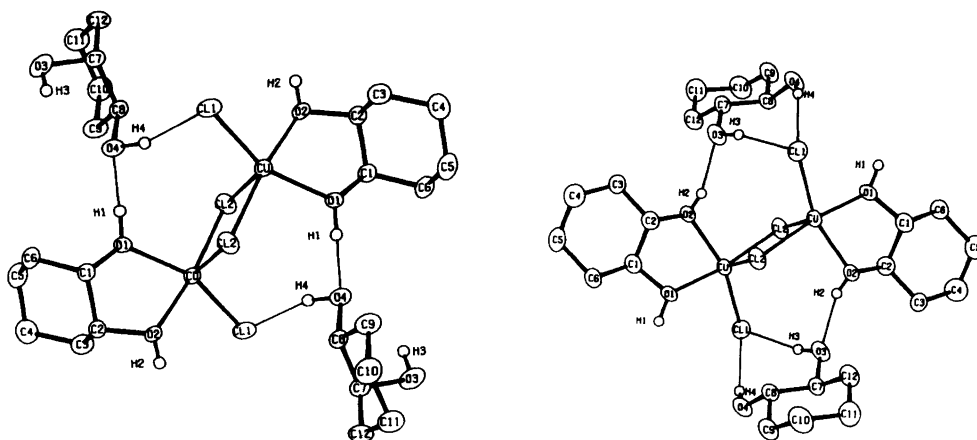


Fig. 1. Perspective views showing the coordination and atomic numbering scheme of $\text{CuCl}_2 \cdot 2\text{cis-chd}$ (left) and $\text{CuCl}_2 \cdot 2\text{trans-chd}$ (right).

Table 2. Atomic coordinates and anisotropic 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 10^4 . Hydrogen atom positions are not refined.

Atom	x	y	z	U_{eq}
CuCl₂·2<i>cis</i>-chd^a				
Cu	-0.1086(0)	0.3646(0)	0.5289(0)	248(3)
C11	-0.2501(1)	0.3928(1)	0.4341(1)	362(6)
C12	-0.0812(1)	0.6099(1)	0.5674(1)	340(6)
O1	0.4525(3)	0.2088(3)	0.1508(2)	278(16)
O2	0.3891(3)	0.3603(3)	0.0072(2)	277(16)
O4	0.3773(3)	-0.0700(4)	0.7809(2)	316(17)
O3	0.2672(3)	-0.0387(4)	0.6091(3)	475(22)
C1	0.4847(3)	0.3667(5)	0.1493(3)	257(21)
C2	0.3973(4)	0.4481(5)	0.0944(3)	251(21)
C3	0.2930(4)	0.4574(6)	0.1451(3)	349(27)
C4	0.3090(5)	0.5296(7)	0.2418(4)	433(31)
C5	0.3889(5)	0.4280(6)	0.2991(3)	421(29)
C6	0.4942(4)	0.4283(6)	0.2498(3)	364(26)
C7	0.2926(4)	0.0919(5)	0.6656(3)	306(24)
C8	0.3942(4)	0.0619(5)	0.7222(3)	264(22)
C9	0.4908(4)	0.0369(6)	0.6613(3)	359(27)
C10	0.5074(4)	0.1731(7)	0.5967(4)	420(30)
C11	0.4077(5)	0.2039(6)	0.5382(4)	437(30)
C12	0.3115(4)	0.2256(6)	0.5993(4)	397(29)
H1	0.500	0.150	0.180	
H2	0.339	0.399	-0.020	
H3	0.259	-0.106	0.660	
H4	0.341	-0.030	0.840	
H5	0.558	0.375	0.120	
H6	0.428	0.551	0.080	
H7	0.244	0.513	0.120	
H8	0.260	0.352	0.140	
H9	0.330	0.642	0.240	
H10	0.240	0.532	0.280	
H11	0.401	0.482	0.360	
H12	0.362	0.336	0.320	
H13	0.516	0.542	0.240	
H14	0.539	0.366	0.300	
H15	0.234	0.116	0.720	
H16	0.404	0.152	0.760	
H17	0.547	0.024	0.700	
H18	0.480	0.057	0.620	
H19	0.568	0.155	0.560	
H20	0.521	0.262	0.640	
H21	0.395	0.121	0.480	
H22	0.407	0.297	0.520	
H23	0.248	0.243	0.560	
H24	0.321	0.321	0.640	
CuCl₂·2<i>trans</i>-chd^b				
Cu	0.6219(0)	0.4207(0)	0.4617(0)	284(3)
C11	0.7849(1)	0.4062(1)	0.6256(1)	386(5)
C12	0.4507(1)	0.4075(1)	0.5785(1)	357(5)
O1	0.7555(3)	0.4260(2)	0.3349(3)	396(17)
O2	0.5152(3)	0.3736(2)	0.3016(3)	369(16)

Table 2. *Continued.*

O3	0.2604(3)	0.4039(2)	0.2440(3)	415(18)
O4	0.0214(3)	0.4195(2)	0.3797(3)	448(19)
C1	0.7135(4)	0.3777(4)	0.2147(4)	451(27)
C2	0.5723(4)	0.3978(4)	0.1850(4)	441(26)
C3	0.5100(4)	0.3496(3)	0.0646(4)	411(24)
C4	0.5871(5)	0.3667(5)	-0.0491(5)	573(33)
C5	0.7283(6)	0.3532(5)	-0.0244(5)	673(38)
C6	0.7921(4)	0.4033(4)	0.1051(4)	421(25)
C7	0.1681(4)	0.3324(3)	0.2595(4)	343(22)
C8	0.1040(4)	0.3397(3)	0.3836(4)	365(23)
C9	0.0166(4)	0.2578(3)	0.3983(5)	425(26)
C10	0.0900(5)	0.1685(4)	0.3933(5)	513(31)
C11	0.1534(5)	0.1600(4)	0.2661(5)	509(30)
C12	0.2435(4)	0.2415(3)	0.2545(5)	417(25)
H1	0.842	0.435	0.341	
H2	0.441	0.395	0.295	
H3	0.236	0.457	0.273	
H4	0.053	0.477	0.370	
H5	0.684	0.439	0.205	
H6	0.561	0.466	0.171	
H7	0.420	0.377	0.045	
H8	0.504	0.289	0.085	
H9	0.553	0.326	-0.123	
H10	0.572	0.432	0.080	
H11	0.726	0.413	0.030	
H12	0.795	0.300	0.030	
H13	0.780	0.472	0.073	
H14	0.753	0.358	0.030	
H15	0.093	0.332	0.178	
H16	0.177	0.344	0.456	
H17	-0.079	0.248	0.338	
H18	-0.013	0.266	0.481	
H19	0.029	0.108	0.396	
H20	0.160	0.156	0.461	
H21	0.081	0.159	0.193	
H22	0.208	0.158	0.340	
H23	0.311	0.247	0.327	
H24	0.987	0.280	0.202	

^a Lattice parameters: $a=12.542$, $b=8.830$, $c=14.265$ Å, $\beta=90.24^\circ$. ^b Lattice parameters: $a=10.282$, $b=14.675$, $c=10.379$ Å, $\beta=96.79^\circ$.

The structures were solved by direct methods (MULTAN)¹² yielding the positions of Cu and Cl atoms; the remaining nonhydrogen atoms were found from a difference Fourier map. Refinements were carried out with the X-RAY SYSTEM 1976.¹³ An isotropic refinement led to the R -values of 0.102 and 0.104 for the *cis*- and *trans*-compounds, respectively. The anisotropic refinement of nonhydrogen atoms and the inclusion of 24 H-atoms at fixed positions with

constant $U=0.05$ resulted in the final R -values of 0.042 and 0.047. The scattering factors were those given by Cromer and Mann for neutral atoms.¹⁴ In the refinement, unit weights were employed. The final positional parameters and isotropic temperature factors are listed in Table 2. The observed and calculated structure factors and anisotropic temperature factors are available from the authors upon request.



Fig. 2. Distances around the copper(II) ion in the structures of CuCl₂·2*cis*-chd (left) and CuCl₂·2*trans*-chd (right). $i = -x, -y, -z$ for *cis* and $1-x, 1-y, 1-z$ for *trans* compound.

RESULTS AND DISCUSSION

The structures of both compounds contain di- μ -chlorobridged copper(II) dimers joined together by hydrogen bonds of uncoordinated diol molecules. The Cu(II) ions are surrounded by five ligand donor atoms at bonding distance; two of them are oxygens from the diol molecules and two are chlorine atoms. The fifth coordination site is occupied by a chlorine of adjacent molecule.

The arrangement and numbering of atoms are shown in Fig. 1, while Fig. 2 gives the bond lengths around Cu. Additional data for the dimers are listed in Table 3. It may be noted that the lengths of Cu-O and Cu-Cl bonds are of same magnitude as found in [CuCl₂(eg)] and [CuCl₂(eg)]·1/2H₂O where they are 1.958–2.004 Å and 2.32–2.267 Å for Cu-O and Cu-Cl, respectively.¹⁵ The Cu–Cu distances are 3.718(1) Å in CuCl₂·2*cis*-chd and 3.579(1) Å in CuCl₂·2*trans*-chd, which clearly indicates the

absence of copper–copper bonds in the dimers.

The Cu-Cl-Cu bridging angle is 95.0(1) and 93.3(1)° for the CuCl₂·2*cis*-chd and CuCl₂·2*trans*-chd, respectively. Their values are similar to those found in copper(II) di- μ -chlorobridged dimers having a tetragonal pyramidal (TP) geometry.¹⁶

The bond distances involving C and O atoms are listed in Table 4. The coordination of diol to copper leads to the expected lengthening of C-O distances (1.450(6)–1.469(5) Å) as compared to the uncoordinated diol (1.437(6)–1.450(6) Å). Bond angles are also influenced by the coordination although in general they are near to the tetrahedral angle of 109.5°.

The values of the torsion angles of the coordinated and uncoordinated diols, presented in Table 5, also reflect the same effect. The torsion angles of six membered carbon rings show that all diol molecules have a chair conformation and that in

Table 3. Selected distances (Å) and angles (°) around the Cu atom, see also Fig. 1.^a

	CuCl ₂ ·2 <i>cis</i> -chd	CuCl ₂ ·2 <i>trans</i> -chd
Cu–Cu ⁱ	3.718(1)	3.579(1)
Cl1–Cu–Cl2	99.21(1)	98.3(1)
Cl1–Cu–Cl2 ⁱ	111.9(1)	112.3(1)
Cl1–Cu–O1	149.1(1)	89.6(1)
Cl1–Cu–O2	90.4(1)	151.6(1)
Cl2–Cu–O1	92.4(1)	171.4(1)
Cl2–Cu–O2	170.4(1)	91.7(1)
Cl2 ⁱ –Cu–Cl2	85.0(1)	86.7(1)
Cl2 ⁱ –Cu–O1	97.5(1)	93.5(1)
Cl2 ⁱ –Cu–O2	90.9(1)	94.7(1)
O1–Cu–O2	79.6(1)	79.6(1)
Cu–Cl2–Cu ⁱ	95.0(1)	93.3(1)

^a Symmetry code: $i = -x, -y, -z$ for CuCl₂·2*cis*-chd and $i = 1-x, 1-y, 1-z$ for CuCl₂·2*trans*-chd.

Table 4. Bonding distances (Å) and angles (°) in cyclohexane diol molecules.

	CuCl ₂ ·2 <i>cis</i> -chd	CuCl ₂ ·2 <i>trans</i> -chd
O1–C1	1.452(5)	1.456(6)
O2–C2	1.469(5)	1.450(6)
O3–C7	1.442(6)	1.437(6)
O4–C8	1.450(6)	1.444(6)
C1–C2	1.525(6)	1.478(7)
C2–C3	1.500(7)	1.510(7)
C3–C4	1.532(7)	1.518(7)
C4–C5	1.523(8)	1.457(8)
C5–C6	1.502(8)	1.602(7)
C6–C1	1.537(7)	1.519(7)
C7–C8	1.529(7)	1.519(7)
C8–C9	1.510(7)	1.519(7)
C9–C10	1.530(8)	1.517(8)
C10–C11	1.524(8)	1.545(8)
C11–C12	1.504(8)	1.526(8)
C12–C7	1.532(7)	1.547(7)
Cu–O1–C1	113.0(2)	112.7(2)
Cu–O2–C2	113.8(2)	111.8(2)
O1–C1–C2	105.1(3)	105.5(4)
O1–C1–C6	110.3(4)	113.0(4)
C2–C1–C6	111.4(4)	113.0(4)
O2–C2–C1	103.5(3)	105.2(4)
O2–C2–C3	112.3(4)	113.7(4)
C1–C2–C3	113.9(4)	113.0(4)
C2–C3–C4	110.3(4)	110.9(4)
C3–C4–C5	110.2(5)	116.2(4)
C4–C5–C6	110.9(4)	112.7(5)
C5–C6–C1	113.1(4)	108.3(4)
O3–C7–C8	109.8(4)	113.7(4)
O3–C7–C12	107.8(4)	106.6(3)
C8–C7–C12	109.2(4)	111.0(4)
O4–C8–C7	108.7(4)	110.3(4)
O4–C8–C9	109.5(4)	106.9(3)
C7–C8–C9	113.0(4)	110.5(4)
C8–C9–C10	110.1(4)	112.2(4)
C9–C10–C11	110.9(4)	111.2(4)
C10–C11–C12	111.4(4)	109.0(4)
C11–C12–C7	112.7(4)	110.9(4)

CuCl₂·2*trans*-chd the uncoordinated and coordinated diols are enantiomers.¹⁷ The values of the torsion angles involving one oxygen atom show that in both diols of CuCl₂·2*cis*-chd one OH group is equatorial and the other is axial while in the diols of CuCl₂·2*trans*-chd both OH groups are equatorial.

The values of torsion angles involving two oxygen atoms give an indication whether the chelate ring formed by O1–C1–C2–O2 is λ or σ . The chelate rings formed by (–)*trans*-chd and

(+)*trans*-chd must have the λ - and σ -conformations, respectively.¹⁸ In the CuCl₂·2*trans*-chd presence of inversion center constrains each dimer to contain one λ and one σ chelate ring. For symmetry reasons the diols, which are joined by hydrogen bonds to a dimeric unit, must also be enantiomers. In *cis*-chd there are two enantiomeric conformers which can convert into each other but in the solid state this conversion is rendered impossible. Symmetry reasons require also in CuCl₂·2chd that in the unit cell there are

Table 5. Torsion angles (°) for the cyclohexanediol molecules in CuCl₂·2*cis*-chd and CuCl₂·2*trans*-chd.

	CuCl ₂ ·2 <i>cis</i> -chd	CuCl ₂ ·2 <i>trans</i> -chd
O1-C1-C2-O2	-52.3(4)	-53.2(5)
O1-C1-C2-C3	69.9(5)	-177.7(4)
O1-C1-C6-C5	-67.3(5)	-174.7(4)
O2-C2-C3-C4	171.8(4)	-171.4(4)
O2-C2-C1-C6	-171.7(4)	-177.1(4)
C1-C2-C3-C4	54.6(6)	-51.7(6)
C2-C3-C4-C5	-58.3(6)	48.5(7)
C3-C4-C5-C6	58.6(6)	-48.7(8)
C4-C5-C6-C1	-54.4(6)	50.0(7)
C5-C6-C1-C2	49.0(6)	-55.1(6)
C6-C1-C2-C3	-49.5(5)	58.4(6)
O3-C7-C8-O4	-58.9(6)	66.6(4)
O3-C7-C8-C9	62.9(5)	-175.3(3)
O3-C7-C12-C11	-65.2(7)	-178.3(4)
O4-C8-C9-C10	-176.9(5)	175.6(4)
O4-C8-C7-C12	178.2(5)	-173.1(3)
C7-C8-C9-C10	56.8(6)	55.6(5)
C8-C9-C10-C11	-55.9(7)	-56.8(5)
C9-C10-C11-C12	55.7(6)	56.8(5)
C10-C11-C12-C7	-55.5(7)	-57.0(5)
C11-C12-C7-C8	54.0(8)	57.3(5)
C12-C7-C8-C9	-55.1(6)	-55.1(5)

four of each enantiomers.

The structures of both compounds contain hydrogen bonds of type OH...O and OH...Cl as uncoordinated diol oxygens accept hydrogens from coordinated diol hydroxyl groups while the nonbridging chloride ions are acceptors to two

hydrogen atoms from the uncoordinated diols; the unit cell contents are depicted in Figs. 3 and 4. In CuCl₂·2*cis*-chd the two OH...Cl hydrogen bonds involve different diols, but in CuCl₂·2*trans*-chd they are formed by the same uncoordinated diol (Table 6). The OH...O bond

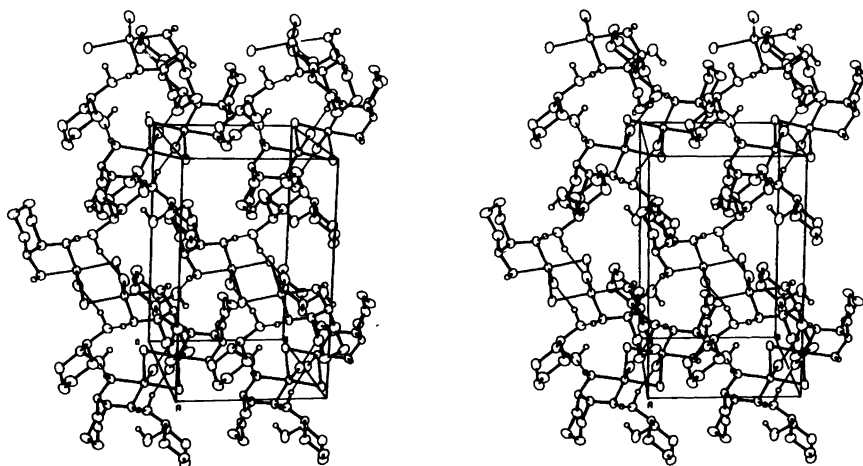


Fig. 3. A stereoview of the structure of CuCl₂·2*cis*-chd showing the unit cell packing.

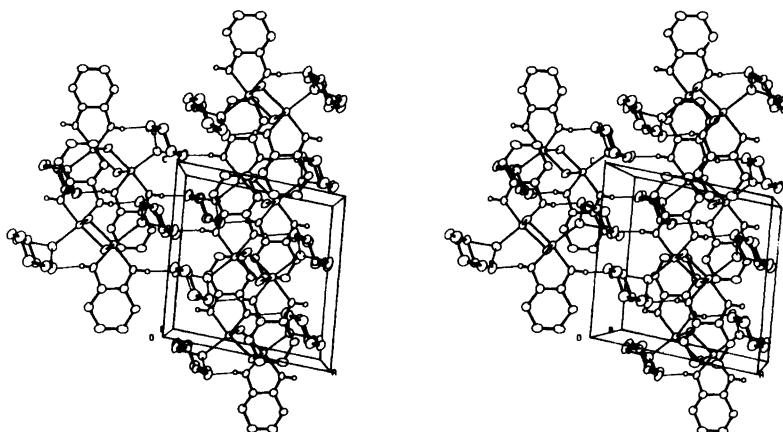


Fig. 4. A stereoview of the structure of $\text{CuCl}_2 \cdot 2\text{trans-chd}$ showing the unit cell packing.

Table 6. Distances (Å) and angles ($^\circ$) involving hydrogen atoms of hydroxyl groups (X=O, Cl).

	O-H	H...X	O-X	O-H...X
$\text{CuCl}_2 \cdot 2\text{cis-chd}$				
O1-H1...O4	0.891	1.781	2.644(5)	162.2
O2-H2...O3	0.808	1.922	2.714(5)	166.5
O3-H3...Cl1	0.941	2.872	3.192(4)	101.4
O3-H3...O4	0.941	2.289	2.822(5)	115.3
O4-H4...Cl1	1.025	2.140	3.133(4)	162.5
$\text{CuCl}_2 \cdot 2 \text{trans-chd}$				
O1-H1...O4	0.898	1.850	2.720(4)	162.7
O2-H2...O3	0.818	1.877	2.655(4)	158.5
O3-H3...Cl1	0.882	2.290	3.197(4)	167.5
O4-H4...Cl1	0.918	2.384	3.248(3)	156.9

lengths and angles are normal for medium strength hydrogen bonds as found in many carbohydrate structures.¹⁹

The hydrogen bond lengths involving chlorine as an acceptor appear normal when compared to the same type of compounds. In the $\text{CuCl}_2 \cdot 2\text{cis-chd}$ the distances and angles around H(3) suggest that there is a bifurcated hydrogen bond which is not an unusual feature in molecular crystals.²⁰ The IR spectrum of $\text{CuCl}_2 \cdot 2\text{cis-chd}$ is in agreement with the weak character of this bond showing a band at 3522 cm^{-1} . Nevertheless, the hydrogen bonding plays an important role in the formation and stability of the compounds investigated.

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