

Copper(II) Complexes of 2-Amino-2-hydroxymethyl-1,3-propanediol. Part 4. Synthesis, Structure and Thermal Behavior of *trans*-Bis[2-amino-2-hydroxymethyl-1,3-propanediolato-*O,N*]copper(II) Potassium Fluoride and Bromide, $[\text{Cu}(\text{C}_4\text{H}_{10}\text{NO}_3)_2]\text{KF} \cdot 3\text{H}_2\text{O}$ and $[\text{Cu}(\text{C}_4\text{H}_{10}\text{NO}_3)_2]\text{KBr} \cdot 2\text{H}_2\text{O}$

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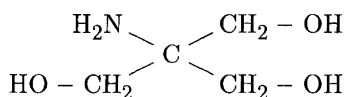
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Kotila, S. and Valkonen, J., 1994. Copper(II) Complexes of 2-Amino-2-hydroxymethyl-1,3-propanediol. Part 4. Synthesis, Structure and Thermal Behavior of *trans*-Bis[2-amino-2-hydroxymethyl-1,3-propanediolato-*O,N*]copper(II) Potassium Fluoride and Bromide, $[\text{Cu}(\text{C}_4\text{H}_{10}\text{NO}_3)_2]\text{KF} \cdot 3\text{H}_2\text{O}$ and $[\text{Cu}(\text{C}_4\text{H}_{10}\text{NO}_3)_2]\text{KBr} \cdot 2\text{H}_2\text{O}$. – Acta Chem. Scand. 48: 312–318 © Acta Chemica Scandinavica 1994.

Crystal structures of two copper(II) potassium halides with 2-amino-2-hydroxymethyl-1,3-propanediol (= tris, deprotonated form abbreviated trisH_{-1}) as a ligand have been determined from single-crystal X-ray data and refined to the final *R* values of 0.040 (F^-) and 0.036 (Br^-). The compounds are almost isomorphous except that the fluoride compound has disordered fluoride ions and an extra disordered water molecule. The compounds crystallize in the orthorhombic space group *Pcca* (No. 54) with four molecules in a unit cell. Crystal data for $[\text{Cu}(\text{trisH}_{-1})_2]\text{KF} \cdot 3\text{H}_2\text{O}$ are as follows: $a = 18.814(2)$, $b = 9.623(1)$, $c = 8.678(2)$ Å, $V = 1571.2(4)$ Å³, and for $[\text{Cu}(\text{trisH}_{-1})_2]\text{KBr} \cdot 2\text{H}_2\text{O}$: $a = 18.482(2)$, $b = 9.805(1)$, $c = 8.776(3)$ Å, $V = 1590.4(5)$ Å³.

The structure is a tunnel compound with neutral copper–tris complexes (square-planar *trans* coordination of nitrogen and oxygen) polymerized by hydrogen bonding in the *c*-direction forming hydrophobic and hydrophilic cavities along the same axis. Potassium occupies the hydrophilic cavity and is surrounded by eight oxygens with K–O distances 2.776(5)–2.988(4) Å. Halogenides are located in the hydrophobic cavity. The three-dimensional network consists of hydrogen bonds and the coordination bonds of potassium linking the complex molecules together. Thermal behaviour is characterized by TG in air and nitrogen atmospheres.

2-Amino-2-hydroxymethyl-1,3-propanediol (tris) also known as tris(hydroxymethyl)aminomethane (tham) or commercially as Trizma Base, is a widely used buffering agent in biochemical studies¹ in the physiological pH range 7–9.



Tris chelates with transition metals are usually formed via amino and one or two hydroxymethyl groups. The reaction of aminoalcohols with metal cations also yields

deprotonated complexes with coordinated alkoxy groups, and these deprotonated species are abbreviated trisH_{-1} .

We are especially interested in solid tris compounds with copper(II) as a metal cation. In our earlier articles we have reported the synthesis, structures and thermal behavior of three *trans* complexes, $[\text{Cu}(\text{trisH}_{-1})_2]$, $[\text{Cu}(\text{trisH}_{-1})_2(\text{H}_2\text{O})]$ and $[\text{Cu}(\text{trisH}_{-1})_2] \cdot 5\text{H}_2\text{O}$,² and four *cis* compounds, $[\text{Cu}(\text{trisH}_{-1})(\text{tris})(\text{NO}_3)]$,³ $[\text{Cu}(\text{trisH}_{-1})(\text{tris})\text{Na}(\text{ClO}_4)_2]$,³ $[\text{Cu}(\text{trisH}_{-1})(\text{tris})(\text{H}_2\text{O})]_2\text{SO}_4$,⁴ and $[\text{Cu}(\text{trisH}_{-1})(\text{tris})(\text{H}_2\text{O})]_2\text{CrO}_4$.⁴ The structures of the following copper–tris complexes are also known, $[\text{Cu}(\text{trisH}_{-1})_2]\text{NaClO}_4 \cdot \text{H}_2\text{O}$,⁵ $[\text{Cu}(\text{trisH}_{-1})\text{Cl}]_4$,⁶ and $[\text{Cu}(\text{trisH}_{-1})(\text{tris})]_2\text{Br}_2$.⁶ In this paper we present two new tris compounds containing two metal cations and having a tunnel structure, namely $[\text{Cu}(\text{trisH}_{-1})_2]\text{KF} \cdot 3\text{H}_2\text{O}$ and $[\text{Cu}(\text{trisH}_{-1})_2]\text{KBr} \cdot 2\text{H}_2\text{O}$.

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Experimental

Preparation of [Cu(trisH₋₁)₂]KX·nH₂O, where X = F⁻ (n = 3), Br⁻ (n = 2). Both compounds were prepared by the same procedure using CuF₂·2H₂O (Fluka, min. 97%) or CuBr₂ (B.D.H. Ltd., min. 98%) as starting materials. The reaction was also tested with copper(II) chloride, but under these conditions tris forms polynuclear complexes giving a green tetramer, [Cu(trisH₋₁)Cl]₄,⁶ as a product. Attempts to prepare similar sodium compounds using NaOH in the reaction were not successful either.

A 250 cm³ round-bottomed, two-neck flask was fitted with a refluxing condenser and a thermometer. Into the flask 0.015 mol of tris (Sigma, 99–99.5%) was placed and dissolved in 40 cm³ of ethanol by refluxing. At the same time 0.0075 mol of copper(II) halide was dissolved separately in 30 cm³ of ethanol by heating. The warm copper halide solution was added to the tris solution, the reaction temperature was raised to 70°C, and the mixture was maintained at this temperature for 5 min. The reaction was then cooled to 45°C, the clear solution was separated from the undissolved copper halide by decantation, and the solution was made alkaline with KOH (Riedel-de-Haën, 0.015 mol KOH in 30 cm³ EtOH). The solution was filtered with a sintered glass crucible and allowed to crystallize at room temperature; needle-shaped brownish crystals were obtained as a product. The first crystal fraction appearing within 2 h is usually of poor quality and not suitable for a single-crystal determination. After 2 h the mother liquor was decanted to another Erlenmeyer flask, and a few drops of water were added to the solution. The second fraction, crystallizing overnight, yields crystals of a better size and quality for X-ray measurements. The compounds are not very

stable; if they are ground into powder and kept in an air atmosphere, they slowly transform into the blue monohydrate complex, [Cu(trisH₋₁)₂(H₂O)]₂² and potassium halide (confirmed by IR).

Possible side-products in the reaction are the anhydrous and monohydrate *trans* complexes, [Cu(trisH₋₁)₂] and [Cu(trisH₋₁)₂(H₂O)]₂.² The formation of the monohydrate complex is favored especially if the reaction temperature is too high or prolonged heating takes place during the reaction.

Thermal analysis. Thermal behaviour of the complexes in air and nitrogen atmospheres was determined with a Perkin-Elmer thermogravimetric analyzer TGA7. The sample size varied between 3.0 ± 0.3 mg, and the crystalline samples were analyzed with a heating rate 2°C min⁻¹ and gas flow 50 cm³ min⁻¹. Temperature ranges in air and nitrogen atmospheres were 25–950 and 25–900°C, respectively. To remove oxygen from the oven when using nitrogen atmosphere, the equipment was flushed with nitrogen for 30–45 min before the temperature program was initiated. Observed and theoretical weight losses are reported in Table 1.

Crystal structure determinations. Single-crystal X-ray measurements were made with an Enraf-Nonius CAD-4 diffractometer using MoK_α radiation. The fluoride compound was measured in a glass capillary containing a drop of mother liquor, whereas the crystal of the bromide compound was covered with epoxy glue and mounted on a glass fiber. Crystal data and experimental details are given in Table 2.

Accurate cell constants were obtained by a least-squares analysis of 25 centered reflections. Three reflec-

Table 1. Thermal decomposition of [Cu(trisH₋₁)₂]KF·3H₂O (1) and [Cu(trisH₋₁)₂]KBr·2H₂O (2).

Compound	Lost in reaction	T/°C	Weight loss (%)	
			ΔObs.	ΔTheor.
Air atmosphere				
1	3 H ₂ O	25–125	11.6	13.0
	Org. (→CuO+KF)	125–503	54.9	53.9
	KF	727–907	11.6	14.0
	Total reaction→CuO	25–950	75.7	80.9
2	2 H ₂ O	97–145	7.3	7.9
	Org. (→CuO+KBr)	145–525	49.7	48.9
	KBr	625–797	24.9	25.9
	Total reaction→CuO	25–950	83.3	82.7
Nitrogen atmosphere				
1	3 H ₂ O	25–123	13.0	13.0
	Org. (→CuO+KF)	123–618	50.6	53.9
	KF + 1/2O ₂	618–891	23.2	17.8
	Total reaction→Cu	25–900	86.8	84.7
2	2 H ₂ O	84–136	7.9	7.9
	Org. (→Cu+KBr)	136–608	48.0	52.4
	KBr	608–830	25.4	25.9
	Total reaction→Cu	25–900	82.2	86.2

Table 2. Crystallographic experimental data for [Cu(trisH₋₁)₂]KF·3H₂O (1) and [Cu(trisH₋₁)₂]KBr·2H₂O (2).

Compound	1	2
Unit cell determination		
Formula	CuKFO ₉ N ₂ C ₈ H ₂₆	CuBrKO ₈ N ₂ C ₈ H ₂₄
Formula weight	415.94	458.84
Color	Light brown	Brown
Crystal size/mm	0.25 × 0.20 × 0.15	0.28 × 0.03 × 0.03
T/°C	21 ± 1	21 ± 1
Reflections for lattice measurement	25	25
θ-Range for lattice measurement/°	7–13	4–13
Crystal system	Orthorhombic	Orthorhombic
a/Å	18.814(2)	18.482(2)
b/Å	9.623(1)	9.805(1)
c/Å	8.678(2)	8.776(3)
V/Å ³	1571.2(4)	1590.4(5)
Z	4	4
d _{calc} /g cm ⁻³	1.76	1.92
λ(MoK _α)/Å	0.710 73	0.710 73
μ(MoK _α)/Å	17.11	41.62
F(000)	868	932
Space group	Pcca(No. 54)	Pcca(No. 54)
Data collection and refinement		
θ range for data collection/°	2–30	2–30
Scan method	ω/2θ	ω/2θ
Scan speed in ω/° min ⁻¹	0.92–5.50	0.59–5.50
Scan width in ω/°	0.60 + 0.34 tan θ	0.50 + 0.34 tan θ
No. of measured reflections	2630	2662
Reflections used in refinement, I > 3σ(I)	1339	1132
Absorption correction (min./max.)	0.71/1.41	0.86/1.25
Max. shift/error	0.00	0.00
Max. in final Δρ/e Å ⁻³	0.48	0.58
No. of parameters refined	102	98
R	0.040	0.036
R _w ^a	0.048	0.040
S = [Σw(F _o - F _c) ² / (n - m)] ^{1/2}	2.048	2.381

$$^a w = 1/\sigma^2(F_o).$$

tions were monitored every 60 min as an intensity check, and the crystal orientation was confirmed after every 500 reflections. The total losses in intensity during data collection were 1.0% in 29.8 h (F) and 2.1% in 36.6 h (Br). The data obtained were corrected for linear decay as well as Lorentz and polarization effects. Absorption corrections were made according to Walker and Stuart.⁷

Positions of the heavy atoms (Cu, Br) in the bromide compound were solved by direct methods (SHELXS-86).⁸ The remaining non-hydrogen atoms were located by subsequent electron density calculations and refined by full-matrix least-squares methods with anisotropic thermal parameters. The hydrogens attached to carbons and nitrogens were included in their calculated positions after isotropic refinement (C–H, N–H = 0.95 Å), and the hydrogens in hydroxy groups and water molecules were located from a difference Fourier map, after anisotropic refinement. All hydrogens were refined as riding atoms with a fixed isotropic temperature parameter $B = 5.00 \text{ \AA}^2$.

The structure of the bromide compound was determined first because the results from the direct methods were clear. In the case of the fluoride compound, the disorder of fluorides and the smaller number of heavy atoms made it difficult to obtain a conclusive solution from the

direct methods, so we took advantage of the fact that the two compounds are almost isomorphous and used the non-hydrogen atom coordinates of the bromide compound as a starting set in the refinement of the fluoride compound.

Table 3. Atomic positional parameters and equivalent isotropic temperature factors^a with e.s.d.s in parentheses for [Cu(trisH₋₁)₂]KF·3H₂O (1).

Atom	x	y	z	B _{eq} /Å ²
Cu100	0.500	0.000	0.000	1.51(1)
O111	0.4144(2)	-0.0660(3)	-0.0906(4)	1.97(6)
O112	0.3308(2)	0.2703(4)	-0.2336(4)	3.06(7)
O113	0.3237(2)	0.3780(4)	0.1028(4)	2.39(6)
N111	0.4403(2)	0.1493(4)	0.0889(4)	1.61(6)
C111	0.3539(2)	0.0132(5)	-0.0433(5)	1.96(7)
C112	0.3757(2)	0.1618(4)	-0.0058(6)	1.48(6)
C113	0.3947(3)	0.2402(5)	-0.1520(6)	2.06(9)
C114	0.3150(3)	0.2318(5)	0.0815(6)	2.09(8)
K1	0.250	0.500	-0.1407(2)	2.36(2)
F1 ^b	0.4644(5)	0.4675(9)	0.097(1)	5.9(2) ^c
OW1	0.3704(2)	0.6733(4)	-0.0329(5)	3.41(8)
OW2 ^b	0.4654(6)	0.447(1)	0.160(2)	6.2(3) ^c

^a $B_{eq} = \frac{1}{3} \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$. ^b Disordered fluoride and water were refined one at a time while the other one was fixed. ^c pp = 0.50.

Table 4. Atomic positional parameters and equivalent isotropic temperature factors^a with e.s.d.s in parentheses for [Cu(trisH₋₁)₂]KBr·2H₂O (2).

Atom	x	y	z	B _{eq} /Å ²
Cu100	0.500	0.000	0.000	1.28(1)
O111	0.4101(2)	-0.0611(4)	-0.0842(5)	1.80(7)
O112	0.3354(2)	0.2836(5)	-0.2120(5)	2.68(9)
O113	0.3265(2)	0.3750(4)	0.1250(5)	2.08(7)
N111	0.4409(2)	0.1420(5)	0.1032(5)	1.48(8)
C111	0.3510(3)	0.0185(6)	-0.0376(6)	1.5(1)
C112	0.3748(3)	0.1615(5)	0.0094(6)	1.20(8)
C113	0.3977(3)	0.2455(6)	-0.1280(7)	1.9(1)
C114	0.3150(3)	0.2320(6)	0.0992(6)	1.6(1)
Br1	0.500	0.44931(8)	0.250	2.42(1)
K1	0.250	0.500	-0.1159(2)	2.05(3)
OW1	0.3704(2)	0.6812(4)	-0.0126(5)	2.50(8)

^a See Table 3.

After the refinement of the fluoride disorder was complete, the *R*-value was still 0.051, and there was a maximum of 1.28 e Å⁻³ in the residual electron density map, which proved to be an extra disordered water molecule (TG analysis also confirmed the amount of water). The coordinates of these disordered fluorides and water are very close, so simultaneous anisotropic refinement of both atoms was impossible. In all other respects, the procedures for the fluoride and bromide structure determinations were identical.

All calculations were done on a MicroVAX 3100 computer using MolEN⁹ software. The atomic scattering factors, including the contribution from anomalous dispersion, were taken from Ref. 10. The figures were drawn with the SCHAKAL¹¹ program. The final atomic positional parameters and equivalent isotropic temperature factors for non-hydrogen atoms are given in Tables 3 and 4. The molecular bond distances and angles for the complexes are reported in Tables 5 and 6 and the distances associated with the coordination sphere of potassium ion are shown in Table 7. Tables of anisotropic thermal parameters, coordinates of hydrogen atoms and listings of observed and calculated structure factors are available from the authors on request.

Table 5. Bond distances (in Å) with e.s.d.s in parentheses for [Cu(trisH₋₁)₂]KF·3H₂O (1) and [Cu(trisH₋₁)₂]KBr·2H₂O (2).

Bond	1	2
Cu100—O111	1.901(3)	1.915(4)
Cu100—N111	1.980(4)	1.988(4)
O111—C111	1.429(5)	1.404(6)
O112—C113	1.425(6)	1.418(7)
O113—C114	1.429(6)	1.436(7)
N111—C112	1.472(6)	1.486(7)
C111—C112	1.523(6)	1.526(7)
C112—C113	1.520(7)	1.520(8)
C112—C114	1.526(6)	1.524(7)

Table 6. Bond angles (in °) with e.s.d.s in parentheses for [Cu(trisH₋₁)₂]KF·3H₂O (1) and [Cu(trisH₋₁)₂]KBr·2H₂O (2).

Angle	1	2
O111—Cu100—N111	85.6(1)	85.3(2)
O111—Cu100—N111 ^a	94.4(1)	94.7(2)
Cu100—O111—C111	112.3(3)	112.9(3)
Cu100—N111—C112	108.1(3)	106.8(3)
O111—C111—C112	110.4(3)	111.4(4)
O112—C113—C112	108.5(4)	109.2(4)
O113—C114—C112	114.4(4)	114.7(4)
N111—C112—C111	105.3(3)	105.6(4)
N111—C112—C113	108.2(3)	106.3(4)
N111—C112—C114	112.1(4)	111.6(4)
C111—C112—C113	110.5(4)	111.3(4)
C111—C112—C114	108.7(3)	110.3(4)
C113—C112—C114	111.8(4)	111.5(4)

^a Symmetry operation applied is $-x, -y, -z$.

Table 7. Bond distances (in Å) of potassium coordination sphere for [Cu(trisH₋₁)₂]KF·3H₂O (1) and [Cu(trisH₋₁)₂]KBr·2H₂O (2).

Bond	1	2
K—O112	2.801(4)	2.776(5)
K—O113	2.787(4)	2.822(4)
K—O113 ^a	2.873(4)	2.945(4)
K—OW1	2.965(4)	2.988(4)

^a Symmetry operation applied is $x, 1-y, z-0.5$.

Results and discussion

Thermal analysis. The thermal behaviour of the complexes under study is summarized in Table 1. The decomposition process of both complexes was very similar in both atmospheres. The degradation happened in four basic steps; first, the crystalline water was dehydrated in one step, the organic part was then decomposed in two phases and finally potassium halide was sublimated at an elevated temperature (600–900°C). The final product in an air atmosphere was CuO, and metallic copper in a nitrogen atmosphere.

In an air atmosphere the organic part decomposed in two separate steps; the main reaction happened at 125–221°C (F) or 145–228°C (Br) and the second, smaller

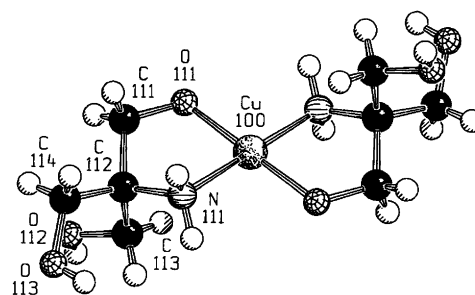


Fig. 1. SCHAKAL projection of the molecule in both structures (coordinates taken from 2).

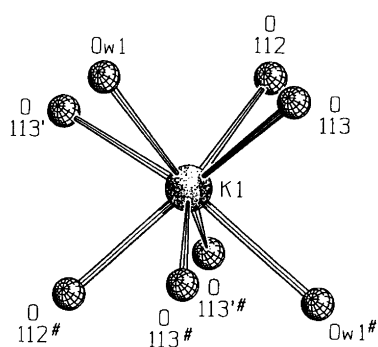


Fig. 2. Coordination sphere of potassium in both structures (coordinates taken from **2**). Symmetry operation applied for O113' is $x, 1-y, z-0.5$, and the other half of the coordination sphere (marked with #) is generated by symmetry operation $0.5-x, 1-y, z$.

reaction took place at 400–500°C. In a nitrogen atmosphere these reactions occurred in the same temperature area at 130–280°C, the result being an overlapped DTG peak with two maxima for the organic ligand. At 280°C 74.5% (F) or 86.3% (Br) of the organic part has decomposed, and above 280°C the decomposition of the remaining organic residue was very slow. The sublimation temperature of potassium halide was not much affected by the atmosphere, but the corresponding DTG peaks were narrower in air. Furthermore, the temperature range in which alkaline halides start to sublimate was checked with KBr, NaCl and NaF under the same conditions.

TG analysis clearly shows that the water content of the fluoride compound is higher than in the bromide compound. On the other hand, the crystalline water in the

fluoride compound is very easily dehydrated, so the samples must be analyzed immediately after they are removed from the mother liquor.

Molecular structures. The complex molecule of the bromide compound is presented in Fig. 1, and the representative bond lengths and angles for both compounds are listed in Tables 5 and 6. The basic molecular structure in both compounds is a mononuclear neutral complex with two tris ligands coordinated to the copper atom via amino and deprotonated hydroxy groups. The asymmetric unit is half of the molecule, and the other half is generated by inversion with respect to the copper atom. The coordination sphere is a square-planar *trans* coordination, and the metal–ligand distances range from 1.901(3) (Cu–O) to 1.980(4) Å (Cu–N) in the fluoride compound; the corresponding distances in the bromide compound are 1.915(4) and 1.988(4) Å, respectively. The five-membered chelate rings are *envelope* conformers, where Cu100, O111, N111, and C111 define the plane and C112 is bent out of the plane. The deviations of C112 from this plane are 0.583(5) Å for **1** and 0.594(5) Å for **2**, and the whole molecule is an *anti* isomer, where the chelate rings are bent in opposite directions from the coordination plane. The terminal hydroxymethyl groups can take an axial (ax) or an equatorial (eq) position in relation to this chelate plane. The corresponding distances for C113 (ax) and C114 (eq) atoms from the plane are 2.090(5) and 0.196(5) Å in the fluoride compound and 2.095(6) and 0.252(6) Å in the bromide compound.

The bond lengths and angles shown in Tables 5 and 6 coincide with the previously reported structures of copper–tris complexes.^{2–6} The molecular structures

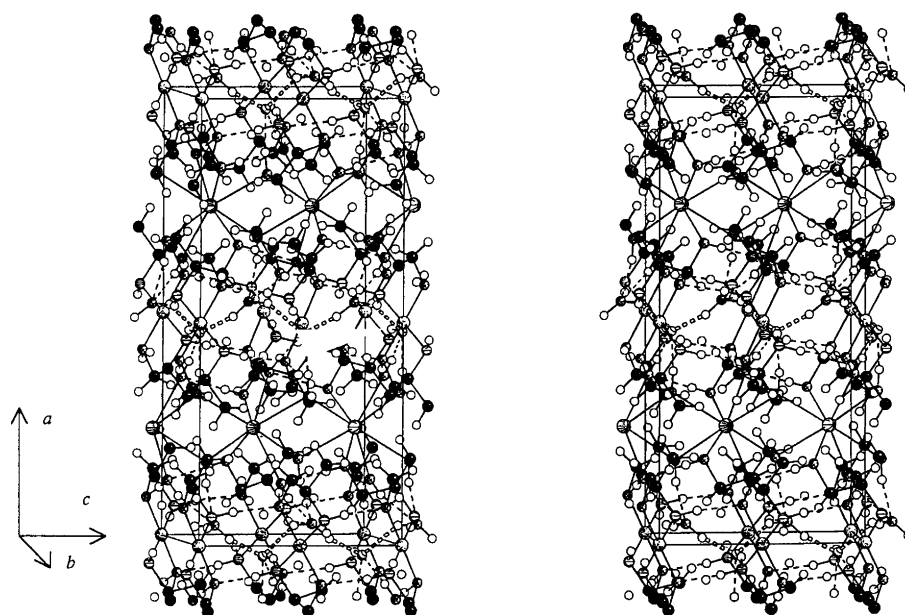


Fig. 3. A stereoscopic representation of $[\text{Cu}(\text{trisH}_{-1})_2]\text{KF}\cdot 3\text{H}_2\text{O}$ (**1**). Hydrogen bonds are indicated by dashed lines and only one orientation of disordered fluoride and OW2 is shown.

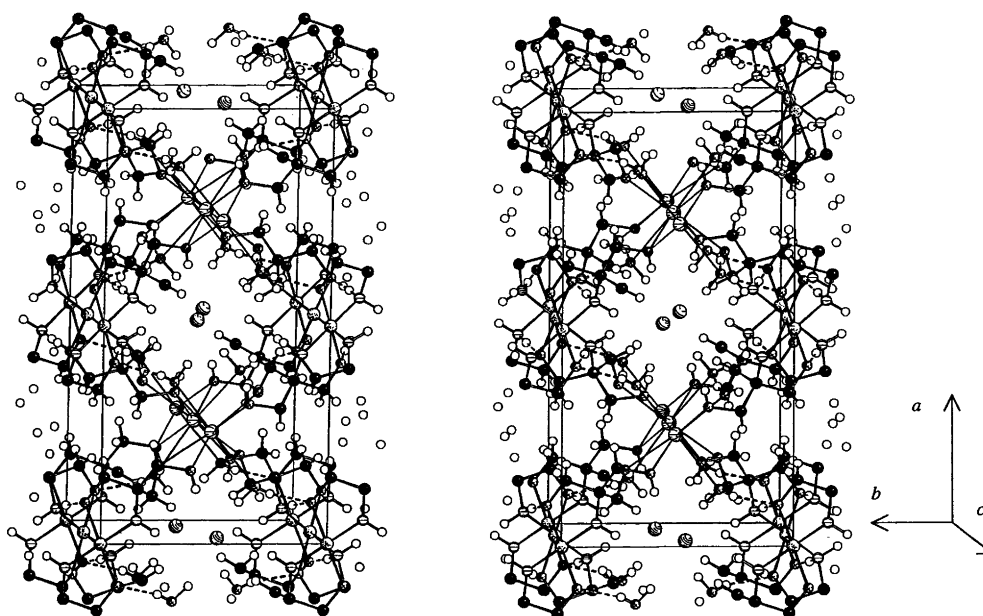


Fig. 4. A stereoscopic representation of $[\text{Cu}(\text{trisH}_{-1})_2]\text{KBr}\cdot 2\text{H}_2\text{O}$ (2). Hydrogen bonds are indicated by dashed lines.

of $[\text{Cu}(\text{trisH}_{-1})_2]$ and $[\text{Cu}(\text{trisH}_{-1})_2]\cdot 5\text{H}_2\text{O}^2$ in particular are almost identical with these complexes, differing mainly in the orientation of the terminal hydroxymethyl groups.

Crystal structures. The coordination sphere of the potassium ion is shown in Fig. 2, and stereoscopic projections of the crystal structures and hydrogen bonding are presented in Figs. 3 and 4.

Both compounds share the same basic structure in which the uncharged molecules are polymerized in the *c*-direction by $\text{N111}-\text{H}\cdots\text{O111}'$ hydrogen bonds, forming a zigzag chain, and the potassium(I) and halogenide ions occupy the hydrophilic and hydrophobic cavities, formed as a consequence of the polymerization. The crystalline water OW1 also assists in joining the molecules together; with one strong hydrogen bond the water molecule is connected to the deprotonated hydroxy oxygen ($\text{OW1}-\text{HW}\cdots\text{O111}$), and another hydrogen bond is formed between OW1 and the axial hydroxymethyl group of the adjacent complex molecule ($\text{OW1}\cdots\text{H}'-\text{O112}'$). In the direction of the *a*-axis the interactions between molecules are weak van der Waals forces between CH_2 groups.

The potassium ion plays a major role in connecting the molecular chains in all three directions. Potassium is located in a special position with multiplicity 4 (the general position has multiplicity 8), and it is surrounded by six hydroxy oxygens and two water molecules. The coordination sphere of potassium can be described as a square-antiprism with K–O distances falling in the range 2.776(5)–2.988(4) Å (Table 7). With these K–O bonds each potassium is directly linking four molecules together, and indirectly two more molecules via hydrogen-bonded

crystalline water. Similar K–O bond lengths and geometries have also been observed with other potassium complexes containing alkoxy, ether, carbonyl or amide oxygens and crystalline water.^{12–15}

The differences in the structures arise from the halides: their ionic radii and ability to form hydrogen bonds are not the same. The halogenides are located in the hydrophobic cavity where hydrogens are pointing inside the cavity. The bromide ion is large enough to fill the cavity, so there is no disorder, and each bromide is surrounded by six hydrogens with Br–H distances in the range 2.43–2.64 Å. On the other hand, the fluoride anion is so small that there are two centrosymmetrically related positions for fluoride in each cavity with 50% occupational probability. Furthermore, each fluoride is accompanied by an extra water molecule (OW2), which has almost the same coordinates as fluoride. Structurally, this means that in the cavities, every other position is taken by fluoride and every other by water molecule, and the whole hydrophobic tunnel is filled with a hydrogen-bonded chain of fluorides and water molecules, $\cdots\text{F1}\cdots\text{HW21}-\text{OW2}-\text{HW22}\cdots\text{F1}'\cdots\text{HW21}'-\text{OW2}'-\text{HW22}'\cdots$. The chain has two orientations, in which the positions of fluoride and water oxygen are reversed, and the probability of each orientation is 50%. The F \cdots O distances in the chain are 2.494 and 2.721 Å. In addition, this chain forms five hydrogen bonds per each F1 and OW2 pair with terminal hydroxy groups, amino hydrogens and OW1.

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Received 29 August, 1993.