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A Redetermination of the Structure of Strontium Chloride Hexahydrate, $SrCl_2.6H_2O$

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Abstract. $M_r = 266.64$, trigonal, $P321$, $a = 7.958$ (5), $c = 4.124$ (5) Å, $V = 226.2$ (4) Å^3, $Z = 1$, $D_m = 2.0$ (1), $D_x = 1.957$ (4) Mg m^{-3}, Mo $K\alpha$, $\lambda = 0.7107$ Å, $\mu = 6.39$ mm^{-1}, $F(000) = 132$, $T = 300$ K, $R = 0.020$ for 354 independent reflexions. Infinite columns of nine-coordinate Sr atoms lie along the threefold axes; three H_2O molecules are equatorially coordinated to each Sr atom while the other six H_2O molecules are also bound to adjacent Sr atoms. A helical system of hydrogen bonding involves both the H_2O molecules and the Cl^- ions.

Introduction. The structure of $SrCl_2.6H_2O$ was first described by Jensen (1940). His findings are confirmed by the present study, which was undertaken to investigate a possible orientational disorder of the equatorially bound water molecules. Adams & Trumble (1974) carried out a single-crystal Raman spectral study of $SrCl_2.6H_2O$ in which they inferred, from the tentative assignment of a Raman band to a ν(Sr—O) mode, that the hydrogen atoms of the equatorially bound H_2O molecules were disordered between the potential-energy minima corresponding to the existence of O—H···Cl hydrogen bonds. This positional disorder would reduce the instantaneous C_3 symmetry (but not, of course, the time-averaged symmetry exhibited by the reciprocal lattice) at the Sr atom causing an otherwise forbidden band to become allowed. Disorder of the hydrogen atoms between these alternative sites should be clearly detectable in difference electron density maps based on accurately refined coordinates for the strontium, chlorine and oxygen atoms.

Experimental. Crystal density in CCl_4 using Archimedes' principle; data collection from a needle 0·04 × 0·008 × 0·008 cm, Philips PW1100 diffractometer, graphite-monochromated Mo $K\alpha$, ω–2θ scan, width 1·1° θ, speed 0·04° θ s^{-1}, lattice parameters from 25 high-angle reflexions, three standard reflexions varied <4%, 1037 measured with $10 < 2\theta < 60°$, $(\sin\theta/\lambda)_{\max} = 0.70$ Å^{-1}, hkl: h $\bar{9}$–5, k 1–10, l 0–5, intensities corrected for Lp effects but not for absorption, 354 unique ($R_{\rm int} = 0.03$) with $I > \sigma(I)$ [$\sigma(I)$ previously defined by Gafner & Kruger (1974)]; structural analysis based on coordinates quoted by Jensen, refined by full-matrix anisotropic least squares based on F_c; H-atom positions from a difference map refined with isotropic temperature factors; $R = 0.020$, $R_w = 0.017$, $w = 0.559/\sigma^2(F_o)$; $(\Delta/\sigma)_{\max}$ in final cycle = 0·002, max. $\Delta\rho$ excursion in final difference Fourier synthesis within ± 0.5 e Å^{-3}; f, f' and f'' from *International Tables for X-ray Crystallography* (1974); program *SHELX76* (Sheldrick, 1976).

Discussion. Atomic coordinates and derived parameters are listed in Tables 1 and 2.*

The structure of the salt and the atomic nomenclature are shown in Fig. 1. Infinite columns of $Sr(H_2O)_6^{2+}$ units lie along threefold axes. Each Sr is coordinated by nine water molecules; six of these are bridging and three are equatorially bound. Each Cl^- ion

Table 1. *Atom coordinates* (×10^4) *and isotropic temperature factors* (Å^2 × 10^4 *for* Sr *and* Cl, *otherwise* Å^2 × 10^3) *with e.s.d.'s in parentheses*

	x	y	z	$U_{\rm eq}/U_{\rm iso}$
Sr	0	0	0	165 (2)*
Cl	3333	−3333	4192 (2)	312 (4)*
O(*e*)	3220 (4)	0	0	35 (2)*
O(*b*)	−2208 (4)	0	5000	25 (1)*
H(*e*)	3369 (55)	−869 (53)	516 (102)	61 (13)
H(*b*)	−3267 (56)	−945 (48)	5176 (96)	54 (13)

* $U_{\rm eq} = (U_{11}U_{22}U_{33})^{1/3}$.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s*

Sr—O(*e*)	2·562 (4)	Sr—O(*b*)	2·709 (2)
O(*e*)—H(*e*)	0·787 (34)	O(*b*)—H(*b*)	0·804 (37)
O(*b*)—Sr—O(*e*)	130·4 (1)	H(*e*)—O(*e*)—H(*e*)	108 (6)
O(*b*)—Sr—O(*b*)	68·3 (1)	H(*b*)—O(*b*)—H(*b*)	109 (5)

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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 39068 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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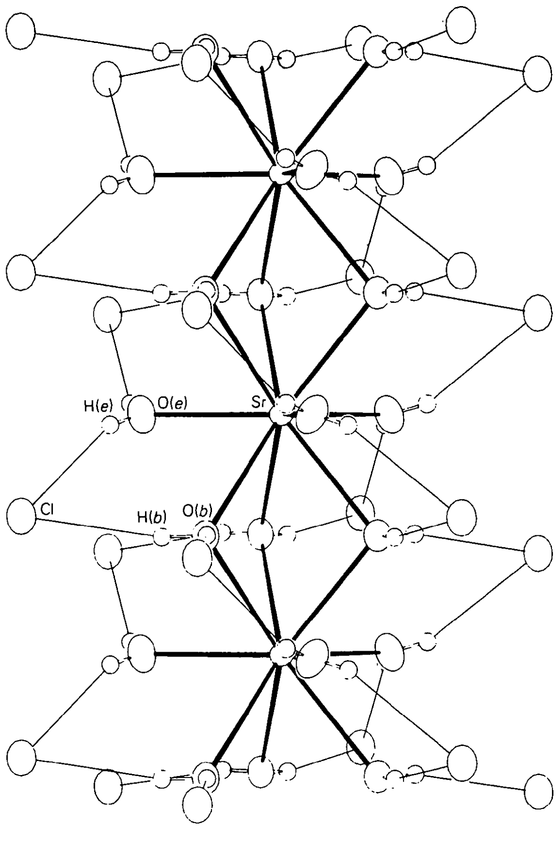

Fig. 1. Part of the $SrCl_2.6H_2O$ structure showing the atomic nomenclature and hydrogen bonding (indicated by thin lines).

is hydrogen-bonded to both an equatorial O [Cl···O(*e*) 3·205 (4), Cl···H(*e*) 2·47 (4) Å] and a bridging O [Cl···O(*b*′) 3·213 (4), Cl···H(*b*′) 2·44 (4) Å] in such a way that triple helices of [···H(*e*)–O(*e*)–H(*e*)···Cl···H(*b*)–O(*b*)–H(*b*)···Cl···] chains of hydrogen bonds spiral round the ···Sr···Sr··· columns. Each Cl^- ion takes part in three of these bonding systems, thus forming six hydrogen bonds.

Careful inspection of the final difference electron density map indicated no residual density >0·27 e $Å^{-3}$ in a region which would correspond to the alternative hydrogen position. As the peak for H(*e*) appeared in a difference map with a height of 0·85 e $Å^{-3}$, we conclude that the disorder proposed by Adams & Trumble (1974) is not present.

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Structure Determination of Diantimony Tritin Hexasulphide, $Sn_3Sb_2S_6$, by High-Resolution Transmission Electron Microscopy

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Abstract. $M_r = 792{\cdot}0$, orthorhombic, *Pnma*, $a = 23{\cdot}18\,(1)$, $b = 3{\cdot}965\,(1)$, $c = 34{\cdot}94\,(1)$ Å, $V = 3211$ Å^3, $Z = 12$, $D_x = 4{\cdot}91$ Mg m^{-3}, Cu $K\alpha_1$, $\lambda = 1{\cdot}54056$ Å, $\mu = 107{\cdot}2$ mm^{-1}, $F(000) = 4176$, $T = 295\,(3)$ K. The structure was determined by means of high-resolution transmission electron microscopy. $Sn_3Sb_2S_6$ has a block structure, consisting of lozenge-shaped units of TlI (*B*33) type structure related by glide reflection symmetry.

Introduction. High-resolution transmission electron microscopy (HRTEM) permits the direct imaging of crystal structures at resolutions down to approximately 2·5 Å (Iijima, 1971, 1975; Horiuchi, Matsui & Bando, 1976; Bursill & Wilson, 1977). Although such resolution is in general not quite sufficient to resolve individual atoms in projected crystal structures, HRTEM has been used to determine structures that are based on simpler building units whose structure can be inferred from the

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