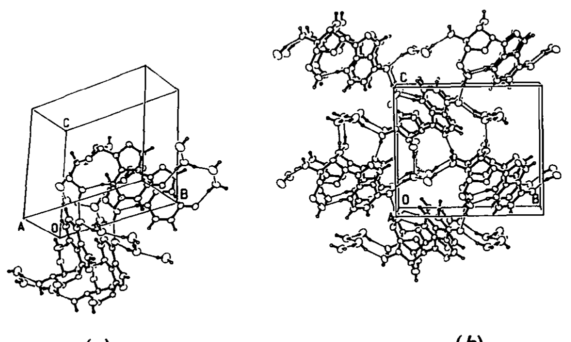

Fig. 7. (*a*) Unit-cell projection of (3) viewed perpendicular to the base plane of the shaded molecule. Sugar CH hydrogens have been omitted. Hydrogen bonds are indicated by thin lines. (*b*) Unit-cell projection viewed approximately along the *a* axis illustrating the channel occupied by the hydrogen-bonded chain of water molecules.

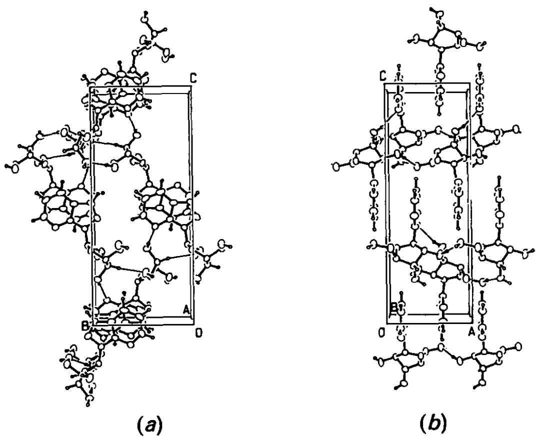

Fig. 8. Packing diagrams of unit cell of (4). Sugar CH hydrogens (except H3′) have been omitted. Hydrogen bonds are indicated by thin lines. (*a*) Projection along the *a* axis illustrating the base stacking and high degree of overlap. (*b*) Projection along the *b* axis showing the zipper-like base stacking and the lack of hydrogen bonding interactions in the *c* direction.

Fig. 8*b*), a feature resulting from the absence of hydrogen-bondable functions on the base ring opposite the ribose moiety.

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Isotypic Structures of (I) Tri(2-hydroxyethyl)ammonium Hydrogensulfide and (II) Tri(2-hydroxyethyl)ammonium Chloride

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Abstract. (I) $[NH(CH_2CH_2OH)_3]HS$, $M_r = 183{\cdot}27$, trigonal, $R3c$, $a = 8{\cdot}394\,(2)$, $c = 23{\cdot}513\,(4)$ Å, $V = 1434{\cdot}8$ Å^3, $Z = 6$, $D_x = 1{\cdot}27$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71069 Å, $\mu = 0{\cdot}291$ mm^{-1}, $F(000) = 600$, room temperature, $R = 0{\cdot}022$ for 278 unique observed reflections. (II) $[NH(CH_2CH_2OH)_3]Cl$, $M_r = 185{\cdot}65$,

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trigonal, $R3c$, $a = 8{\cdot}289\,(2)$, $c = 22{\cdot}946\,(3)$ Å, $V = 1365{\cdot}3$ Å^3, $Z = 6$, $D_x = 1{\cdot}35$ Mg m^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}382$ mm^{-1}, $F(000) = 600$, room temperature, $R = 0{\cdot}019$ for 435 unique observed reflections. The isotypic crystal structures consist of puckered layers of hydrogen-bonded cations, $[NH(CH_2CH_2OH)_3]^+$, and anions, SH^- or Cl^-. Within a layer every cation donates and every anion accepts three hydrogen bonds, O—H···S or O—H···Cl. The layers are stacked along the c axis in a sixfold repeating mode. Within the cations there are trifurcated intramolecular hydrogen bonds N—H(···O)$_3$.

Introduction. In the course of our studies of crystal structures of compounds with hydrogen-bonding functions we have carried out X-ray structure analyses of the salts (I) and (II). The investigations and their results are reported below.

Experimental. (I) and (II) were prepared by the reaction of triethanolamine (2,2′,2″-nitrilotriethanol) with hydrogen sulfide and hydrogen chloride, respectively. Single crystals were obtained by recrystallization from triethanolamine and hydrochloric acid, respectively.

X-ray measurements were performed on a Syntex $P2_1$ diffractometer. Graphite monochromator. Lattice constants from setting angles of 15 strong reflections. Intensities by variable ω-scan technique. Three standard reflections every 50 reflections showed only small random variations. No correction for absorption. Criterion for observed reflections $I > 1{\cdot}96\sigma_I$. (I) Crystal 0·2 × 0·2 × 0·1 mm. Lattice constants from reflections with $26 < 2\theta < 35°$. 754 reflections were measured up to $(\sin\theta)/\lambda = 0{\cdot}595$ Å^{-1} in the hemisphere $-10 \le h \le 0$, $-10 \le k \le 10$, $-10 \le l \le 10$, using a rhombohedral setting with $a = 9{\cdot}215\,(2)$ Å and $\alpha = 54{\cdot}19\,(2)°$. Transformation to hexagonal axes. 284 unique reflections after merging equivalent ones, $R_{\text{int}} = 0{\cdot}017$. Four unobserved reflections. Two reflections with too large intensities were excluded from structure refinement. (II) Crystal 0·2 × 0·1 × 0·1 mm. Lattice parameters from reflections with $27 < 2\theta < 38°$. 2827 reflections were measured up to $(\sin\theta)/\lambda = 0{\cdot}704$ Å^{-1} in the hemisphere $0 \le h \le 12$, $-12 \le k \le 12$, $-12 \le l \le 12$, using a rhombohedral setting with $a = 9{\cdot}022\,(1)$ Å and $\alpha = 54{\cdot}69\,(1)°$. Transformation to hexagonal axes. 449 unique reflections after merging equivalent ones, $R_{\text{int}} = 0{\cdot}040$. No unobserved reflections. 14 reflections with too large intensities were excluded from structure refinement.

Heavy-atom method. Full-matrix least-squares refinement, function minimized $\sum w(\Delta F)^2$, observed reflections only, weighted according to $w = [\sigma_F^2 + c|F_o|^2]^{-1}$. H atoms were located by difference-Fourier method. Complex atomic scattering factors from *International Tables for X-ray Crystallography* (1974). In final refinement one scale factor, coordinates of all atoms, and anisotropic (non-H atoms) and isotropic (H atoms) thermal parameters varied. (I) 57 parameters varied, $c = 0{\cdot}0004$, $wR = 0{\cdot}027$, $S = 1{\cdot}236$, Δ/σ in last cycle at most 0·01. $\Delta\rho_{\min}$ and $\Delta\rho_{\max}$ in final difference Fourier synthesis −0·12 and +0·10 e Å^{-3}, respectively. (II) 55 parameters varied, $c = 0{\cdot}0001$, $wR = 0{\cdot}026$, $S = 1{\cdot}588$, Δ/σ in last cycle at most 0·01. $\Delta\rho_{\min}$ and $\Delta\rho_{\max}$ in final difference Fourier synthesis −0·12 and +0·17 e Å^{-3}, respectively. Final atomic parameters of (I) and (II) are listed in Table 1.*

Table 1. *Atomic coordinates and (equivalent) isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

	x	y	z	U^*
(I)				
S	0·0	0·0	0·4	0·0424 (3)
O	0·2886 (3)	0·1945 (3)	0·1684 (1)	0·0598 (8)
N	0·0	0·0	0·0928 (1)	0·0265 (7)
C1	0·1862 (2)	0·1503 (2)	0·0738 (1)	0·0354 (8)
C2	0·3343 (3)	0·1648 (3)	0·1131 (1)	0·0412 (9)
H(S)	0·0	0·0	0·443 (4)	0·082 (22)
H(O)	0·383 (5)	0·236 (5)	0·185 (2)	0·066 (9)
H(N)	0·0	0·0	0·132 (3)	0·057 (16)
H11	0·208 (3)	0·122 (3)	0·038 (1)	0·032 (5)
H12	0·186 (4)	0·268 (5)	0·076 (1)	0·040 (6)
H21	0·448 (6)	0·272 (6)	0·100 (1)	0·076 (9)
H22	0·345 (4)	0·057 (4)	0·114 (1)	0·041 (6)
(II)				
Cl	0·0	0·0	0·4	0·0335 (1)
O	0·3003 (2)	0·1926 (2)	0·15893 (5)	0·0461 (4)
N	0·0	0·0	0·08230 (6)	0·0221 (4)
C1	0·1892 (1)	0·1502 (1)	0·06246 (6)	0·0290 (3)
C2	0·3407 (2)	0·1600 (2)	0·10147 (5)	0·0334 (4)
H(O)	0·400 (6)	0·236 (6)	0·179 (1)	0·076 (8)
H(N)	0·0	0·0	0·121 (2)	0·038 (10)
H11	0·203 (3)	0·115 (3)	0·024 (1)	0·034 (5)
H12	0·188 (3)	0·265 (4)	0·066 (1)	0·046 (4)
H21	0·454 (4)	0·264 (4)	0·089 (1)	0·054 (5)
H22	0·346 (3)	0·049 (3)	0·102 (1)	0·041 (4)

* $U = \frac{1}{3}(U_{11}a^{*2}a^2 + U_{12}a^*b^*ab\cos\gamma + \cdots)$ for the non-H atoms.

The calculations were performed with the program systems *XTL/XTLE* (Syntex, 1976) and *SHELXTL* (Sheldrick, 1983). The program *ORTEP*II (Johnson, 1976) was used to generate the illustrations.

Discussion. The salts (I) and (II) are isotypic. Each crystal structure consists of puckered layers of hydrogen-bonded cations, $[NH(CH_2CH_2OH)_3]^+$, and anions, SH^- or Cl^-, extended parallel to the ab plane. Within a layer every cation donates and every anion accepts three hydrogen bonds, O—H···S or O—H···Cl (all ions are located on threefold rotation

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* Lists of structure factors, anisotropic thermal parameters and bond lengths and angles involving H atoms, for (I) and (II), have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52488 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

axes). This is shown for (I) in Figs. 1 and 2. The H atoms of the anions SH^- are not involved in the hydrogen-bonding system. Along the *c* axis the layers are stacked in the sixfold repeating mode *AB′CA′BC′*.

In the cations the 2-hydroxyethyl groups surround the H atom bonded to the N atom. In this way trifurcated intramolecular hydrogen bonds $\mathrm{N{-}H({\cdots}O)_3}$ are formed, strongly bent at the proton. Interatomic distances and angles are listed in Table 2. The values are not corrected for thermal motion and the systematic error of X-ray methods to produce O—H, N—H and S—H (H···S, H···Cl and H···O) distances which are shorter (longer) than the internuclear separations.

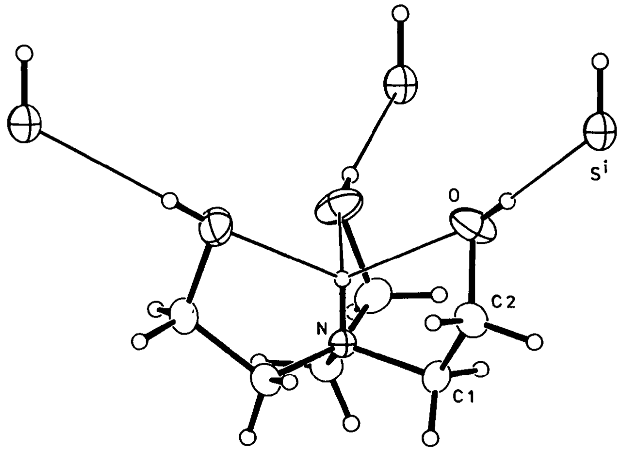

Fig. 1. The cation $[\mathrm{NH(CH_2CH_2OH)_3}]^+$, hydrogen-bonded to three anions SH^- in (I). Thermal ellipsoids correspond to the 50% probability level, H atoms are of arbitrary size.

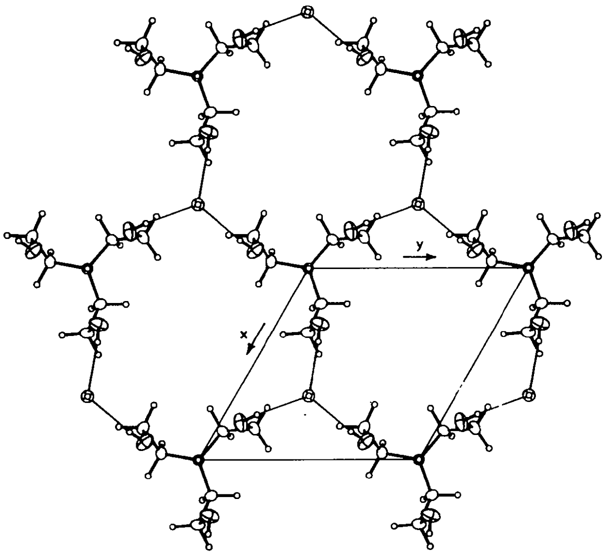

Fig. 2. Layer of hydrogen-bonded cations and anions in (I) viewed down the *c* axis.

Table 2. *Selected interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

(I)			
N—C1	1·505 (2)	C1—C2	1·504 (3)
C2—O	1·413 (3)	N···O	2·782 (3)
O···S$^{\mathrm{i}}$	3·172 (2)	N—H(N)	0·94 (6)
H(N)···O	2·30 (2)	O—H(O)	0·79 (4)
H(O)···S$^{\mathrm{i}}$	2·38 (4)	S—H(S)	1·01 (9)
C1—N—C1$^{\mathrm{ii}}$	111·5 (1)	N—C1—C2	110·8 (2)
C1—C2—O	107·1 (2)	C1—N—H(N)	107 (1)
N—H(N)···O	112 (2)	C2—O—H(O)	103 (2)
O—H(O)···S$^{\mathrm{i}}$	174 (4)		
(II)			
N—C1	1·505 (1)	C1—C2	1·511 (2)
C2—O	1·420 (2)	N···O	2·805 (2)
O···Cl$^{\mathrm{i}}$	3·156 (2)	N—H(N)	0·88 (4)
H(N)···O	2·36 (1)	O—H(O)	0·85 (4)
N(O)···Cl$^{\mathrm{i}}$	2·31 (4)		
C1—N—C1$^{\mathrm{ii}}$	111·3 (1)	N—C1—C2	111·0 (1)
C1—C2—O	107·0 (1)	C1—N—H(N)	108 (1)
N—H(N)···O	112 (2)	C2—O—H(O)	108 (2)
O—H(O)···Cl$^{\mathrm{i}}$	177 (4)		

Symmetry code: (i) $\frac{2}{3}-y,\ \frac{1}{3}-x,\ \frac{1}{3}-\frac{1}{2}+z$; (ii) $y-x,\ -x,\ z$.

Considering the N and S (or Cl) atoms only, the layers exhibit a similar structure to those in grey arsenic (six-membered rings with chair conformation and *trans*-fused in all directions). The geometry of the cations resembles that in three salts with organic anions (Starova, Frank-Kamenetskaya, Fundamenskii, Semenova & Voronkov, 1981; Shklover, Gridunova, Struchkov, Voronkov, Kryukova & Mirskova, 1983; van Mier, Kanters & Poonia, 1988) and also that of the molecules in crystals of the free base, triethanolamine (Mootz, Brodalla & Wiebcke, 1989).

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