Table 1. *Fractional atomic coordinates and* U_{eq} *values* (Å²)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N(1)	0·0217 (1)	0·1015 (2)	0·9689 (2)	0·044 (1)
N(2)	0·1432 (1)	−0·1976 (3)	0·5758 (3)	0·052 (1)
C(1)	0·1022 (1)	0·0356 (3)	0·7333 (3)	0·041 (1)
C(2)	0·1536 (2)	0·2298 (3)	0·6370 (3)	0·046 (1)
C(3)	0·2987 (2)	0·2424 (3)	0·5812 (3)	0·054 (1)
C(4)	0·3675 (2)	0·0568 (4)	0·3048 (3)	0·063 (1)
C(5)	0·3531 (2)	−0·2070 (4)	0·2686 (4)	0·061 (1)
C(6)	0·2171 (2)	−0·2878 (3)	0·2949 (3)	0·053 (1)

Table 2. *Bond distances* (Å) *and angles* (°)

N(1)—N(1′)	1·419 (2)	N(1′)—N(1)—C(1)	111·5 (1)
N(1)—C(1)	1·297 (2)	C(6)—N(2)—C(1)	126·1 (2)
N(2)—C(1)	1·356 (2)	N(2)—C(1)—C(2)	119·0 (1)
N(2)—C(6)	1·449 (2)	N(1)—C(1)—N(2)	123·5 (1)
C(1)—C(2)	1·491 (2)	N(1)—C(1)—C(2)	117·5 (1)
C(2)—C(3)	1·533 (2)	C(1)—C(2)—C(3)	114·8 (1)
C(3)—C(4)	1·520 (2)	C(2)—C(3)—C(4)	114·3 (1)
C(4)—C(5)	1·521 (3)	C(3)—C(4)—C(5)	115·4 (2)
C(5)—C(6)	1·517 (2)	C(4)—C(5)—C(6)	114·6 (1)
		C(5)—C(6)—N(2)	113·8 (1)

Symmetry code: (′) $-x$, $-y$, $2-z$.

Crystallography (1974, Vol. IV). Atomic parameters are given in Table 1,* bond distances and angles in Table 2. The molecule with atomic labelling is shown in Fig. 1.

Related literature. The azacycloheptane ring occurs in 1-azacycloheptane hydrochloride (Cameron & Scheeren, 1977). Numerous structures involving the azine C=N—N=C grouping have been described; only in a few of these are the C atoms part of a ring. In one of these there is an N atom in the 2-position of the ring, 4,5-dihydro-6-(2-thienyl)-3(2*H*)-pyridazinone azine (Will, Farag & El-Kordy, 1984). Others involving carbon rings are *N*,*N*′-bis[2-(4-chlorophenylamino)-2-cyclopenten-1-ylidene]hydrazine (Villa, Manfredotti, Guastini & Pocar, 1980) and bis(1,2,3,4-tetrahydro-6-methoxy-2-naphthylidene)hydrazinecarboximidamide sulfate monohydrate (Pitzele, Moormann, Gulliken, Albin, Bianchi, Palicharla, Sanguinetti & Walters, 1988).

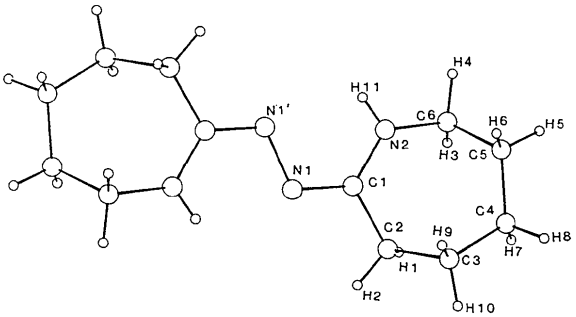

Fig. 1. View of molecule with atomic labelling.

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* Lists of structure factors, H-atom parameters, bond distances involving H atoms, anisotropic vibrational factors and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54352 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(2-aminoethyl)amine Perchlorate

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Abstract. $[N(CH_2CH_2NH_3)_3](ClO_4)_3$, $M_r = 447{\cdot}6$, cubic, $P2_13$, $a = 11{\cdot}858$ (5) Å, $V = 1667{\cdot}4$ Å^3, $Z = 4$, $D_x = 1{\cdot}78$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 5{\cdot}49$ cm^{-1}, $F(000) = 928$, $T = 293$ K, $R = 0{\cdot}038$ for 646 reflections with $I > 3\sigma(I)$. The cations and anions of the title compound all lie on a threefold axis of the cubic unit cell. There are significant hydrogen-bonding interactions between cation-NH_3^+ groups and perchlorate-O atoms, but no such cation–cation interactions.

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Experimental. Reaction of tris(2-aminoethyl)amine, tren, with perchloric acid in aqueous solution gives white microcrystalline $[N(CH_2CH_2NH_3)_3](ClO_4)_3$. However, good single crystals of this salt were obtained as a byproduct of an attempted preparation of an iron(II) complex of a Schiff base derived from tren and pyrrole-2-carboxaldehyde. The failure of this preparation is somewhat surprising in the light of the successful synthesis of the iron(III) complex of the hexadentate Schiff base from pyrrole-2-carboxaldehyde and trien, the open-chain analogue of tren (Rothin, Banbery, Berry, Hamor, Jones & McCleverty, 1980).

Intensities were measured from a crystal with dimensions 0·61 × 0·55 × 0·42 mm on a Stoe Stadi-2 diffractometer using graphite-monochromated Mo $K\alpha$ radiation. Accurate cell parameters were obtained by a least-squares refinement of the setting angles of 321 reflections in the angular range $7 \leq 2\theta \leq 30°$. Data were collected by ω-scan technique. 2521 reflections were measured over the range $7 \leq 2\theta \leq 54°$ with $-15 \leq h \leq 15$, $-15 \leq k \leq 15$, $0 \leq l \leq 15$. Monitored check reflections indicated no crystal movement or decay. The reflections merged to a unique set of 696 reflections with 646 having $I > 3\sigma(I)$ regarded as observed ($R_{\rm int} = 0.025$). Data were corrected for Lorentz and polarization effects. No absorption correction was applied to the data. All calculations were performed on a VAX 8650. Systematic absence: $h00$, $h \neq 2n$ only. This is consistent only with $P2_13$ or $P4_232$; refinement in the former proved successful. The structure was solved by direct methods using the *TREF* option of *SHELXS*86 (Sheldrick, 1986); all subsequent calculations used *SHELX*76 (Sheldrick, 1976) with full-matrix least-squares calculations on F magnitudes. All the non-H atoms were refined anisotropically. The H atoms of the NH_3 group were located from a difference Fourier map and the positional parameters refined. The remaining H atoms were included in calculated positions (C—H = 1·08 Å) with a common fixed isotropic parameter. Final $R = 0{\cdot}038$ and $wR = 0{\cdot}042$, $w = [\sigma^2(F) + 0{\cdot}002F^2]^{-1}$, ratio of max. (shift/$\sigma$) = 0·53, 85 parameters refined. Max. and min. peak heights in the difference Fourier map were 0·39 and $-0{\cdot}41$ e Å^{-3}. Atomic scattering factors as given in *SHELX*.

Final atomic coordinates are given in Table 1.* Selected bond distances and angles are in Table 2. Hydrogen-bonding interactions are shown in Fig. 1.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2) *for* $N(CH_2CH_2NH_3)_3.3ClO_4$

$U_{\rm eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{\rm eq}$
Cl(1)	0·27777 (7)	0·27777 (7)	0·27777 (7)	0·0318 (4)
Cl(2)	0·56188 (7)	0·56188 (7)	0·56188 (7)	0·0297 (4)
Cl(3)	0·04327 (8)	0·04327 (8)	0·04327 (8)	0·0329 (4)
O(1)	0·20763 (23)	0·20763 (23)	0·20763 (23)	0·0438 (12)
O(2)	0·3229 (3)	0·36705 (29)	0·2108 (3)	0·0605 (21)
O(3)	0·63278 (22)	0·63278 (22)	0·63278 (22)	0·0361 (10)
O(4)	0·6001 (3)	0·44813 (23)	0·57197 (28)	0·0497 (15)
O(5)	−0·0259 (3)	−0·0259 (3)	−0·0259 (3)	0·0790 (23)
O(6)	0·0121 (4)	0·1587 (3)	0·0301 (5)	0·0840 (28)
N(1)	0·21963 (23)	−0·21963 (23)	−0·21963 (23)	0·0232 (9)
C(1)	−0·1526 (3)	−0·1436 (3)	−0·2919 (3)	0·0307 (16)
C(2)	−0·2194 (4)	−0·0987 (3)	−0·3904 (3)	0·0353 (17)
N(2)	−0·2417 (3)	−0·1869 (3)	0·4759 (3)	0·0414 (17)
H(31)	−0·278 (4)	−0·248 (4)	−0·448 (4)	0·0500 (0)
H(32)	−0·271 (4)	−0·156 (4)	−0·530 (4)	0·0500 (0)
H(33)	−0·184 (5)	−0·222 (5)	−0·491 (5)	0·0500 (0)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

O(1)—Cl(1)	1·440 (5)	O(2)—Cl(1)	1·428 (3)
O(3)—Cl(2)	1·456 (4)	O(4)—Cl(2)	1·428 (3)
O(5)—Cl(3)	1·422 (6)	O(6)—Cl(3)	1·426 (4)
C(1)—N(1)	1·476 (4)	C(2)—C(1)	1·508 (5)
N(2)—C(2)	1·480 (5)	H(31)—N(2)	0·90 (5)
H(32)—N(2)	0·82 (5)	H(33)—N(2)	0·83 (5)
H(32)···O(2)	2·249	H(31)···O(3)	2·013

C(2)—C(1)—N(1)	112·5 (3)	C(1)—N(1)—C(1)	109·8 (2)
N(2)—C(2)—C(1)	112·0 (3)	H(31)—N(2)—C(2)	113·8 (28)
H(32)—N(2)—C(2)	107 (3)	H(32)—N(2)—H(31)	116 (4)
H(33)—N(2)—C(2)	111 (4)	H(33)—N(2)—H(31)	94 (5)
H(33)—N(2)—H(32)	115 (5)		

H atoms bonded to N2 were located and the positional parameters were refined. The remaining H atoms were included in calculated positions. The geometry of the perchlorate groups was normal.

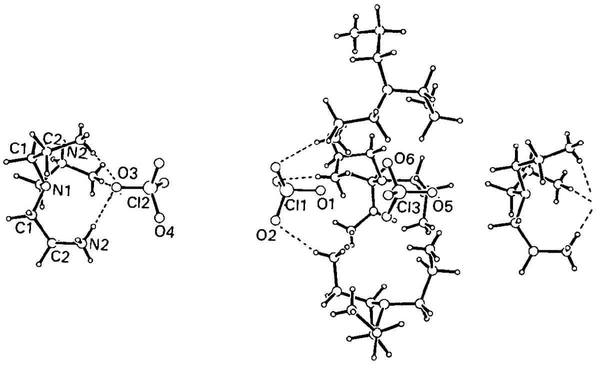

Fig. 1. Molecular geometry, atomic numbering and hydrogen bonds (dashed).

Related literature. There is significant hydrogen bonding between cation's three $—NH_3^+$ groups and one oxygen of the adjacent perchlorate anion. This and its neighbour have single hydrogen-bonding

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* Lists of structure factors, anisotropic thermal parameters, complete bond lengths and angles involving H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54334 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

interactions with $\mathrm{trenH_3^{3+}}$ cations to one side of the chain of ions depicted in Fig. 1, but the third perchlorate anion in the sequence has no significant hydrogen-bonding interactions at all. It is of interest that there is no hydrogen bonding between $\mathrm{trenH_3^{3+}}$ cations, in contrast to the situation in, for example, $[\mathrm{phen_2H}](\mathrm{ClO_4})$, where hydrogen bonds link pairs of 1,10-phenanthroline molecules (Maresca, Natile & Fanizzi, 1989).

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Structures of Two Crown-Ether Derivatives of 9-Acridone

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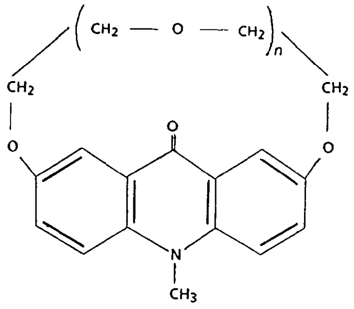
Abstract. (I) *N*-Methyl-2,5,8,11,14,17,20-heptaoxa-25-azatetracyclo[19.7.5.$0^{24,32}$,$0^{26,30}$]tritriaconta-1(29),-21(33),22,24(32),26(30),27-hexaen-31-one trihydrate, $C_{26}H_{33}NO_8.3H_2O$, M_r = 541·6, triclinic, $P\bar{1}$, a = 7·389 (1), b = 13·255 (1), c = 14·448 (2) Å, α = 88·60 (1), β = 78·58 (1), γ = 81·48 (1)°, V = 1371·7 (2) Å^3, Z = 2, D_x = 1·31 g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 8·68 cm^{-1}, F(000) = 580, T = 291 K, R = 0·047 for 3376 observed reflections. (II) *N*-Methyl-2,5,8,11,14,17-hexaoxa-22-azatetracylco[16.7.5.0.^{21,29}.$0^{23,27}$]triaconta-1(26),18(30),19,-21(29),23(27),24-hexaen-28-one, $C_{24}H_{29}NO_7$, M_r = 443·5, triclinic, $P\bar{1}$, a = 8·426 (2), b = 11·420 (2), c = 11·739 (4) Å, α = 87·20 (3), β = 79·33 (3), γ = 77·88 (2)°, V = 1085·3 (5) Å^3, Z = 2, D_x = 1·36 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 1·08 cm^{-1}, F(000) = 472, T = 291 K, R = 0·039 for 3229 observed reflections. In these substituted acridone derivatives one molecule contains both a crown ether moiety with its complexing capabilities and a heterocyclic N atom able to modify these properties. In the two compounds, the crown ether chain is nearly perpendicular to the acridinic skeleton which is planar. In (I) the water molecules take part in hydrogen bonds with the crown ether [O37···O11 = 3·03 (1), O35···O36 = 2·81 (1) Å] and with each other [O36···O37 = 2·93 (1), O38···O36 = 2·84 (1), O38···O37 (x + 1, y, z) = 2·89 (1) and O38···O37 (1 − x, 1 − y, 1 − z) = 2·88 (1) Å]. The bond lengths suggest partial localization of double bonds in the benzene rings.

Experimental. Crystals obtained by evaporation from ethanol. D_m not measured. Crystal sizes: (I) 0·31 × 0·22 × 0·13, (II) 0·2 × 0·2 × 0·4 mm. Lattice parameters refined using (I) 16, (II) 30 reflections in the range (I) 6 ≤ 2θ ≤ 38, (II) 6 ≤ 2θ ≤ 25°. Huber four-circle diffractometer, graphite-monochromated radiation, Cu $K\alpha$ for (I) and Mo $K\alpha$ for (II). For (I) 4936 $h \pm k \pm l$ independent reflections with $\sin\theta/\lambda \leq$ 0·60 Å^{-1}, 3376 with $I \geq 2{\cdot}5\sigma(I)$. For (II) 4269 $h \pm k$



(I) n = 5
(II) n = 4

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