

Tris(2-ammonioethyl)amine tribromide

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Key indicators

Single-crystal X-ray study

$T = 100$ K

Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å

R factor = 0.063

wR factor = 0.145

Data-to-parameter ratio = 33.4

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $[\text{N}(\text{CH}_2\text{CH}_2\text{NH}_3)_3]\text{Br}_3$, is a salt of a triprotonated tetramine. The tertiary amine N atom sits on a threefold axis, as do the three bromide anions. Each of the primary amines is protonated, while the tertiary amine is not protonated. The compound is isostructural with the chloride and the perchlorate analogs.

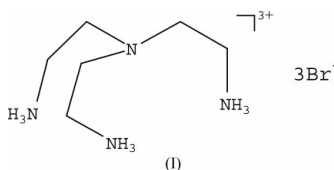
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Comment

During an attempted synthesis of $[\{\text{Ni}(\text{tren})\text{Br}_2\}(\text{ClO}_4)_2]$, where tren is tris(2-aminoethyl)amine, several tan crystals were noted amongst the predominantly purple product. These tan crystals proved to be the title compound, (I).



The title compound is isostructural (it has the same ordering of the cations and anions within the unit cell) with the analogous chloride (Ilioudis *et al.*, 2000) and perchlorate (Burgess *et al.*, 1991) salts. The tertiary amine N atom (N2) sits on a threefold axis, as do the three bromide ions. If the tertiary amine nitrogen is considered to be the origin, in all three structures the anions are located along the cube diagonal threefold axis at roughly the same positions: 0.28, 0.52 and 0.79 (expressed as a fraction of the diagonal). Intriguingly, for the chloride analog (Ilioudis, *et al.*, 2000), all three chloride ions were reported to hydrogen bond to the alkylammonium groups [the N–Cl distances are 3.176 (1), 3.223 (1) and 3.243 (2) Å], while for the perchlorate analog (Burgess *et al.*, 1991) only two of the perchlorate ions were reported to hydrogen bond to the alkylammonium groups [the N–O distances are 2.907 (5) and 3.016 (5) Å, while the non-bonded N–O separation is 3.225 (6) Å]. For the title compound, the three N–Br distances are 3.296 (5), 3.359 (4) and 3.395 (5) Å.

The distances within the H_3tren trication are the same, within experimental error, for all three structures.

Experimental

$[\text{Ni}(\text{H}_2\text{O})_6](\text{ClO}_4)_2$ (0.1839 g, 0.5048 mmol) was dissolved in 15 ml of CH_3CN , resulting in a blue solution. Tris(2-aminoethyl)amine (0.084 ml, 0.56 mmol) and HNET_3Br (0.0925 g, 0.508 mmol) were added to the solution, giving a white powder under a light blue solution. Methanol was added dropwise until the powder dissolved. The resultant blue–purple solution was placed in a desiccator, which

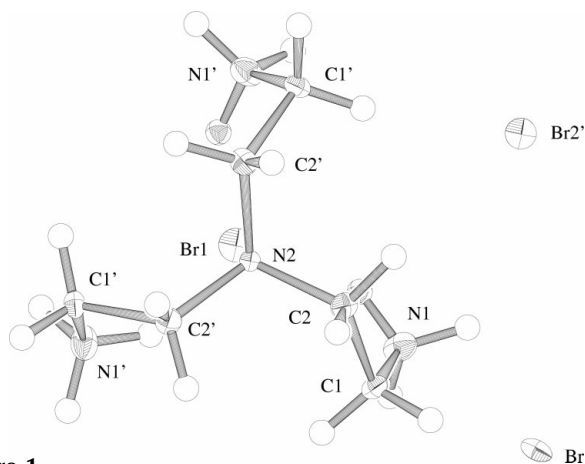


Figure 1
ORTEP view of the $\text{H}_3\text{trenBr}_3$ trication, with displacement ellipsoids drawn at the 50% probability level.

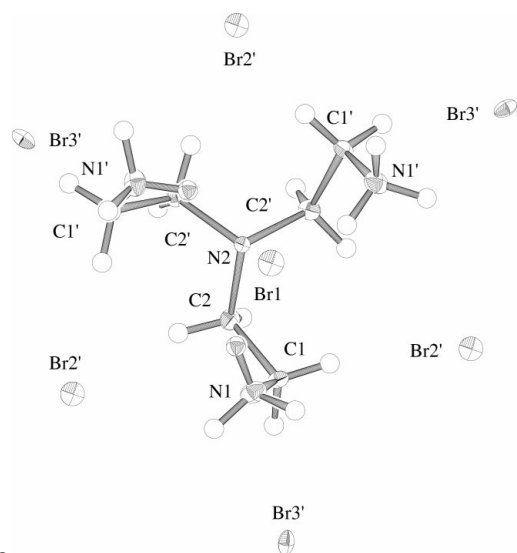


Figure 2
ORTEP view, showing the hydrogen bonding of one $\text{H}_3\text{tren}^{3+}$ trication with seven bromide ions.

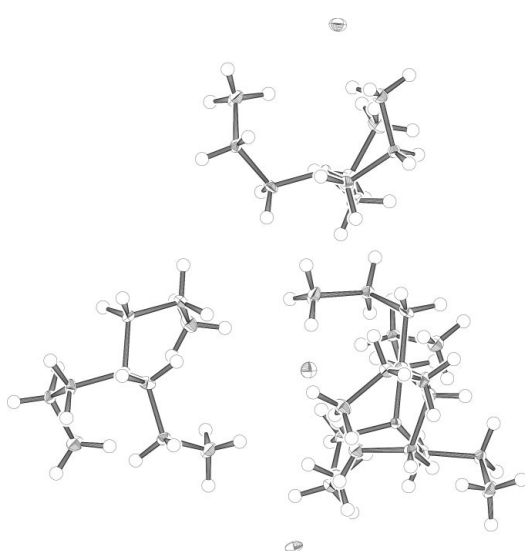


Figure 3
ORTEP view, showing one $\text{H}_3\text{tren}^{3+}$ trication (top) interacting with Br1, and three $\text{H}_3\text{tren}^{3+}$ trications interacting with Br2 and Br3 (bottom).

contained a beaker of conc. H_2SO_4 . Purple and tan crystals appeared after a few days. The tan crystals proved to be the title compound.

Crystal data

$\text{C}_6\text{H}_{21}\text{N}_4^{3+}\cdot 3\text{Br}^-$
 $M_r = 388.97$
 Cubic, $P2_13$
 $a = 11.038(2) \text{ \AA}$
 $V = 1344.8(4) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.921 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation

Cell parameters from 1167 reflections
 $\theta = 2.5\text{--}35.5^\circ$
 $\mu = 9.00 \text{ mm}^{-1}$
 $T = 100 \text{ K}$
 Fragment, tan
 $0.27 \times 0.22 \times 0.17 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer (with Oxford Cryostream)
 ω scans with κ offsets
 Absorption correction: multi-scan (HKL SCALEPACK; Otwinowski & Minor, 1997)
 $T_{\text{min}} = 0.164$, $T_{\text{max}} = 0.218$
 14942 measured reflections

1598 independent reflections
 1335 reflections with $I > 3\sigma(I)$
 $R_{\text{int}} = 0.042$
 $\theta_{\text{max}} = 35.5^\circ$
 $h = -18 \rightarrow 18$
 $k = -12 \rightarrow 12$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 $R[F^2 > 3\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.145$
 $S = 1.02$
 1335 reflections
 40 parameters
 H-atom parameters not refined

$w = 1/[\sigma^2(F_o) + 0.00331|F_o|^2]$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.73 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -1.36 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983), 1338 Friedel pairs
 Flack parameter = 0.0586 (6)

Table 1

Selected geometric parameters (\AA , $^\circ$).

N1—C1	1.501 (6)	C1—C2	1.512 (7)
N2—C2	1.468 (5)		
C2—N2—C2 ⁱ	109.1 (3)	N2—C2—C1	112.3 (4)
N1—C1—C2	110.7 (4)		

Symmetry code: (i) z, x, y .

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N1—H1 \cdots Br1	0.95	2.54	3.296 (5)	136
N1—H2 \cdots Br3	0.95	2.76	3.359 (4)	122
N1—H3 \cdots Br2	0.95	2.81	3.395 (5)	121

Symmetry codes: (ii) $x - \frac{1}{2}, \frac{1}{2} - y, -z$.

H atoms were treated as riding in idealized positions ($\text{C—H} = 0.95 \text{ \AA}$), with $U_{\text{iso}} = 1.2U_{\text{eq}}$ of the parent atom. While the compound itself is not chiral in solution, the crystal structure was determined (Flack, 1983). The deepest hole is located 0.053 \AA from atom N2. The orientation is away from where one might expect a H atom (that is, on the C2 side, toward Br2 not toward Br1).

Data collection: COLLECT (Nonius, 2000); cell refinement: DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO and SCALEPACK; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: teXsan for Windows (Molecular Structure Corporation, 1997–1999); software used to prepare material for publication: teXsan for Windows.

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