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Ethane-1,2-diammonium dibromide: a redetermination at 100 K

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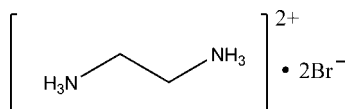
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.013; wR factor = 0.035; data-to-parameter ratio = 17.2.

In the redetermined [for the previous study, see Sjøtofte (1976). *Acta Chem. Scand. Ser. A*, **30**, 309–311] crystal structure of the title compound, $\text{C}_2\text{H}_{10}\text{N}_2^{2+} \cdot 2\text{Br}^-$, the H atoms have been located and the hydrogen-bonding scheme is described. The ethane-1,2-diammonium cation lies over a crystallographic inversion centre and straddles a crystallographic mirror plane with the C and N atoms in special positions. In the crystal, the cations and anions are linked by $\text{N}-\text{H} \cdots \text{Br}$ and $\text{N}-\text{H} \cdots (\text{Br}, \text{Br})$ hydrogen bonds, which generate various ring and chain motifs including an $R_{10}^5(32)$ loop.

Related literature

For the previous structure, see: Sjøtofte (1976). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For information on the Cambridge Database, see: Allen (2002).



Experimental

Crystal data

 $\text{C}_2\text{H}_{10}\text{N}_2^{2+} \cdot 2\text{Br}^-$ $M_r = 221.94$ Monoclinic, $C2/m$ $a = 15.144$ (2) Å $b = 4.7598$ (7) Å $c = 4.8146$ (7) Å $\beta = 101.323$ (2)° $V = 340.30$ (8) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 11.80$ mm⁻¹
 $T = 100$ K $0.36 \times 0.24 \times 0.20$ mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (AXScale; Bruker, 2010)
 $T_{\min} = 0.101$, $T_{\max} = 0.201$

3261 measured reflections
481 independent reflections
475 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.013$
 $wR(F^2) = 0.035$
 $S = 1.17$
481 reflections
28 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.62$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.39$ e Å⁻³

Table 1
Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N1}-\text{H2A} \cdots \text{Br1}$	0.83 (4)	2.89 (3)	3.324 (2)	115 (3)
$\text{N1}-\text{H2A} \cdots \text{Br1}^i$	0.83 (4)	3.00 (2)	3.4808 (14)	120 (1)
$\text{N1}-\text{H2B} \cdots \text{Br1}^{ii}$	0.88 (2)	2.48 (2)	3.3326 (14)	163 (2)

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

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINTE* (Bruker, 2010); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *pubCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5586).

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supplementary materials

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Ethane-1,2-diammonium dibromide: a redetermination at 100 K

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Comment

As part of our ongoing study of the structural characteristics of organic-inorganic layered diammonium salts, the crystal structure of ethane-1,2-diammonium dibromide, (I), was determined. A search of the Cambridge Structural Database (Version 5.31, May 2010 release; Allen, 2002) revealed that the crystal structure of (I) had been previously determined 34 years ago (Søtofte, 1976) at room temperature. The information in the CSD CIF file however appears incomplete and the author also states that the contributions from the hydrogen atoms in the structure was ignored. Here we report the redetermined structure of the title compound at 100 K. All the H atom positions were clearly visible in the difference Fourier map and they were independently refined with ADP's constrained to values of 1.2 and 1.5 times the isotropic U values of the C and N atoms on which they ride. We also show packing arrangements, hydrogen bonding interactions, hydrogen bonding motifs as well as calculated torsion angles (Table 3) that were previously not reported.

The ethane-1,2-diammonium cation lies over a centre of inversion and also straddles a mirror plane. The asymmetric unit contains one bromide anion and half of the ethane-1,2-diammonium cation (Figure 1).

Figure 2 illustrates the packing of the title compound viewed down the *b* axis. The ethane-1,2-diammonium cations are stacked above one another in the *ac* plane linked together by hydrogen bonds.

A close-up view of the hydrogen bonding interactions can be viewed in Figure 3. The hydrogen bond distances and angles for (I) can be found in Table 2. The hydrogen bonding network is three-dimensional and particularly complex, consisting of a variety of ring and chain motifs (identified using graphs sets in Mercury (Macrae *et al.*, 2006). Because of the complexity and number of different motifs identified, we focus on one particularly interesting hydrogen-bonding ring motif in the structure that appears to be in the shape of a T (Figure 4.) and it was chosen to best describe the highest level hydrogen bonding motif evident in the crystal structure.

Figure 4 shows a view of five diammonium cations and five bromide anions (viewed down the *c* axis) that are hydrogen bonded together to form a large, 32-membered T-shaped ring motif with graph set notation $R^5_{10}(32)$. Other ring motifs are evident - eight ring motifs and four chain motifs were identified from Mercury (Macrae *et al.*, 2006) but are not depicted here.

Experimental

Compound (I) was prepared by adding 1,2-diamino-ethane (0.50 g, 2.25 mmol) to 47% hydrobromic acid (HBr, 2 ml, 37.07 mmol, Merck) in a sample vial. The mixture was then refluxed at 363 K for 2 h. The solution was cooled at 2 K h⁻¹ to room temperature. Colourless blocks of (I) were collected.

Refinement

H atoms were clearly visible from the difference Fourier map. They were independently refined with the constraints $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{N})$. For (I), the highest peak in the final difference map is 0.80\AA from Br1 and the deepest hole is 0.98\AA from C1.

Figures

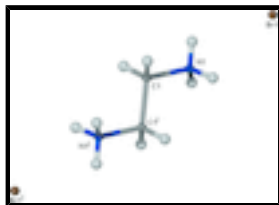


Fig. 1. : Molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. Atoms labelled with (i) are at the symmetry position $(1 - x, y, 1 - z)$

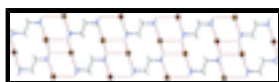


Fig. 2. : Packing arrangement of (I) viewed down the b axis and rotated slightly. Hydrogen bonds are indicated by dashed lines.

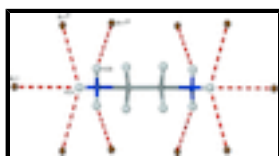


Fig. 3. : Close-up view of (I) viewed down the b axis with a slight offset clearly showing the hydrogen-bonding interactions. Hydrogen bonds are indicated by dashed lines.

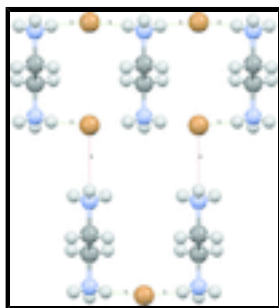
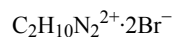


Fig. 4. : Close up view of (I) viewed down the c axis showing the T-shaped ring motif involving five diammonium cations and five bromide anions.

Ethane-1,2-diammonium dibromide

Crystal data



$$M_r = 221.94$$

Monoclinic, $C2/m$

Hall symbol: $-C 2y$

$$a = 15.144 (2) \text{\AA}$$

$$b = 4.7598 (7) \text{\AA}$$

$$c = 4.8146 (7) \text{\AA}$$

$$\beta = 101.323 (2)^\circ$$

$$V = 340.30 (8) \text{\AA}^3$$

$$Z = 2$$

$$F(000) = 212$$

$$D_x = 2.166 \text{ Mg m}^{-3}$$

$$\text{Mo K}\alpha \text{ radiation, } \lambda = 0.71073 \text{\AA}$$

Cell parameters from 2937 reflections

$$\theta = 2.7\text{--}28.4^\circ$$

$$\mu = 11.80 \text{ mm}^{-1}$$

$$T = 100 \text{ K}$$

Block, colourless

$$0.36 \times 0.24 \times 0.20 \text{ mm}$$

Data collection

Bruker APEXII CCD diffractometer	481 independent reflections
Radiation source: fine-focus sealed tube graphite	475 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.024$
Absorption correction: multi-scan (AXScale; Bruker, 2010)	$\theta_{\text{max}} = 28.4^\circ$, $\theta_{\text{min}} = 2.7^\circ$
$T_{\text{min}} = 0.101$, $T_{\text{max}} = 0.201$	$h = -20 \rightarrow 20$
3261 measured reflections	$k = -6 \rightarrow 6$
	$l = -6 \rightarrow 6$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.013$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.035$	$w = 1/[\sigma^2(F_o^2) + (0.0165P)^2 + 0.5674P]$
$S = 1.17$	where $P = (F_o^2 + 2F_c^2)/3$
481 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
28 parameters	$\Delta\rho_{\text{max}} = 0.62 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.39 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.051 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.47174 (14)	0.0000	0.3516 (4)	0.0119 (4)
N1	0.37420 (13)	0.0000	0.3658 (4)	0.0113 (4)
Br1	0.151104 (12)	0.0000	0.17162 (4)	0.01051 (13)
H1A	0.4815 (14)	0.166 (5)	0.246 (4)	0.013*

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H2A	0.344 (2)	0.0000	0.203 (8)	0.016*
H2B	0.3589 (15)	0.147 (5)	0.457 (5)	0.016*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0081 (9)	0.0184 (11)	0.0088 (9)	0.000	0.0007 (7)	0.000
N1	0.0097 (8)	0.0157 (9)	0.0079 (8)	0.000	0.0001 (7)	0.000
Br1	0.00972 (15)	0.01224 (16)	0.00940 (15)	0.000	0.00147 (8)	0.000

Geometric parameters (\AA , $^\circ$)

C1—N1	1.492 (3)	N1—H2A	0.83 (4)
C1—C1 ⁱ	1.515 (4)	N1—H2B	0.88 (2)
C1—H1A	0.97 (2)		
N1—C1—C1 ⁱ	109.8 (2)	C1—N1—H2A	109 (2)
N1—C1—H1A	106.2 (12)	C1—N1—H2B	112.5 (14)
C1 ⁱ —C1—H1A	112.3 (12)	H2A—N1—H2B	108.7 (19)

Symmetry codes: (i) $-x+1, -y, -z+1$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H2A \cdots Br1	0.83 (4)	2.89 (3)	3.324 (2)	115 (3)
N1—H2A \cdots Br1 ⁱⁱ	0.83 (4)	3.00 (2)	3.4808 (14)	120.(1)
N1—H2B \cdots Br1 ⁱⁱⁱ	0.88 (2)	2.48 (2)	3.3326 (14)	163 (2)

Symmetry codes: (ii) $-x+1/2, -y-1/2, -z$; (iii) $-x+1/2, -y+1/2, -z+1$.

Fig. 1

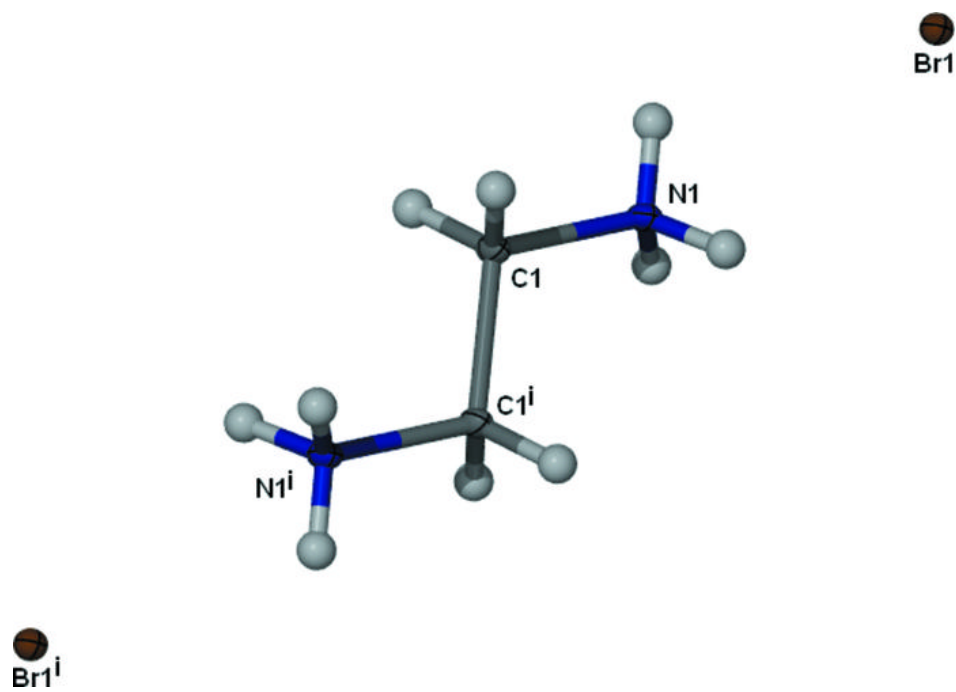


Fig. 2

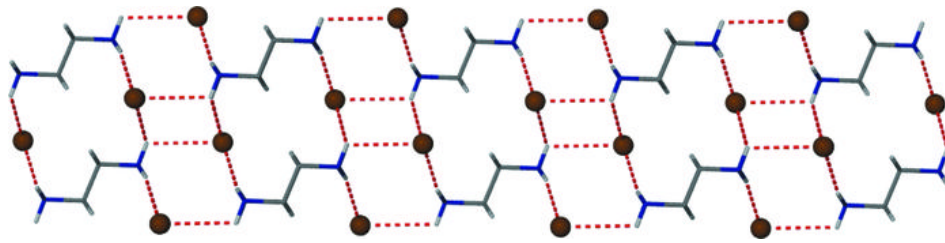


Fig. 3

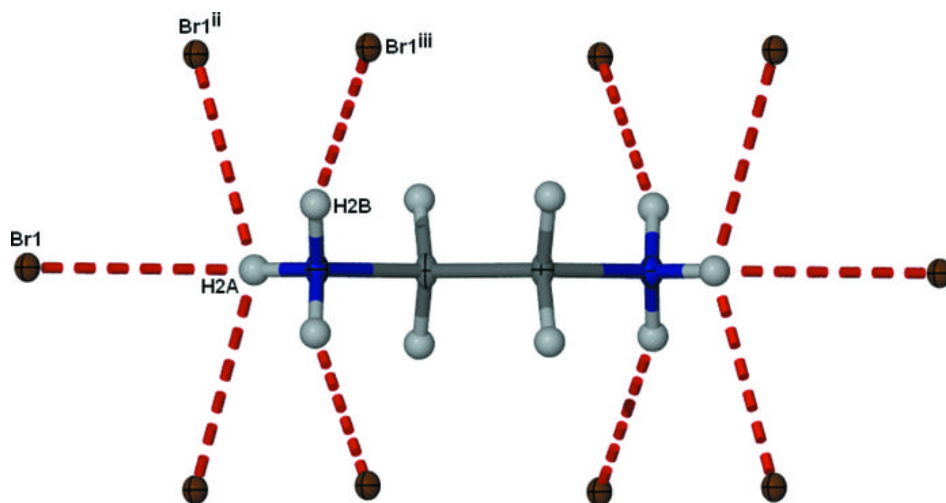


Fig. 4

