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Butane-1,4-diammonium diiodide

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Key indicators

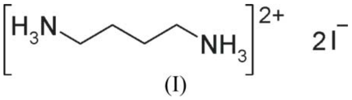
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.019
wR factor = 0.048
Data-to-parameter ratio = 24.5

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

The crystal structure of the title compound, $C_4H_{14}N_2^{2+}\cdot 2I^-$, exhibits ionic layers separated by hydrocarbon layers. The hydrocarbon layers stack in a herring-bone fashion and hydrogen bond to the iodide ions. The cation sits on a centre of inversion. The structure is compared with that of the related material butane-1,4-diammonium dichloride.

Comment

Organic–inorganic hybrid perovskites have been studied extensively. We are interested in determining the crystal strucures of simple monoammonium or diammonium hydrocarbon chains. While synthesizing the orange-coloured hybrid perovskite $(H_3NC_4H_8NH_3)PbI_4$ (Billing & Lemmerer, 2006), colourless crystals of the title compound, (I), appeared in the solution while cooling. A search of the Cambridge Structural Database (Version 5.27, November 2005 release; Allen 2002) revealed that the crystal structure of the related material butane-1,4-diammonium dichloride was studied 26 years ago (Chandrasekhar & Pattabhi, 1980). The two structures are isostructural and form the same two-dimensional hydrogen-bonding network in the *bc* plane. The chloride, however, has an extra short N···Cl contact that links the two-dimensional layers along the *a* direction.



(I)

In (I), the butane-1,4-diammonium cation sits on a centre of inversion, and, hence, the asymmetric unit contains one-half of the cation together with one anion (Fig. 1). Fig. 2 clearly indicates a monodimensional arrangement in which a single layer of butane-1,4-diammonium cation is embedded between two consecutive ionic layers, forming an alternating hydrocarbon–ionic structure in the *ab* plane. Each hydrocarbon layer is hydrogen bonded to the iodide ions above and below it. Perpendicular to this plane, the butane-1,4-diammonium cations pack in a herringbone arrangement (Fig. 3).

In the crystal structure, the intermolecular N—H···I hydrogen bonds (Table 1) form an extensive network. Two ammonium H atoms, H1*A* and H1*B*, participate in hydrogen bonding, forming rings with the graph-set notation $R_2^2(8)$. Hydrogen-bonded rings with the graph-set notation $R_3^2(13)$, which involve all three ammonium H atoms, are also observed in (I). The smaller ring has the shape of a rectangle, and both the long and the short edges are shared with the larger ring, which has five sides (Fig. 3). The fifth side is made up of the

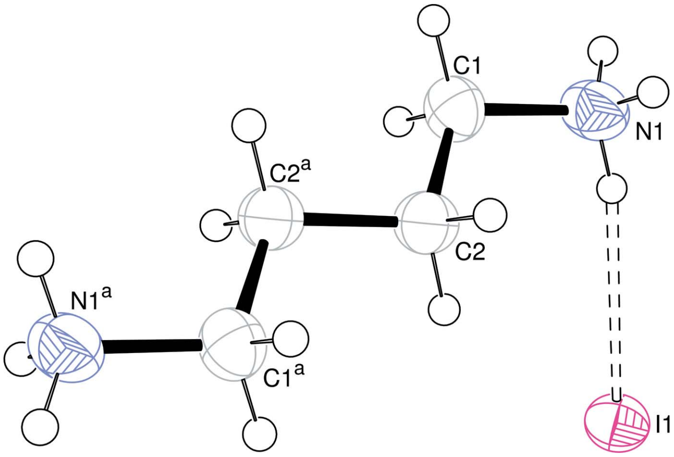


Figure 1
View of (I), with the atomic numbering scheme and displacement ellipsoids at the 50% probability level. Atoms labelled with the superscript a are at the symmetry position ($-x$, $-y$, $-z$). The dashed lines denote a hydrogen bond.

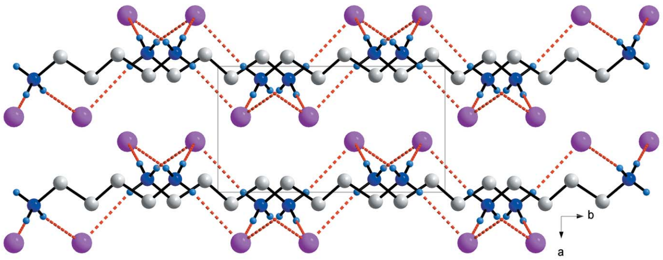


Figure 2
Packing diagram of (I). Each layer of butane-1,4-diammonium cations is sandwiched by a layer of iodide anions. The dashed lines denote hydrogen bonds.

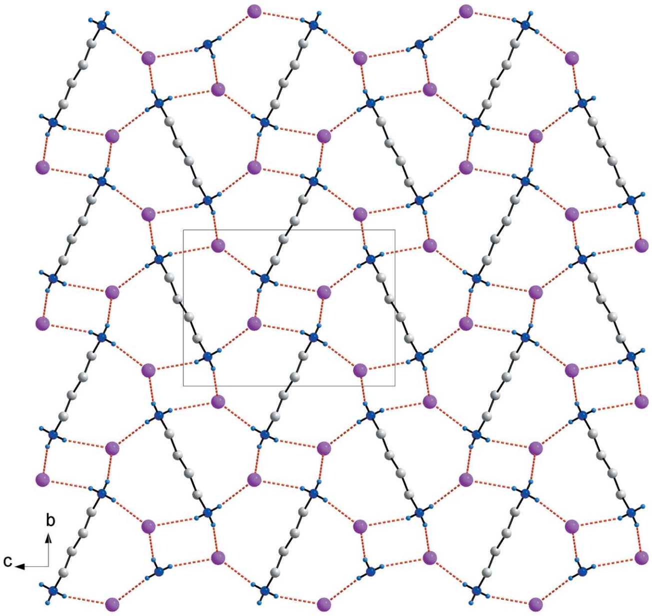


Figure 3
The two-dimensional hydrogen-bonded (dashed lines) network in (I), viewed down the *a* axis.

butane-1,4-diammonium cation. All the edges of the rings are shared to form an infinite two-dimensional network of hydrogen bonds in the *ac* plane.

Experimental

PbI_2 (0.148 g, 0.321 mmol) was dissolved in 47% HI (3 ml) in a sample vial. Thereafter, $C_4H_{12}N_2$ (0.060 g, 0.809 mmol) was added and the precipitate was dissolved by refluxing for 2 h at 363 K. The solution was cooled slowly at 2 K h^{-1} to room temperature. Colourless crystals of the title compound were separated from the orange crystals. Analysis calculated for $C_4H_{14}I_2N_2$: C 13.97, H 4.10, N 8.14%; found: C 13.78, H 4.28, N 8.14%.

Crystal data

$C_4H_{14}N_2^{2+}\cdot 2I^-$
$M_r = 343.97$
Monoclinic, $P2_1/c$
$a = 4.8983$ (8) Å
$b = 8.8210$ (14) Å
$c = 11.981$ (2) Å
$\beta = 93.711$ (3)°
$V = 516.61$ (15) Å^3
$Z = 2$
$D_x = 2.211$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 6.02$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.24 × 0.18 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: integration (*XPREP*; Bruker, 1999)
$T_{min} = 0.262$, $T_{max} = 0.455$
4077 measured reflections
954 independent reflections
890 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 25.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.048$
$S = 1.05$
954 reflections
39 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0253P)^2 + 0.1016P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.60$ e Å^{-3}
$\Delta\rho_{min} = -0.59$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.096 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···I1^{i}	0.89	2.84	3.650 (3)	152
N1—H1B···I1^{ii}	0.89	2.72	3.578 (2)	162
N1—H1C···I1	0.89	2.74	3.616 (3)	170

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

H atoms were geometrically positioned and refined in the riding-model approximation, with C—H = 0.97 Å, N—H = 0.89 Å, and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(N)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

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