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2,4,6-Trimethylanilinium iodide

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Key indicators

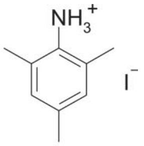
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.023
wR factor = 0.060
Data-to-parameter ratio = 21.0

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

In the crystal structure of 2,4,6-trimethylanilinium iodide, $C_9H_{11}NH_3^+\cdot I^-$, both ions are situated on mirror planes. The trimethylanilinium ions stack head-to-tail with no π–π interactions. The ammonium cations and I^- anions interact *via* hydrogen bonds, forming ladder-like columns parallel to the *b* axis. The structure is compared to the related anilinium iodide.

Comment

The crystal structure of 2,4,6-trimethylanilinium iodide, (I), is reported as part of a study of anilinium halide salts. The Cambridge Structural Database (Version 5.27, January 2006 release; Allen, 2002) contains the following anilinium iodides: *p*-fluoroanilinium iodide (Klebe *et al.*, 1983), pentadeuteroanilinium iodide (Fecher & Weiss, 1986), 2-methyl-4-nitroanilinium iodide (Lemmerer & Billing, 2006), 2-iodoanilinium iodide and 3-iodoanilinium iodide (Gray & Jones, 2002).



(I)

Both cation and anion lie on mirror planes. The atomic numbering scheme for (I) is shown in Fig. 1 and Fig. 2 shows the layers of 2,4,5-trimethylanilinium cations and I^- anions, alternating along the *a* axis. The cations are parallel to the *ac*

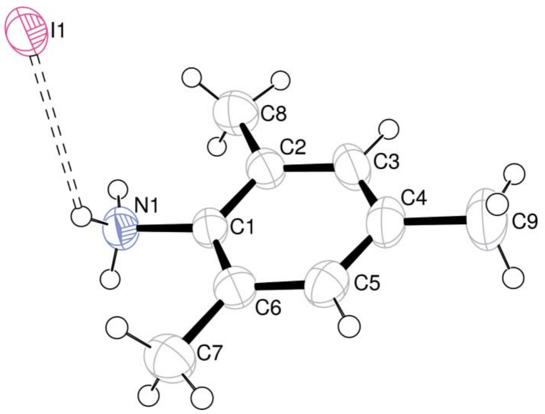


Figure 1
The cation and anion of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radius. The double dashed line indicates an N—H···I interaction.

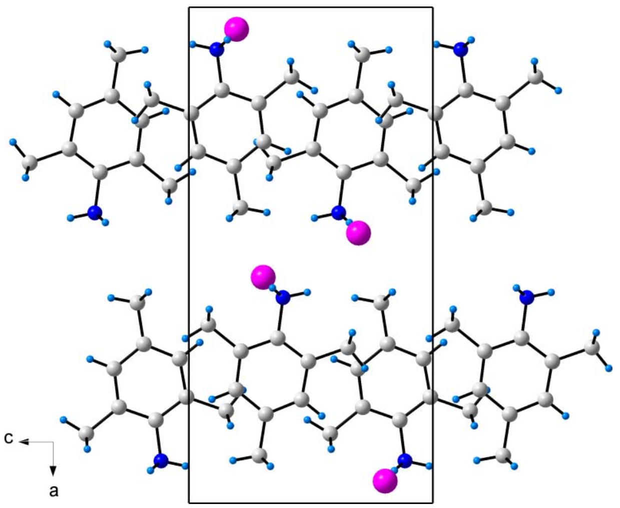


Figure 2
Packing diagram of (I), viewed along the *b* axis, with hydrogen bonds shown as red lines.

plane and are rotated by 8.33 (5)° away from the *a* axis, measured through the N1···C9 vector. Adjacent aromatic rings are separated by a centroid-to-centroid distance of 5.55 (2) Å, which is too long for π–π stacking interactions.

Compound (I) has a similar packing arrangement to anilinium iodide, (II) (Fecher & Weiss, 1986). In contrast to (I), in (II) the anilinium cations are situated on a general position and the angle between the planes through the cations is 47.2 (1)°, while the nearest centroid-to-centroid distance is 4.80 (2)°, with no π–π stacking interactions evident.

In the crystal structure of (I), the ions are linked together by N1—H1$B^{\rm i}$···I1···H1B—N1—H1$B^{\rm i}$···I1 hydrogen bonds, forming infinite chains parallel to [010] [symmetry code: (i) x, $\frac{1}{2} - y$, z]. Adjacent chains are linked in the [101] direction by an N1—H1A···I1 hydrogen bridge, forming a ladder-like column that extends along the *b* axis (see Fig. 3 and Table 1). This motif of ladder-like columns has previously been observed in 2-methyl-4-nitroanilinium iodide and 2-methyl-4-nitroanilinium bromide (Lemmerer & Billing, 2006) and is also a feature of (II).

Experimental

2,4,6-Trimethylaniline (0.060 g, 4.43 mmol) was added to 4 ml of 47% HI and the resulting precipitate was dissolved by refluxing at 363 K for 12 h. The solution was cooled slowly to room temperature at a rate of 2 K h^{-1}, giving colourless single crystals of (I) suitable for X-ray diffraction analysis.

Crystal data

$C_9H_{14}N^+\cdot I^-$
M_r = 263.11
Orthorhombic, *Pnma*
a = 17.814 (2) Å
b = 6.8175 (8) Å
c = 8.7449 (11) Å
V = 1062.0 (2) Å^3
Z = 4
D_x = 1.646 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.96 mm^{-1}
T = 293 (2) K
Plate, colourless
0.44 × 0.26 × 0.12 mm

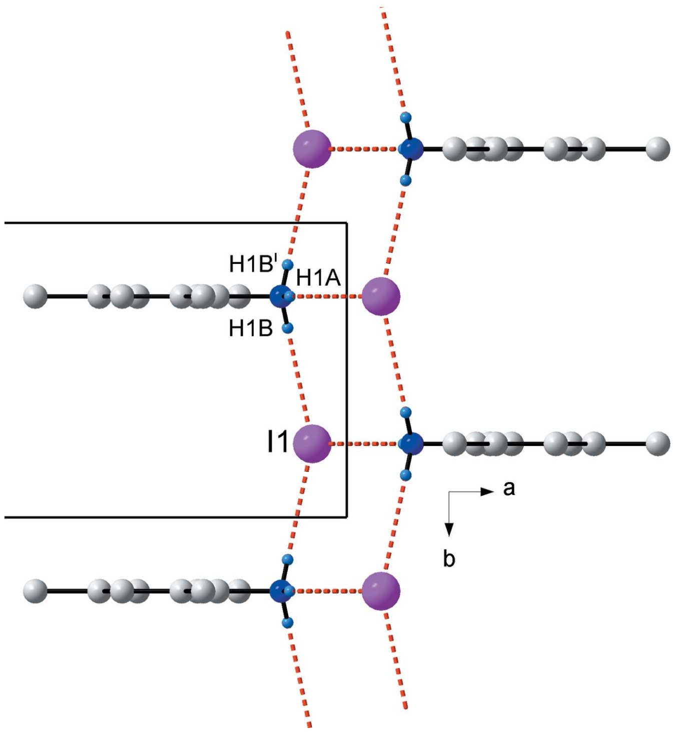


Figure 3
The columns of the hydrogen bonds (red lines) between the ammonium groups and the I$^-$ anions in (I). [Symmetry code: (i) x, $\frac{1}{2} - y$, z.]

Data collection

Bruker SMART 1K CCD area-detector diffractometer
ω scans
Absorption correction: integration (*XPREP*; Bruker, 1999)
$T_{\rm min}$ = 0.333, $T_{\rm max}$ = 0.708
7017 measured reflections
1426 independent reflections
1243 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.058
$\theta_{\rm max}$ = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.060
S = 1.06
1426 reflections
68 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0297P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.002
$\Delta\rho_{\rm max}$ = 0.44 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.67 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0045 (5)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···I1	0.89	2.73	3.5570 (8)	155
N1—H1*A*···I1$^{\rm i}$	0.89	2.81	3.562 (2)	143

Symmetry code: (i) $-x + 2, -y + 1, -z$.

All the H atoms were located in a difference Fourier map and then refined in idealized positions in the riding-model approximation, with C—H = 0.93 or 0.96 Å and N—H = 0.89 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(aromatic C), 1.5$U_{\rm eq}$(methyl C) or 1.5$U_{\rm eq}$(ammonium 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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