

Redetermination of trimethylammonium  
iodide at 93 KGraeme J. Gainsford\* and Tim  
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The crystal structure of the title compound,  $C_3H_{10}N^+ \cdot I^-$ , originally determined by Sheldrick & Sheldrick [*Acta Cryst.* (1970), **B26**, 1334–1338], has been redetermined at 93 K. Both ions lie on a mirror plane. They form  $N-H \cdots I$  hydrogen-bonded units in the crystal structure.

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## Key indicators

Single-crystal X-ray study

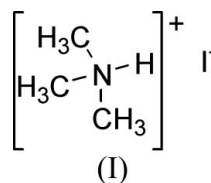
 $T = 93$  KMean  $\sigma(N-C) = 0.002$  Å $R$  factor = 0.010 $wR$  factor = 0.024

Data-to-parameter ratio = 14.4

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

## Comment

The title compound, (I), was inadvertently formed as a by-product from a reaction investigating the synthesis of amino-borane derivatives in tetrahydrofuran. The less soluble trimethylammonium iodide crystallized out as colourless crystals, leaving the basic cyclic aminoborane in solution. Although this reaction has not been reported previously, reactions of primary and secondary amines with boron halides have been documented (Nöth *et al.*, 1964).



The compound has been studied before at room temperature by film methods (Sheldrick & Sheldrick, 1970, and references therein). We present here a 'state of the art' low-temperature data set with a total data collection time of less

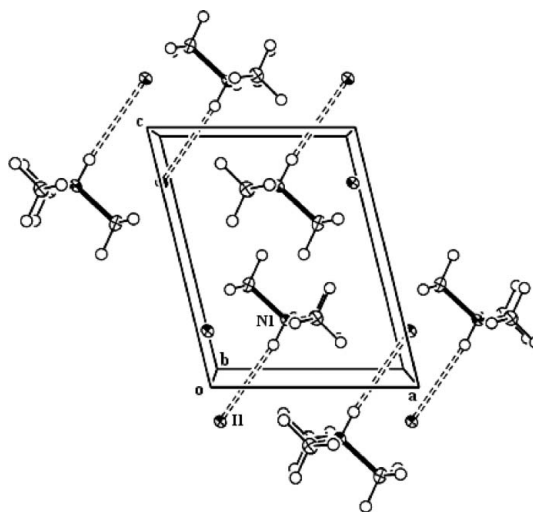


Figure 1

An ORTEP-3 (Farrugia, 1997) view, down the  $b$  axis, of the unit-cell contents of (I), shown with 50% probability displacement ellipsoids.  $N-H \cdots I$  hydrogen bonds are shown as dashed lines.

than 3 h and conventional *R* factor of 0.010, which is reflected in the ability to refine H atoms in the presence of the much larger scattering I atom given well defined crystals. I, NH and one CH lie on a mirror plane. The structure contains hydrogen-bonded units (Table 2 and Fig. 1), as indicated in the earlier study. The N—H...I angle, expected to be near linear, compares with the reported range for dimethylamino iodide structures (Cambridge Structural Database, Version 5.27, update of May 2006; Allen, 2002) of 157–179° [e.g. 175° in Schneider & Schier (2004)]. The film determination geometry, even without allowance for the different temperature of data collection, is statistically identical although of considerably lower resolution.

### Experimental

Iodine (6.61 g, 0.026 mol) was added in small aliquots to a solution of trimethylamine borane (1.9 g, 0.026 mol) containing ethylenediamine (0.78 g, 0.013 mol) in tetrahydrofuran (100 ml). On concentration of the solution, suitable crystals were deposited.

#### Crystal data

$C_3H_{10}N^+ \cdot I^-$	$Z = 2$
$M_r = 187.02$	$D_x = 1.969 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation
$a = 5.5309 (7) \text{ \AA}$	$\mu = 4.94 \text{ mm}^{-1}$
$b = 8.2737 (10) \text{ \AA}$	$T = 93 (2) \text{ K}$
$c = 7.1000 (9) \text{ \AA}$	Block, colourless
$\beta = 103.8010 (10)^\circ$	$0.37 \times 0.18 \times 0.16 \text{ mm}$
$V = 315.52 (7) \text{ \AA}^3$	

#### Data collection

Bruker SMART CCD area-detector diffractometer	1741 measured reflections
$\varphi$ and $\omega$ scans	735 independent reflections
Absorption correction: multi-scan (Blessing, 1995)	730 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.304$ , $T_{\max} = 0.454$	$R_{\text{int}} = 0.011$
	$\theta_{\text{max}} = 27.9^\circ$

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0077P)^2 + 0.1167P]$
$R[F^2 > 2\sigma(F^2)] = 0.010$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.024$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.23$	$\Delta\rho_{\text{max}} = 0.34 \text{ e \AA}^{-3}$
735 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
51 parameters	Extinction correction: <i>SHELXL97</i>
All H-atom parameters refined	Extinction coefficient: 0.045 (2)

**Table 1**

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

N1—C1	1.498 (3)	N1—H3	0.81 (3)
N1—C2	1.4977 (18)	C1—H1	0.94 (3)
C1—N1—C2	111.26 (11)	C2—N1—H3	108.7 (11)
C1—N1—H3	105 (2)		

**Table 2**

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H3...I1 <sup>i</sup>	0.81 (3)	2.70 (3)	3.500 (2)	173 (3)

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

All H atoms were refined with isotropic displacement parameters [ $N-H = 0.81 (3) \text{ \AA}$  and  $C-H = 0.94 (2)–1.00 (2) \text{ \AA}$ ].

Data collection: *SMART* (Siemens, 2001); cell refinement: *SAINT* (Siemens, 2001); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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