Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

N-Methyl-*N*-propylpyrrolidinium iodide

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The asymmetric unit of the title compound, $C_8H_{18}N^+\cdot I^-$, contains one crystallographically independent *N*-methyl-*N*-propylpyrrolidinium cation and one iodide anion, both located in general positions. No significant hydrogen bonding between the carbon-bound H atoms and the halide anion is observed.

Received 8 August 2005
Accepted 9 August 2005
Online 12 August 2005

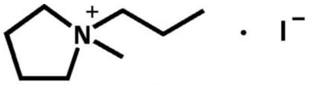
Key indicators

Single-crystal X-ray study
T = 170 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.027
wR factor = 0.073
Data-to-parameter ratio = 29.5

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

Comment

N-Methyl-*N*-alkylpyrrolidinium compounds have attracted increasing attention over the last few years with regard to low-melting-point ionic materials. It has been well established that a combination of *N*-methyl-*N*-alkylpyrrolidinium cations with weakly basic anions, such as $(CF_3SO_2)_2N^-$, BF_4^-, $CF_3SO_3^-$, PF_6^- and CH_3COO^-, leads to the formation of so-called ionic liquids (Golding *et al.*, 2001), compounds that have a melting point below 373 K (Wasserscheid, 2002). Pyrrolidinium-based compounds are of interest for applications in electrochemistry, as media for organic and inorganic reactions and spectroscopy (Seddon, 1997; MacFarlane *et al.*, 1999; Arenz *et al.*, 2005; Babai & Mudring, 2005).



(I)

We report here the crystal structure of *N*-methyl-*N*-propylpyrrolidinium iodide, (I), which serves as an reactant in

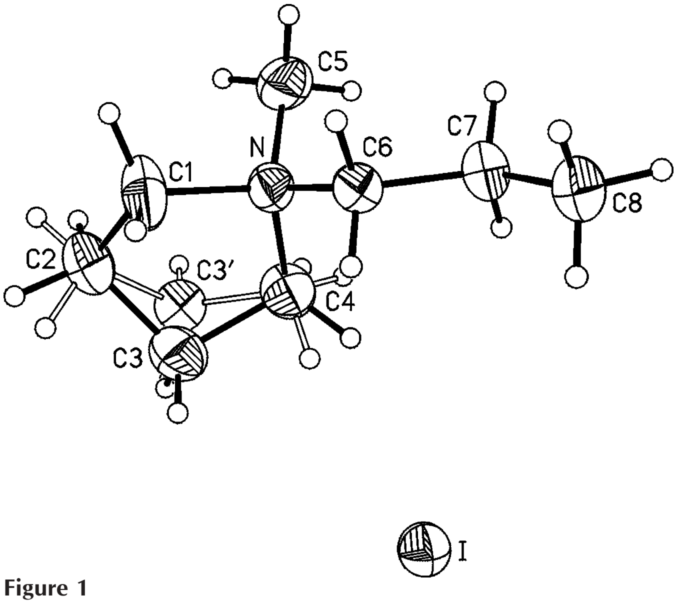


Figure 1
The asymmetric unit of the crystal structure of (I), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

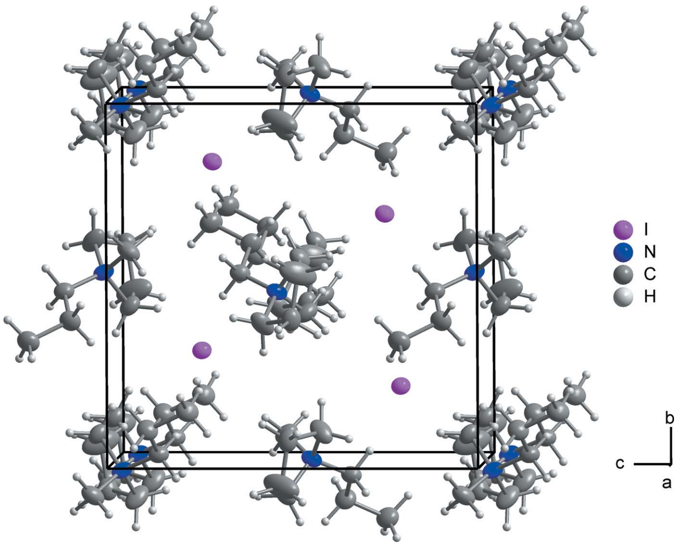


Figure 2
The unit cell contents of (I). Disorder of the pyrrolidinium ring has been omitted for clarity.

the synthesis of many ionic liquid compounds (Sun *et al.*, 2003; MacFarlane *et al.*, 1999; Golding *et al.*, 2001) but itself melts at about 415 K (Castriota *et al.*, 2005).

Although Raman studies have recently been performed on *N*-methyl-*N*-propylpyrrolidinium iodide (Castriota *et al.*, 2005), the crystal structure of this compound is so far unknown. According to the synthetic procedure described below, we were able to obtain crystals of *N*-methyl-*N*-propylpyrrolidinium iodide which had sufficient quality for single-crystal X-ray structure analysis. Fig. 1 shows the asymmetric unit of the title compound and Fig. 2 illustrates the three-dimensional structure, which is reminiscent of a (strongly) distorted zinc blende structure when considering the centers of gravity of the *N*-methyl-*N*-propylpyrrolidinium cation and the iodide anion. In the *N*-methyl-*N*-propylpyrrolidinium cation, C1—N—C4—C3 = −3.2 (4)°, C1—N—C3′—C4 = 33.7 (6)°, C1—C2—C3—C4 = −43.7 (4)° and C1—C2—C3′—C4 = 14.9 (9)°; mean C—C = 1.52 Å. The ring N atom carries both a methyl [N—C5 = 1.518 (5) Å] and a propyl group [N—C6 = 1.520 (5) Å]. The propyl chain shows an all-*trans* zigzag conformation, with a mean C—C interatomic distance of 1.55 Å. Notheworthy is the disorder of the pyrrolidinium ring (Fig. 1), which explains the observed broad Raman bands (Castriota *et al.*, 2005).

Experimental

The title compound, *N*-methyl-*N*-propylpyrrolidinium iodide, (I), was obtained after treatment of *N*-methyl-*N*-propylpyrrolidinium bis(trifluoromethanesulfonyl)amide (4 mmol, 1.62 g, 2.2 ml) with CaI_2 (1 mmol, 290 mg) at 393 K. Colorless single crystals form as an insoluble product after subsequent cooling (2 K min^{-1}) to room temperature. White transparent prismatic crystals of *N*-methyl-*N*-propylpyrrolidinium iodide were first inspected under an optical microscope. Crystals suitable for structure analysis were sealed in glass capillaries (0.3 mm diameter) and their quality checked by Laue photographs. A complete data set was taken of the best specimen. All operations were carried out under an inert argon atmosphere.

Crystal data

$C_8H_{18}N^+\cdot I^-$
$M_r = 255.13$
Orthorhombic, $P2_12_12_1$
$a = 7.6816$ (8) Å
$b = 11.8247$ (15) Å
$c = 12.0196$ (14) Å
$V = 1091.8$ (2) $Å^3$
$Z = 4$
$D_x = 1.552$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 20726 reflections
$\theta = 2.4–29.3°$
$\mu = 2.88$ mm^{-1}
$T = 170$ (2) K
Prism, white
0.30 × 0.20 × 0.10 mm

Data collection

Stoe IPDS-II diffractometer
φ scans
Absorption correction: numerical [*X-RED32* (Stoe & Cie, 2002) and *X-SHAPE* (Stoe & Cie, 2001)]
$T_{min} = 0.468$, $T_{max} = 0.697$
14164 measured reflections
2891 independent reflections
2796 reflections with $I > 2\sigma(I)$
$R_{int} = 0.075$
$\theta_{max} = 29.3°$
$h = -10 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.073$
$S = 1.09$
2891 reflections
98 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 0.7962P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.77$ e $Å^{-3}$
$\Delta\rho_{min} = -0.73$ e $Å^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0164 (12)
Absolute structure: Flack (1983), 1228 Friedel pairs
Flack parameter: −0.01 (4)

Table 1
Selected geometric parameters (Å, °).

N—C5	1.518 (5)	C2—C3’	1.557 (11)
N—C6	1.520 (5)	C3—C4	1.530 (6)
N—C1	1.539 (4)	C3’—C4	1.493 (11)
N—C4	1.545 (4)	C6—C7	1.529 (4)
C1—C2	1.500 (5)	C7—C8	1.540 (5)
C2—C3	1.547 (6)		
C5—N—C6	111.0 (3)	N—C1—H1*B*	110.7
C5—N—C1	108.5 (3)	H1*A*—C1—H1*B*	108.8
C6—N—C1	110.4 (3)	C1—C2—C3	100.1 (3)
C5—N—C4	110.6 (3)	C1—C2—C3’	107.9 (5)
C6—N—C4	110.4 (3)	C4—C3—C2	104.9 (4)
C1—N—C4	105.9 (3)	C4—C3’—C2	106.2 (8)
C2—C1—N	105.3 (3)	C3’—C4—N	103.7 (5)
C2—C1—H1*A*	110.7	C3—C4—N	103.9 (3)
N—C1—H1*A*	110.7	N—C6—C7	115.1 (3)
C2—C1—H1*B*	110.7	C6—C7—C8	109.3 (3)

All H atoms were positioned with idealized geometry (methylene C—H = 0.99 Å and methyl C—H = 0.98 Å) and refined with fixed isotropic displacement parameters [$U_{iso}(H) = 1.5U_{eq}$(methyl C) and $1.2U_{eq}$(methylene C)] using a riding model. The methyl groups were idealized, then refined as a rigid group allowed to rotate but not tip. Atom C3 and the H atoms attached to C2 and C4 are disordered over two positions and were refined using a split model and restraints for the C—C distances. The major occupied position (0.75) was refined anisotropically and the minor occupied position (0.25) isotropically.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

DIAMOND (Brandenburg, 1996); software used to prepare material for publication: *SHELXL97*.

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