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(*S*)-(–)-1-(4-Methylphenyl)ethylammonium chloride

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.116
Data-to-parameter ratio = 19.2

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

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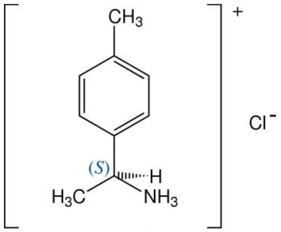
In the chiral title salt, $C_9H_{14}N^+\cdot Cl^-$, cations and anions are held together by N—H···Cl hydrogen bonds, forming a one-dimensional network in the crystal structure.

Comment

Reactions in dry media or under solvent-free conditions are becoming an area of growing interest; they are especially appealing as they provide rapid reaction rates, clean reaction conditions and ease of manipulation (Tanaka & Toda, 2000; Jeon *et al.*, 2005). As part of an ongoing program to develop benign and expeditious methods for organic transformations under solvent-free conditions, we have undertaken the preparation of chiral amides. The title salt, (I) (Fig. 1), was observed as a by-product in such reactions (see *Experimental*).



(I)

The (*p*-tolyl)ethylammonuim cation found in (I) has been crystallographically characterized with a number of anions (*e.g.* Bálint *et al.*, 2004); however no halogenide salts have been reported so far. In contrast, the crystal structure of (*R*)-1-phenylethylammonium bromide, (II), has been reported recently (Fischer, 2006); this is based on a layered network formed through N—H···Br hydrogen bonds. Interestingly, the crystal structure of (I) is different from that observed for (*R*)-1-phenylethylammonium bromide, even though both compounds crystallize in the same space group with similar cell parameters. In (I), ammonium H atoms are involved in N—H···Cl hydrogen bonds (Table 1), forming *R*(8) motifs. Edge-sharing motifs propagate along [100], forming a one-dimensional chain (Fig. 2). Differences in interionic contacts between (I) and (II) are reflected in a limited, but significant modification of the conformation of the cation: the dihedral angle between the benzene ring and $N_{ammonium}/C_{methine}/C_{methyl}$ plane (N1/C1/C2) is 66.02 (12)° in (I), *versus* 72.7° in (II).

Experimental

Under solvent-free conditions, (*S*)-(−)-1-(4-methylphenyl)ethylamine (0.378 g, 2.8 mmol) and pyridine-2,6-dicarbonyl dichloride (0.286 g, 1.4 mmol) were mixed at 298 K. The resulting crude product

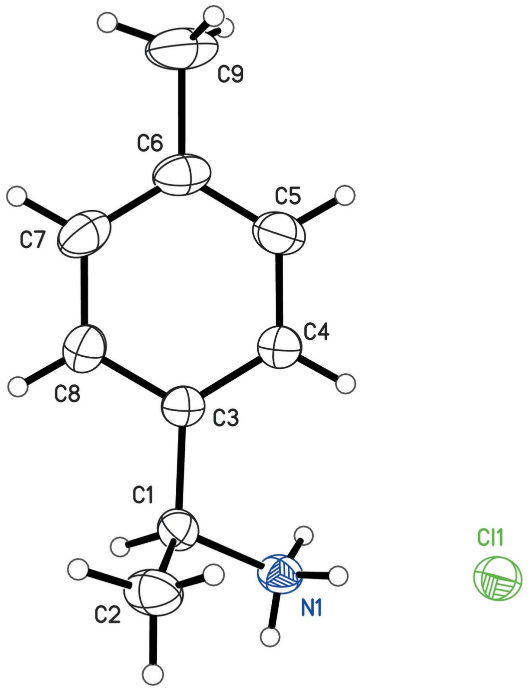


Figure 1
The structure of (I) with displacement ellipsoids drawn at the 30% probability level for non-H atoms.

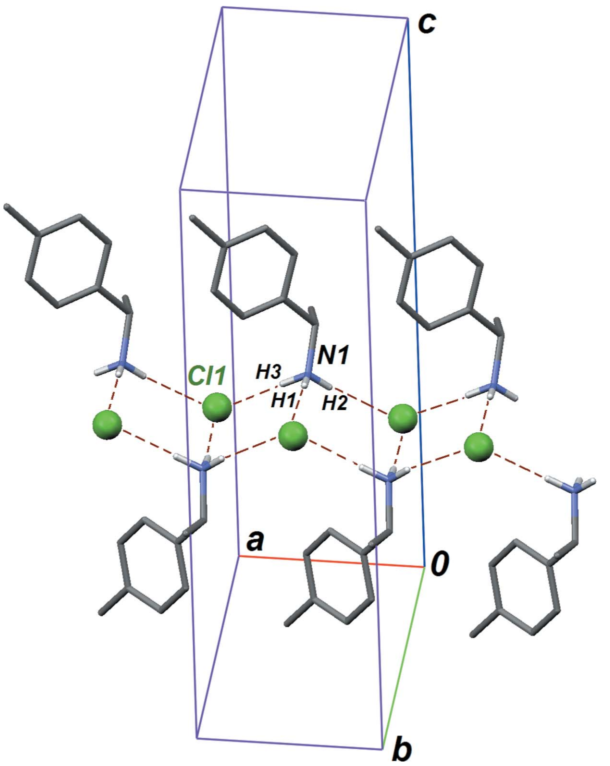


Figure 2
Part of the crystal structure of (I), showing hydrogen bonds as dashed lines. C-bound H atoms have been omitted for clarity.

was recrystallized from ethanol, affording the expected diamide (96% yield) and (I) as a by-product (less than 2% yield).

Crystal data

$C_9H_{14}N^+\cdot Cl^-$
$M_r = 171.66$
Orthorhombic, $P2_12_12_1$
$a = 5.4849$ (9) Å
$b = 9.1520$ (13) Å
$c = 19.867$ (4) Å
$V = 997.3$ (3) Å^3
$Z = 4$
$D_x = 1.143$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.33$ mm^{-1}
$T = 296$ (1) K
Prism, colourless
0.60 × 0.40 × 0.22 mm

Data collection

Bruker P4 diffractometer
$2\theta/\omega$ scans
Absorption correction: ψ scan (*XSCANS*; Siemens, 1996)
$T_{\min} = 0.767$, $T_{\max} = 0.929$
2315 measured reflections
2128 independent reflections
1779 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 30.0°$
3 standard reflections every 97 reflections intensity decay: 5.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.116$
$S = 1.03$
2128 reflections
111 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 0.076P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}
Absolute structure: Flack (1983), 431 Friedel pairs
Flack parameter: 0.01 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1$^{\rm i}$	0.90 (3)	2.29 (3)	3.1552 (19)	160 (2)
N1—H2···Cl1$^{\rm ii}$	0.82 (4)	2.38 (4)	3.157 (2)	159 (3)
N1—H3···Cl1	0.89 (4)	2.29 (4)	3.180 (2)	179 (3)

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

H atoms bonded to C atoms were placed in idealized positions and refined with a riding model approximation. Constrained C—H distances: 0.93 for aromatic CH, 0.96 for methyl CH_3 and 0.98 Å for methine CH. Isotropic displacement parameters: $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for aromatic and methine (CH) and $1.5U_{\rm eq}({\rm C})$ for methyl H atoms. Methyl groups were considered as rigid rotating groups. H atoms bound to N1 were found in a difference map and their coordinates refined freely, with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm N1})$.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL-Plus*; molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXTL-Plus*.

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