

Benzyl-diethylammonium chloride

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Key indicators

Single-crystal X-ray study
 $T = 150$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.035
 wR factor = 0.070
 Data-to-parameter ratio = 19.0

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

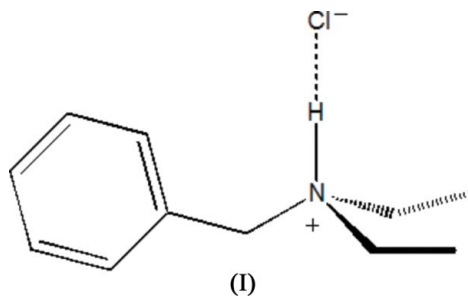
The title compound, $\text{C}_{11}\text{H}_{18}\text{N}^+\cdot\text{Cl}^-$, crystallizes in a non-centrosymmetric polar space group and possesses normal geometric parameters. The crystal structure analysis reveals a discrete ionic species, for which the packing is consolidated by an $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bond and $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds, resulting in a two-dimensional network.

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Comment

The reactivity of (phenylthiomethyl)amines has received considerable attention, not only with regard to their coordination chemistry but also for their use in synthetic organic chemistry (Screttas & Screttas, 1978; Yus, 1996; Gómez *et al.*, 1997; Foubelo *et al.*, 1997). The complexing reagent (lithiomethyl)amine can be obtained from the reduction of (phenylthiomethyl)amines with a solution of lithium naphthalenide ($\text{LiC}_{10}\text{H}_8$) (Gómez *et al.*, 1997). In an attempted reaction of the corresponding (lithiomethyl)amine, a hexane solution of this compound was treated at 195 K with a hexane solution of Ph_2BCl , resulting in the formation of benzyl- N,N' -diethylammonium chloride, (I) (Fig. 1), as a side-product.



The asymmetric unit in the non-centrosymmetric polar space group, $Pna2_1$, of (I) contains only one ion pair. All the geometric parameters for the cation lie within their expected ranges (Allen *et al.*, 1987). The tetrahedrally coordinated N atom has typical bond lengths and angles (Table 1).

As well as coulombic and van der Waals forces, the crystal packing in (I) appears to be controlled by several different intermolecular interactions (Table 2). The most clearcut is an $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bond that links adjacent ionic components, while a combination of $\text{N}-\text{H}\cdots\text{Cl}$ and $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds results in parallel chains of ions in the b -axis direction (Fig. 2). Within a chain, adjacent cations, related by a b -glide plane, are enantiomorphous. In addition, Fig. 2 shows that all the chains propagate in the same direction, *i.e.* all the $\text{N}-\text{H}$ bonds point the same way. The $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds lead to greater complexity than the rather simple motif

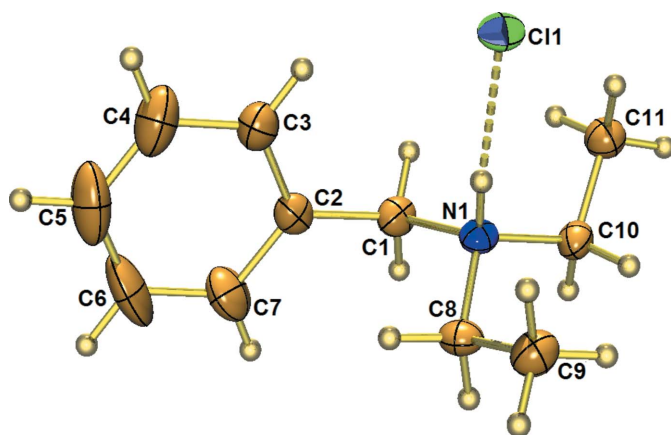


Figure 1
A view of (I), showing the atom-labelling scheme and the N–H...Cl hydrogen bond (dashed lines). Displacement ellipsoids are shown at the 50% probability level.

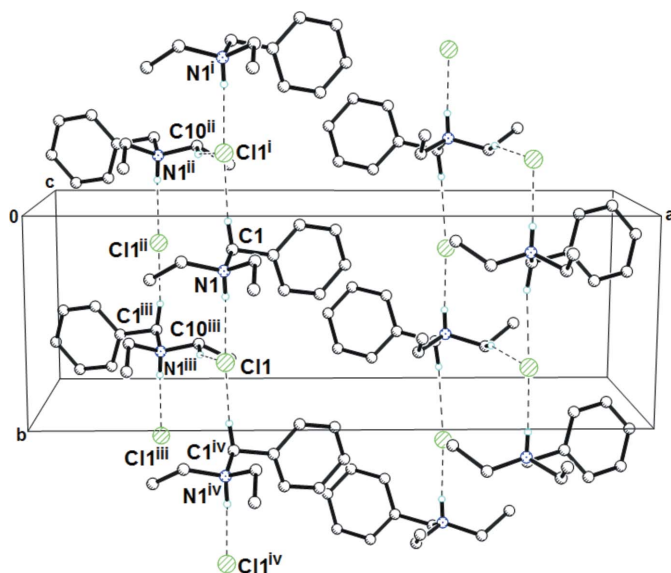


Figure 2
A view of part of the crystal structure of (I), showing the formation of hydrogen-bonded chains along the [010] direction. H atoms not involved in the interactions shown have been omitted. [Symmetry codes: (i) $x, y - 1, z$; (ii) $\frac{3}{2} - x, y - \frac{1}{2}, z - \frac{1}{2}$; (iii) $\frac{3}{2} - x, y - \frac{3}{2}, z - \frac{1}{2}$; (iv) $x, 1 + y, z$.]

generated by the N–H...Cl hydrogen bonds alone. The C–H...Cl hydrogen bond involving C10, in combination with the other hydrogen bonds, generates a sheet parallel to (201). The combination of the [010] chain and the (201) sheet make it sufficient to generate a continuous two-dimensional framework.

Experimental

For the preparation of compound (I), a freshly prepared solution of lithium naphthalenide from lithium granules (0.070 g, 9.98 mmol) and naphthalene (1.279 g, 9.98 mmol) in tetrahydrofuran (25 ml) at 195 K was added to (phenylthiomethyl)amine (1.946 g, 9.98 mmol) pre-cooled to the same temperature. After 30 min a solution of diphenylboron chloride (1.000 g, 4.99 mmol) in hexane (10 ml) was added dropwise over a period of 30 min. The mixture was warmed to

room temperature and stirred for 24 h. The mixture was then filtered, and the solvent and volatiles were removed under vacuum. The remaining naphthalene was removed by steam distillation under vacuum at 333 K. The main product of (I) was extracted with diethyl ether (two portions of 20 ml). A few days later at room temperature, some well formed colourless needle-shaped crystals of (I) were obtained. CHN analysis of a handpicked sample of (I) found: C 67.47, H 8.09, N 6.82%; calculated: C 66.15, H 9.08, N 7.01%.

Crystal data

$C_{11}H_{18}N^+Cl^-$	$Z = 4$
$M_r = 199.71$	$D_x = 1.156 \text{ Mg m}^{-3}$
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation
$a = 20.366 (3) \text{ \AA}$	$\mu = 0.29 \text{ mm}^{-1}$
$b = 7.0174 (9) \text{ \AA}$	$T = 150 (2) \text{ K}$
$c = 8.0292 (11) \text{ \AA}$	Needle, colourless
$V = 1147.5 (3) \text{ \AA}^3$	$0.18 \times 0.06 \times 0.03 \text{ mm}$

Data collection

Bruker SMART APEX CCD diffractometer with an Oxford Cryosystems low-temperature device	6285 measured reflections
φ and ω scans	2335 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1258 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.884, T_{\max} = 1.000$	$R_{\text{int}} = 0.032$
	$\theta_{\max} = 26.4^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.035$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.070$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 0.99$	$\Delta\rho_{\max} = 0.31 \text{ e \AA}^{-3}$
2335 reflections	$\Delta\rho_{\min} = -0.15 \text{ e \AA}^{-3}$
123 parameters	Absolute structure: Flack (1983),
H atoms treated by a mixture of independent and constrained refinement	1069 Friedel pairs
	Flack parameter: 0.09 (5)

Table 1
Selected geometric parameters ($\text{\AA}, ^\circ$).

C1–N1	1.512 (2)	C10–N1	1.4995 (18)
C8–N1	1.492 (2)	N1–H1	0.869 (11)
C2–C1–N1	114.45 (12)	C8–N1–C1	111.84 (11)
N1–C8–C9	113.64 (13)	C10–N1–C1	109.71 (12)
N1–C8–H82	108.8	C8–N1–H1	108.2 (12)
N1–C10–C11	113.92 (13)	C10–N1–H1	107.5 (9)
C8–N1–C10	111.96 (14)	C1–N1–H1	107.4 (14)
C9–C8–N1–C10	68.63 (16)	C11–C10–N1–C1	68.22 (15)
C9–C8–N1–C1	–167.77 (12)	C2–C1–N1–C8	53.45 (16)
C11–C10–N1–C8	–166.99 (12)	C2–C1–N1–C10	178.31 (12)

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–H1...C11	0.869 (11)	2.236 (12)	3.1036 (10)	175.8 (16)
C1–H12...CH ⁱ	0.97	2.78	3.6505 (16)	150
C10–H102...C11 ⁱⁱ	0.97	2.77	3.6815 (16)	157

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

The H atom attached to the N atom was located in a difference Fourier synthesis and its positional parameters were refined. Other H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C–H distances between 0.93 and 0.97 Å. The $U_{\text{iso}}(\text{H})$ values were set equal to 1.5 (methyl H atoms) or 1.2 (other H atoms) times $U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL97*.

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