

2-Chloro-2-phenylethylammonium chloride

Tore Lejon, Truls Ingebrigtsen
and Lars Kr. Hansen*Department of Chemistry, University of Tromsø,
9037 Tromsø, Norway

Correspondence e-mail: larsk@chem.uit.no

The title compound, $C_8H_{11}ClN^+ \cdot Cl^-$, has been obtained as an intermediate in the synthesis of 2-phenylaziridine. There are two molecules in the asymmetric unit and these are linked in the crystal structure by a network of intermolecular $N-H \cdots Cl$ and $C-H \cdots Cl$ hydrogen bonds, plus an intermolecular $Cl \cdots Cl$ interaction.

Received 13 January 2006

Accepted 17 January 2006

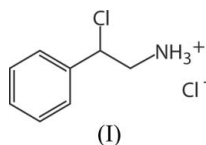
Key indicators

Single-crystal X-ray study
 $T = 298$ K
 Mean $\sigma(C-C) = 0.006$ Å
 R factor = 0.039
 wR factor = 0.098
 Data-to-parameter ratio = 16.5

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

Comment

A number of aziridines have been studied as substrates in ring-opening reactions under neutral conditions. In this respect, 2-phenylaziridine was of interest as it is an analogue of, and exhibits similar properties to, mexiletine (De Luca *et al.*, 2000, 2003). In this synthetic sequence, 2-chloro-2-phenylethylammonium chloride, (I), was produced as an intermediate.



The title compound crystallizes in the monoclinic centrosymmetric space group $P2_1/c$ with two molecules in the asymmetric unit (Fig. 1). The bond lengths are within the normal ranges (Allen *et al.*, 1987). There are short intermolecular hydrogen bonds ($N-H \cdots Cl$ and $C-H \cdots Cl$) influencing the conformation of the two molecules (Taylor & Kennard, 1982). Table 1 lists selected hydrogen bonds shorter than the van der Waals distance (Bondi, 1964). There is also a short intermolecular $Cl \cdots Cl$ contact [$Cl1 \cdots Cl4(1 - x, \frac{1}{2} + y, \frac{3}{2} - z)$] of 3.488 (1) Å with a $C12-Cl1 \cdots Cl4$ angle of 176.5 (1)°.

Experimental

The title compound was synthesized following the procedure of Galindo *et al.* (1997) and crystals suitable for X-ray analysis were obtained by dissolving the compound in ethanol, followed by addition of diethyl ether until the solution was cloudy.

Crystal data

$C_8H_{11}ClN^+ \cdot Cl^-$
 $M_r = 192.08$
 Monoclinic, $P2_1/c$
 $a = 8.4813$ (13) Å
 $b = 20.409$ (5) Å
 $c = 10.939$ (2) Å
 $\beta = 90.719$ (13)°
 $V = 1893.3$ (6) Å³
 $Z = 8$

$D_x = 1.348$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 12-17^\circ$
 $\mu = 0.62$ mm⁻¹
 $T = 298$ (2) K
 Block, colourless
 0.40 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4
diffractometer
 ω - 2θ scans
Absorption correction: ψ scan
(*ABSCALC* in *OSCAIL*;
McArdle & Daly, 1999; North *et al.*, 1968)
 $T_{\min} = 0.789$, $T_{\max} = 0.886$
3600 measured reflections
3311 independent reflections

1320 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = 0 \rightarrow 10$
 $k = 0 \rightarrow 24$
 $l = -12 \rightarrow 12$
2 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.098$
 $S = 0.87$
3311 reflections
201 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0401P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.27 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.24 \text{ e } \text{Å}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N11-H11A \cdots Cl4^i$	0.89	2.24	3.105 (3)	163
$N11-H11B \cdots Cl3^{ii}$	0.89	2.25	3.122 (3)	168
$N11-H11C \cdots Cl3$	0.89	2.46	3.251 (3)	148
$N21-H21A \cdots Cl3^i$	0.89	2.42	3.173 (3)	143
$N21-H21A \cdots Cl2^{iii}$	0.89	2.92	3.433 (3)	119
$N21-H21B \cdots Cl4^{iv}$	0.89	2.22	3.111 (3)	176
$N21-H21C \cdots Cl4$	0.89	2.37	3.180 (3)	151
$C21-H212 \cdots Cl1^i$	0.97	2.74	3.560 (3)	143
$C21-H212 \cdots Cl3^i$	0.97	3.13	3.485 (3)	104

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

All H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C–H distances at 0.93 Å, the CH₂ C–H distances at 0.97 Å and the CH C–H distances at 0.98 Å, with $U_{\text{iso}}(\text{H}) = 1.3U_{\text{eq}}(\text{C})$. The NH₃ N–H distances were fixed at 0.89 Å, with $U_{\text{iso}}(\text{H}) = 1.4U_{\text{eq}}(\text{N})$, and the NH₃ groups were allowed to rotate but not to tip. The quality of the crystal was rather poor and accordingly data were only collected to $\theta_{\text{max}} = 25.0^\circ$.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992); cell refinement: *CELDIM* in *CAD-4-PC Software*; data reduction: *XCAD4* (McArdle & Higgins, 1995); program(s) used to solve structure: *OSCAIL* (Version 9; McArdle, 2005) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX*

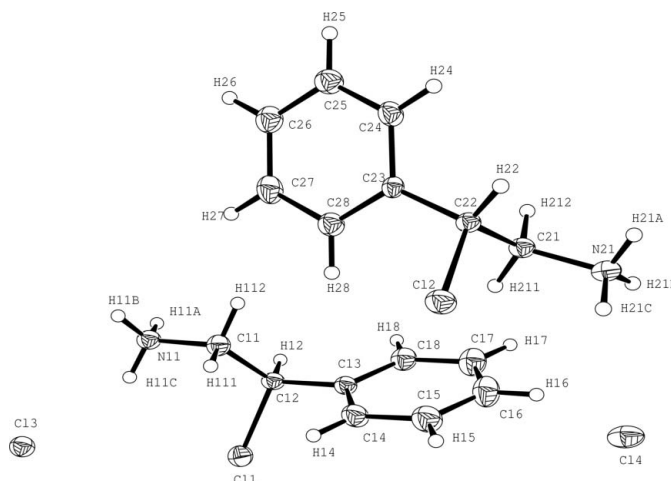


Figure 1

The asymmetric unit of the title compound (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 20% probability level.

(McArdle, 1993); software used to prepare material for publication: *OSCAIL* (McArdle, 2005).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bondi, A. (1964). *J. Chem. Phys.* **68**, 441–451.
De Luca, A., Natuzzi, F., Desaphy, J. F., Loni, G., Lentini, G., Franchini, C., Tortorella, V. & Conte-Camerino, D. (2000). *Mol. Pharmacol.* **57**, 268–277.
De Luca, A., Talon, S., De Bellis, S., Desaphy, J. F., Franchini, C., Lentini, G., Catalano, A., Corbo, F., Tortorella, V. & Conte-Camerino, D. (2003). *Naunyn-Schmiedeberg's Arch. Pharmacol.* **367**, 318–327.
Enraf–Nonius (1992). *CAD-4-PC Software*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.
Galindo, A., Orea, L. F., Gnecco, D., Enriquez, R. G., Toscano, R. A. & Reynolds, W. F. (1997). *Tetrahedron Asymmetry*, **8**, 2877–2879.
McArdle, P. (1993). *J. Appl. Cryst.* **26**, 752.
McArdle, P. (2005). *OSCAIL for Windows*. Version 1.0.7. Crystallography Centre, Chemistry Department, NUI, Galway, Ireland.
McArdle, P. & Daly, P. (1999). *ABSCAL*. PC version. National University of Ireland, Galway, Ireland.
McArdle, P. & Higgins, T. (1995). *XCAD*. National University of Ireland, Galway, Ireland.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.