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(R)-(−)-Mepivacaine hydrochloride enantiomer: a low-temperature study

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Low-temperature structural data are reported for the anaesthetic (2*R*)-(−)-2-(2,6-dimethylphenylaminocarbonyl)-1-methylpiperidinium chloride, $C_{15}H_{23}N_2O^+\cdot Cl^-$, which complement existing room-temperature data for the *S* enantiomer. Crystal packing is largely controlled by infinite N—H···Cl hydrogen-bonded chains.

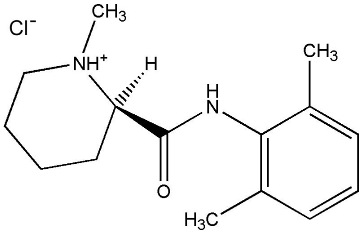
Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.003 Å
R factor = 0.022
wR factor = 0.047
Data-to-parameter ratio = 9.1

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

Comment

As part of a study of solid-state structural relationships between mepivacaine and its solvates (Giannellini *et al.*, 2005), the structure of the (*R*)-(−) enantiomer of mepivacaine hydrochloride, (I), has been refined using 180 K diffraction data. The room-temperature structure of the (*S*)-(+) enantiomer was already known (Csöregh, 1992). No phase transitions are recorded in the course of the 111 K temperature decrease from measurements of the previous study. There is a slightly anisotropic cell-volume reduction, 1.1% overall, the (moderately) largest linear contraction being in the (crystallographic *b*) direction of the infinite N—H···Cl···H—N chains. Results (Fig. 1, and Tables 1 and 2) are presented using the same labelling criteria of the previous study, for convenience. There is close agreement with the previous results, possibly with a smaller spread of values for chemically equivalent bond distances in the present case.



(I)

Experimental

Crystals of (*R*)-(−)-mepivacaine hydrochloride, suitable for X-ray work, were kindly supplied by Monica Jacobsson (Astra Zeneca R&D, Sweden) and were used as received.

Crystal data

$C_{15}H_{23}N_2O^+\cdot Cl^-$
$M_r = 282.80$
Orthorhombic, $P2_12_12_1$
$a = 9.7378$ (10) Å
$b = 10.5790$ (9) Å
$c = 15.2761$ (15) Å
$V = 1573.7$ (3) Å^3
$Z = 4$
$D_x = 1.194$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 952 reflections
$\theta = 6$–58°
$\mu = 2.10$ mm^{-1}
$T = 180$ (2) K
Elongated prism, colourless
0.80 × 0.30 × 0.30 mm

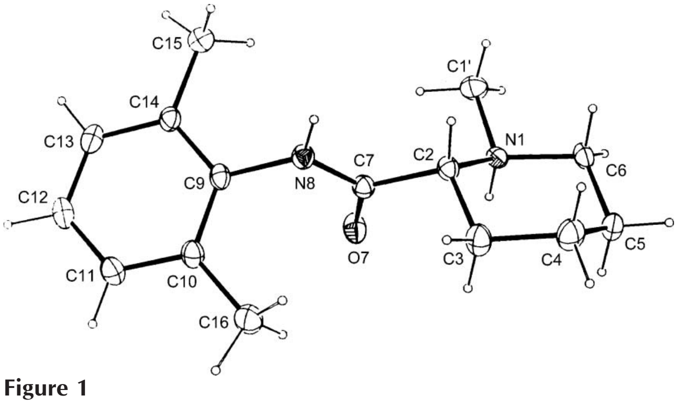


Figure 1
A view of the structure of the protonated molecule, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Data collection

Oxford Diffraction Xcalibur 3 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1986)
$T_{\min} = 0.331$, $T_{\max} = 0.533$
6079 measured reflections
2201 independent reflections
1989 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 59.0°$
$h = -10 \rightarrow 9$
$k = -11 \rightarrow 9$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.047$
$S = 0.96$
2201 reflections
242 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0218P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.11$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0167 (5)
Absolute structure: Flack (1983), 883 Friedel pairs
Flack parameter = 0.023 (11)

Table 1
Selected bond lengths (Å).

N1—C1′	1.494 (2)	N8—C9	1.437 (2)
N1—C2	1.495 (2)	C9—C14	1.396 (2)
N1—C6	1.508 (2)	C9—C10	1.399 (2)
C2—C7	1.526 (2)	C10—C11	1.389 (3)
C2—C3	1.528 (3)	C10—C16	1.505 (3)
C3—C4	1.523 (3)	C11—C12	1.379 (3)
C4—C5	1.512 (3)	C12—C13	1.380 (3)
C5—C6	1.499 (3)	C13—C14	1.391 (2)
C7—O7	1.231 (2)	C14—C15	1.507 (3)
C7—N8	1.335 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1N$\cdots$Cl$^{\rm i}$	0.905 (17)	2.182 (18)	3.0140 (16)	152.5 (16)
N8—H8N$\cdots$Cl	0.841 (18)	2.280 (19)	3.1172 (17)	174.1 (17)

Symmetry code: (i) $-x, y-\frac{1}{2}, \frac{3}{2}-z$.

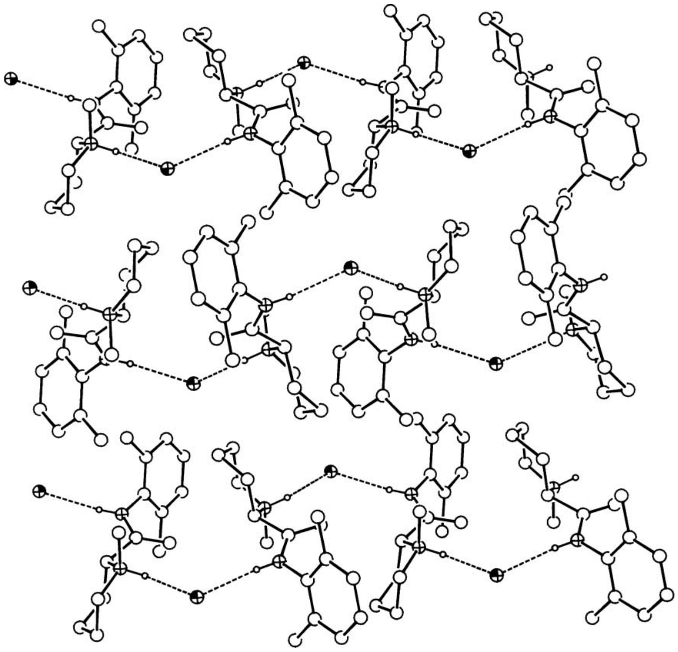


Figure 2
A view of the packing in the proximity of the *bc* face, showing the system of hydrogen bonds (dashed lines) in the structure. Only the H atoms directly involved in hydrogen bonding are shown.

Although the structure of the (S)-(+) enantiomer was known (Csöregh, 1992), that of the present (R)-(−) enantiomer was determined *ab initio* and the assignment of absolute configuration was checked (Flack, 1983). The material does not diffract strongly and it was deemed that collecting data at θ higher than 58° would not yield improvement. In the refinement, H-atom positions, initially assigned geometrically, were allowed to refine, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C,N})$ or $1.5U_{\rm eq}({\rm C_{methyl}})$. C—H bond distances: secondary $\rm CH_2$ = 0.94 (2)–1.01 (2) Å, methyl $\rm CH_3$ = 0.90 (2)–1.04 (2) Å, tertiary CH = 0.93 (3) Å, aromatic CH = 0.92 (2)–0.98 (2) Å and N—H = 0.84 (2)–0.90 (2) Å.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2001); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2001); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); 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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