Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

4-Methoxypiperidinium chloride

Zheng Yao,[a]‡ Xiao-Dan Gu,[a]§ Zhi-Gang Li,[b] Jian-Cheng Deng[a]* and Jing-Wei Xu[b]*

[a]The College of Chemistry, Xiangtan University, Hunan 411105, People's Republic of China, and [b]National Analytical Research Center of Electrochemistry and Spectroscopy, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, People's Republic of China

‡ Graduate School of Xiangtan University, Hunan 411105 People's Republic of China
§ Graduate School of Xiangtan University, Hunan 411105 People's Republic of China

Correspondence e-mail: djcwye@163.com, jwxu@ciac.jl.cn

Received 22 December 2006
Accepted 7 January 2007

The methoxypiperidinium cation of the title compound, $C_6H_{14}NO^+\cdot Cl^-$, adopts a chair conformation. The crystal structure is stabilized by N—H···Cl hydrogen bonds.

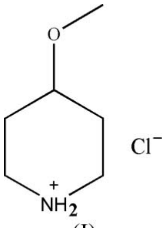
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.079
Data-to-parameter ratio = 18.6

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

Comment

The title compound, (I) (Fig. 1), is a common intermediate in drug synthesis. In (I), the methoxypiperidinium cation adopts a chair conformation. Each N atom donates two H atoms to two symmetry-equivalent halide ions, assembling the ions into chains running along the b axis (Table 1 and Fig. 2).



(I)

Experimental

To a stirred refluxing mixture of sodium hydride (60% in mineral oil; 2.5 g, 62.5 mmol) and dry tetrahydrofuran (50 ml) a solution of *tert*-butyl 4-hydroxypiperidine-1-carboxylate (10 g, 49.7 mmol) in dry

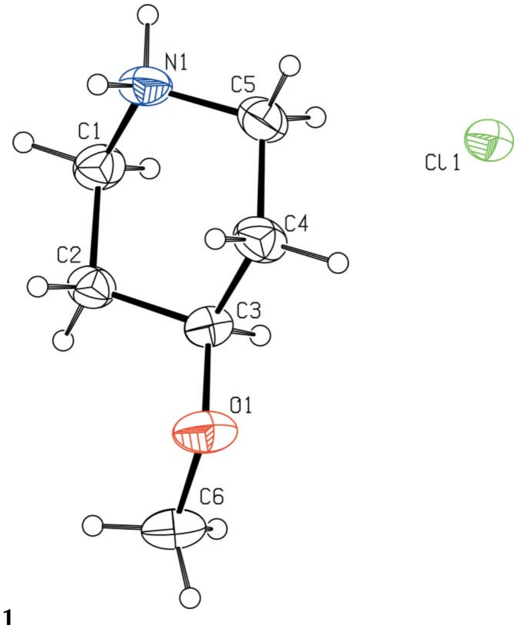


Figure 1
The molecular structure of (I), with the atom-labeling scheme and 30% probability displacement ellipsoids.

tetrahydrofuran (30 ml) was added dropwise. After the mixture had been refluxed for 1.5 h, iodomethane (5 ml) was added. The reaction mixture was refluxed for 2 h, and then cooled to room temperature; water (50 ml) and ethyl acetate (40 ml) were added. The layers were separated. The organic phase was washed with brine (20 ml), dried over sodium sulfate, and concentrated *in vacuo*. The crude liquid was flash chromatographed over silica gel using 1:1 hexane–ethyl acetate as eluent; all volatiles were removed *in vacuo*. A commercial 1.0 *M* methanol solution of hydrogen chloride (50 ml) was added dropwise and the resulting mixture was stirred for 6 h at room temperature. The solvents were removed under vacuum, giving 4-methoxypiperidinium chloride as a white solid (yield 94%). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a dichloromethane solution at room temperature.

Crystal data

$C_6H_{14}NO^+\cdot Cl^-$
$M_r = 151.63$
Monoclinic, $P2_1$
$a = 6.6778$ (13) Å
$b = 7.4041$ (14) Å
$c = 8.6654$ (16) Å
$\beta = 101.666$ (3)°
$V = 419.59$ (14) Å^3
$Z = 2$
$D_x = 1.200$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.39$ mm^{-1}
$T = 293$ (2) K
Plate, colorless
0.22 × 0.21 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.922$, $T_{\max} = 0.964$
2344 measured reflections
1564 independent reflections
1507 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.013$
$\theta_{\max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.079$
$S = 1.05$
1564 reflections
84 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2 + 0.0315P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.043 (6)
Absolute structure: Flack (1983), 673 Friedel pairs
Flack parameter: 0.00 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl1$^{\rm i}$	0.90	2.22	3.121 (2)	176
N1—H1B···Cl1$^{\rm ii}$	0.90	2.22	3.112 (2)	170

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

H atoms were positioned geometrically and refined with displacement parameters with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N), using a riding model, with C—H = 0.98 (CH), 0.97 (CH_2) and 0.96 Å (CH_3). H atoms bonded to nitrogen were refined as riding with an N—H distance of 0.90 Å.

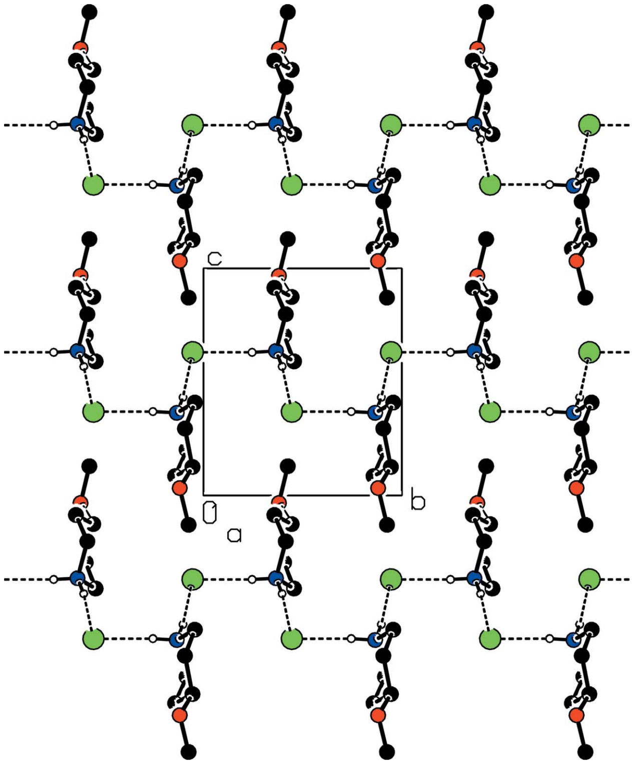


Figure 2
The hydrogen-bonding network in (I), with hydrogen bonds shown as dashed lines. For the sake of clarity, H atoms not involved in hydrogen bonding have been omitted.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

This work is supported by the Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun, People's Republic of China.

References

Bruker (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1998). *SMART*. Version 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2003). *SAINT*. Version 6. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Acta Cryst. (2007). E**63**, o654–o655 Yao *et al.* • $C_6H_{14}NO^+\cdot Cl^-$ **o655**