

Crystal data

C₂₀H₂₆O₃
M_r = 314.41
 Monoclinic
*P*2₁
a = 6.597 (1) Å
b = 11.468 (1) Å
c = 11.103 (1) Å
 β = 91.90 (1)°
V = 839.5 (2) Å³
Z = 2
D_x = 1.244 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 30 reflections
 θ = 3.57–14.40°
 μ = 0.082 mm⁻¹
T = 293 (2) K
 Rectangular plate
 0.58 × 0.46 × 0.10 mm
 Colorless

Data collection

Siemens *P4* diffractometer
 2θ/θ scans
 Absorption correction:
 numerical (Sheldrick;
 1997)
T_{min} = 0.960, *T_{max}* = 0.992
 3366 measured reflections
 2951 independent reflections
 2007 reflections with
I > 2σ(*I*)

R_{int} = 0.038
 θ_{\max} = 25°
 $h = -7 \rightarrow 7$
 $k = -13 \rightarrow 13$
 $l = -1 \rightarrow 13$
 3 standard reflections
 every 97 reflections
 intensity variation: <2%

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.050
wR(*F*²) = 0.099
S = 1.02
 2951 reflections
 215 parameters
 H atoms constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0301P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} < 0.001
 $\Delta\rho_{\max} = 0.12 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.13 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Scattering factors from
International Tables for
Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

O1—C3	1.239 (3)	O3—C20	1.331 (4)
O2—C20	1.202 (4)		
O2—C20—C17	125.4 (3)	O3—C20—C17	112.1 (3)
C16—C17—C20—O2	-14.2 (5)		

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O3—H3...O1 ⁱ	0.82	1.87	2.665 (3)	163
C2—H2A...O2 ⁱⁱ	0.93	2.62	3.495 (4)	157
C4—H4A...O2 ⁱⁱⁱ	0.93	2.63	3.476 (4)	151
C19—H19B...O2 ^{iv}	0.96	2.47	3.351 (4)	152

Symmetry codes: (i) 1 + *x*, *y*, 1 + *z*; (ii) 2 - *x*, *y* - ½, 2 - *z*; (iii) *x* - 1, *y*, *z* - 1; (iv) *x*, *y*, *z* - 1.

There were 1395 Friedel pairs in the refinement, but because there were no significant anomalous scatterers, the analysis was not able to confirm the known absolute configuration.

Data collection: XSCANS (Siemens, 1996). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS97 (Sheldrick, 1997*a*). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997*b*). Molecular graphics: SHELXL97. Software used to prepare material for publication: SHELXL97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1554). Services for accessing these data are described at the back of the journal.

References

- Berkovitch-Yellin, Z. & Leiserowitz, L. (1982). *J. Am. Chem. Soc.* **104**, 4052–4064.
 Borthwick, P. W. (1980). *Acta Cryst.* **B36**, 628–632.
 Brunskill, A. P. J., Lalancette, R. A. & Thompson, H. W. (1997). *Acta Cryst.* **C53**, 903–906.
 Brunskill, A. P. J., Thompson, H. W. & Lalancette, R. A. (1999). *Acta Cryst.* **C55**, 566–568.
 Coté, M. L., Lalancette, R. A. & Thompson, H. W. (1995). *Acta Cryst.* **C51**, 2305–2307.
 Djerassi, C. & Scholz, C. R. (1947). *J. Am. Chem. Soc.* **69**, 2404–2410.
 Fagart, J., Sobrio, F. & Marquet, A. (1997). *J. Labelled Compd Radiopharm.* **39**, 791–795.
 Jones, R. N. & Cole, A. R. H. (1952). *J. Am. Chem. Soc.* **74**, 5653–5661.
 Jones, R. N., Herling, F. & Katzenellenbogen, E. (1955). *J. Am. Chem. Soc.* **77**, 651–661.
 Jönsson, P.-G. (1972). *Acta Chem. Scand.* **26**, 1599–1619.
 Leiserowitz, L. (1976). *Acta Cryst.* **B32**, 775–802.
 Sallmann, F. & Tamm, C. (1956). *Helv. Chim. Acta.* **39**, 1340–1366.
 Sheldrick, G. M. (1997*a*). SHELXS97. Program for the Solution of Crystal Structures. University of Göttingen, Germany.
 Sheldrick, G. M. (1997*b*). SHELXL97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany.
 Siemens (1996). XSCANS User's Manual. Version 2.2. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Thompson, H. W., Vanderhoff, P. A. & Lalancette, R. A. (1991). *Acta Cryst.* **C47**, 1443–1445.

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2-Chloro-6-hydroxypyridinium chloride hydrate

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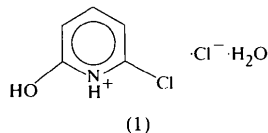
Abstract

The title compound, C₅H₅ClNO⁺·Cl⁻·H₂O, shows typical features of pyridinium cations (*i.e.* wider angles at nitrogen and narrower angles at C-2 and C-6 than in unprotonated pyridines). Short C—Cl and C—O bonds may indicate mesomeric stabilization of the positive charge at the N atom. The extended structure involves classical hydrogen bonds of forms N—H...Cl⁻, O_{hydroxy}—H...O_{water} and O_{water}—H...Cl⁻.

Short Cl \cdots Cl contacts of 3.3460 (8) Å are also observed. Secondary interactions link anions and cations to form a three-dimensional packing pattern.

Comment

We are interested in secondary interactions in pyridine derivatives (see Ahrens & Jones, 1999). We present here the structure of 2-chloro-6-hydroxypyridinium chloride, (1), which crystallized as a monohydrate (attributable to adventitious water in the acetone used as solvent).



In the cation, the N and O atoms are necessarily both protonated; the free base could in principle adopt either the hydroxypyridine or the pyridone tautomeric form, but in reality is the former (Kvick & Olovsson, 1968) (Fig. 1). We have recently discussed this tautomerism (Wijaya *et al.*, 1999) in some detail in connection with the structures of some 2-pyridones. The ring is planar (r.m.s. deviation 0.005 Å).

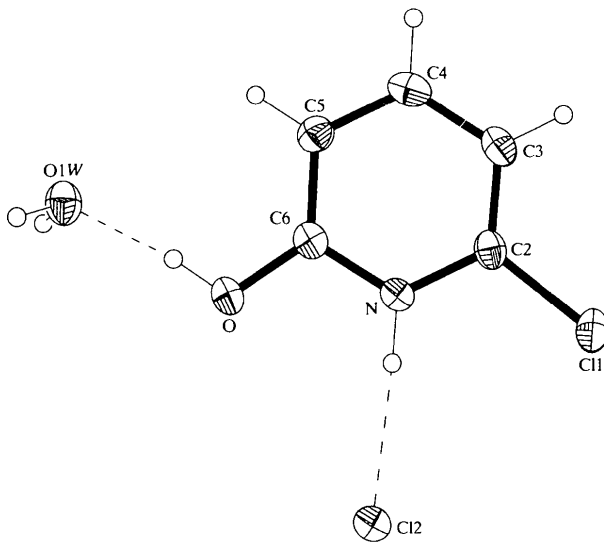


Fig. 1. The structure of the title compound with ellipsoids at the 50% probability level. H-atom radii are arbitrary. The two hydrogen bonds within the asymmetric unit (see text) are shown as dashed lines.

The ring bond angles in pyridine (Mootz & Wussow, 1975) are affected by protonation; in particular, the angle at nitrogen widens from 116.6° (average) to *e.g.* 122.5 (2)° in the dimesylamide salt (Wijaya, 1999) at the expense of the angles at C2/6, which narrow from 123.7 (average) to 120.0° (average). In (1), the angle at nitro-

gen is widened to 122.30 (16) from 117.1 (2)° in the free base; the ring angles at the chlorine- and oxygen-bearing carbon narrow from 125.3 (2) to 120.91 (17)° and from 122.1 (3) to 119.17 (16)°, respectively. The corresponding widening in (1) of the exocyclic angles C3—C2—Cl [123.51 (14)°] and C5—C6—O [126.45 (18)°] is consistent with the increased electronegativity of the protonated N atom.

The influence on bond lengths in pyridine on protonation is less marked. The major differences between the free base and (1) are for C2—Cl [1.740 (3) and 1.712 (2) Å, respectively], C6—O [1.321 (3) and 1.307 (2) Å] and C2—N [1.332 (3) and 1.356 (2) Å]. This is consistent with participation of the lone pairs on chlorine and oxygen in stabilization of the positive charge at nitrogen. However, the N—C6 bond is not significantly lengthened [1.341 (3) → 1.344 (2) Å].

The crystal packing is, as expected, determined by classical hydrogen bonds (Table 2; some possible borderline cases of C—H \cdots X hydrogen bonds are also presented). The Cl \cdots Cl ion acts as an acceptor for hydrogen bonds from the pyridinium NH group and both water

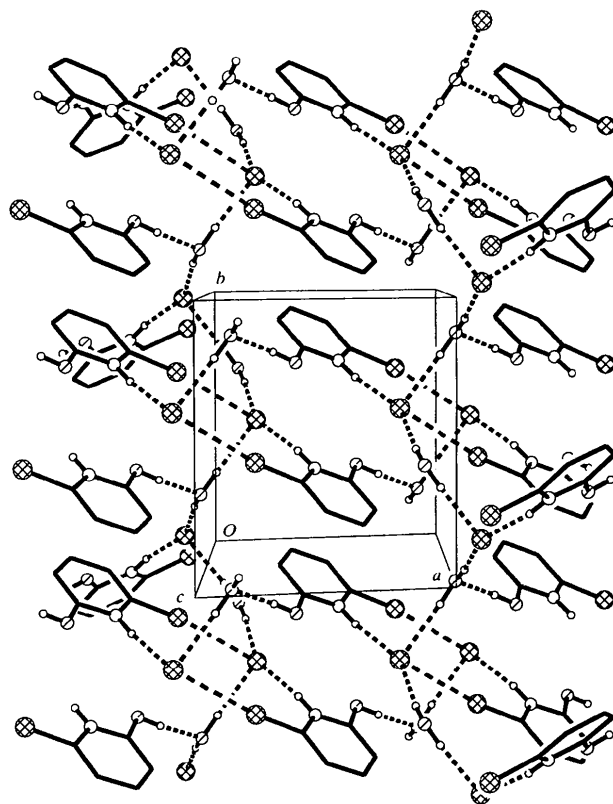


Fig. 2. Packing diagram of the title compound with the view direction approximately parallel to the z axis. H atoms not involved in hydrogen bonds have been omitted. Secondary interactions (hydrogen bonds and Cl \cdots Cl contacts) are indicated as dashed bonds. Water H atoms apparently not involved in hydrogen bonding in fact connect to other layers in the three-dimensional packing.

H atoms, and the water O atom acts as an acceptor from the hydroxyl group. Additionally, there are short C11···C12(2-x, 2-y, 1-z) contacts of 3.3460 (8) Å.

The net result of the secondary interactions is to create a three-dimensional packing pattern. One section of this is shown in Fig. 2. The most striking feature is the formation of ten-membered [C11···C12···H—N—C2]₂ rings.

The results presented here prompted us to undertake a systematic investigation of secondary interactions in pyridinium halides, the results of which will be published elsewhere.

Experimental

Hydrogen chloride gas was passed through a solution of 2-chloro-6-hydroxypyridine (1.00 g, 7.72 mmol) in dichloromethane (10 ml). The product formed as a white precipitate which was filtered off, washed with petroleum ether and dried *in vacuo* [yield 0.82 g (64%)]. Single crystals were obtained by diffusion of petroleum ether into a solution of the product in acetone.

Crystal data

C₅H₅ClNO⁺·Cl⁻·H₂O

M_r = 184.02

Monoclinic

*P*2₁/*c*

a = 7.3471 (10) Å

b = 7.9330 (12) Å

c = 13.6630 (16) Å

β = 104.662 (8)°

V = 770.41 (18) Å³

Z = 4

D_x = 1.587 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 63 reflections

θ = 4.7–12.3°

μ = 0.780 mm⁻¹

T = 173 (2) K

Plate

0.40 × 0.20 × 0.05 mm

Colourless

Data collection

Siemens *P4* diffractometer

ω scans

Absorption correction:

ψ scan (*XEMP*; Siemens, 1994a)

T_{min} = 0.861, *T_{max}* = 0.965

3022 measured reflections

1770 independent reflections

1302 reflections with

I > 2σ(*I*)

R_{int} = 0.023

θ_{max} = 27.49°

h = -9 → 5

k = 0 → 10

l = -17 → 17

3 standard reflections

every 247 reflections

intensity decay: none

Refinement

Refinement on *F*²

R[*F*² > 2σ(*F*²)] = 0.028

wR(*F*²) = 0.065

S = 0.885

1770 reflections

107 parameters

H atoms treated by a

mixture of independent and constrained refinement

w = 1/[σ²(*F_o*²) + (0.0342*P*)²]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.31 e Å⁻³

Δρ_{min} = -0.21 e Å⁻³

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

C11—C2	1.712 (2)	C2—C3	1.362 (3)
O—C6	1.307 (2)	C3—C4	1.398 (3)
N—C6	1.344 (2)	C4—C5	1.377 (3)
N—C2	1.356 (2)	C5—C6	1.396 (3)
C6—N—C2	122.30 (16)	C5—C4—C3	121.59 (18)
N—C2—C3	120.91 (17)	C4—C5—C6	118.42 (18)
N—C2—C11	115.57 (14)	O—C6—N	114.36 (16)
C3—C2—C11	123.51 (14)	O—C6—C5	126.45 (18)
C2—C3—C4	117.59 (17)	N—C6—C5	119.17 (16)

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N—H1···Cl2	0.90 (2)	2.18 (2)	3.0698 (16)	170.3 (18)
O—H6···O1W	0.86 (3)	1.65 (3)	2.504 (2)	172 (3)
O1W—H01···Cl2 ⁱ	0.90 (3)	2.19 (3)	3.0870 (17)	178 (3)
O1W—H02···Cl2 ⁱⁱ	0.84 (3)	2.26 (3)	3.0969 (16)	173 (3)
C3—H3···Cl2 ⁱⁱⁱ	0.95	2.93	3.799 (2)	152.3
C3—H3···O ⁱⁱⁱⁱ	0.95	2.67	3.278 (2)	122.3
C4—H4···Cl1 ^v	0.95	2.93	3.782 (2)	149.5
C5—H5···O1W ^v	0.95	2.67	3.414 (2)	135.2

Symmetry codes: (i) *x* - 1, *y*, *z*; (ii) 1 - *x*, *y* - ½, ½, ½ - *z*; (iii) *x*, ½ - *y*, *z* - ½; (iv) 1 - *x*, *y* - ½, ½ - *z*; (v) -*x*, 1 - *y*, 1 - *z*.

H atoms bonded to nitrogen or oxygen were located from Fourier syntheses and refined freely. Other H atoms were placed in calculated positions and refined using a riding model. A libration correction for the cation (*R_{lib}* = 0.062; Schomaker & Trueblood, 1968) led to the corrected bond lengths C2—C11 = 1.715, N—C2 = 1.360, C2—C3 = 1.366, C3—C4 = 1.401, C4—C5 = 1.381, C5—C6 = 1.400, C6—N = 1.346 and C6—O = 1.311 Å.

Data collection: *XSCANS* (Siemens, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *XP* (Siemens, 1994b). Software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1350). Services for accessing these data are described at the back of the journal.

References

- Ahrens, B. & Jones, P. G. (1999). *Acta Cryst.* **C55**, 1308–1310.
- Kvick, Å. & Olovsson, I. (1968). *Ark. Kemi.* **30**, 71–80.
- Mootz, D. & Wussow, H.-G. (1975). *J. Chem. Phys.* pp. 1517–1522.
- Schomaker, V. & Trueblood, K. N. (1968). *Acta Cryst.* **B24**, 63–76.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Siemens (1991). *XSCANS User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1994a). *XEMP. Empirical Absorption Correction Program*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1994b). *XP. Molecular Graphics Program*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Wijaya, K. (1999). PhD thesis, Technical University of Braunschweig, Germany.
 Wijaya, K., Moers, O., Blaschette, A. & Jones, P. G. (1999). *Z. Naturforsch. Teil B*, **54**, 643–648.

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1,10-Decanediol

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Abstract

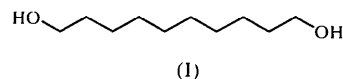
An all-*trans* conformation is observed in the hydrocarbon skeleton of the title compound, $C_{10}H_{22}O_2$, and both terminal hydroxyl groups are also *trans*. The centrosymmetric molecules are arranged in a zigzag manner to make a herring-bone motif, which can be regarded as a model structure of smectic C liquid crystals. These features are very similar to those of the homologues with an even number of C atoms, but different from those with an odd number.

Comment

Crystal structures of many different kinds of monosubstituted long-chain compounds, for example *n*-higher alcohols, have been studied by several workers (*e.g.* Watanabe, 1961; Seto, 1962). However, there are few reports about α,ω -disubstituted *n*-alkanes.

These compounds have a simple linear hydrocarbon chain as a skeleton. The molecular shape could be regarded as a rod-like one, which is one of the typical features of liquid crystalline molecules. Such rod-like molecules in general construct a layer structure similar to the smectic phase found in liquid crystals. One of the present authors reported phase-transition phenomena of long chain α,ω -alkanediols from C_{13} to C_{24} with another researcher (Ogawa & Nakamura, 1999). In addition to this, we have determined crystal structures of five α,ω -alkanediols, 1,11-undecanediol (Nakamura *et al.*, 1999), 1,12-dodecanediol (Nakamura & Setodoi, 1997), 1,13-tridecanediol (Nakamura *et al.*, 1997), 1,14-tetradecanediol (Nakamura & Sato, 1999) and 1,16-hexadecanediol (Nakamura & Yamamoto, 1994). Although the crystal structure of 1,10-decanediol, (I), has already been reported (Wang *et al.*, 1994), it was derived from two-dimensional data; we have obtained a more

extensive single-crystal data set. The results are in good agreement.



The molecular structure of 1,10-decanediol, (I), is shown in Fig. 1. The hydrocarbon skeleton has an all-*trans* conformation and both terminal hydroxyl groups are also *trans* with respect to the skeleton. The centrosymmetric molecules form a layer structure perpendicular to the longest axis and make a herring-bone structure. The inclination angle of the long axis of the molecule to the layer plane is equal in each layer, but the direction of the long axis is opposite in alternate layers as is shown in Fig. 2. This structure could be regarded as a model structure of smectic C liquid crystals.

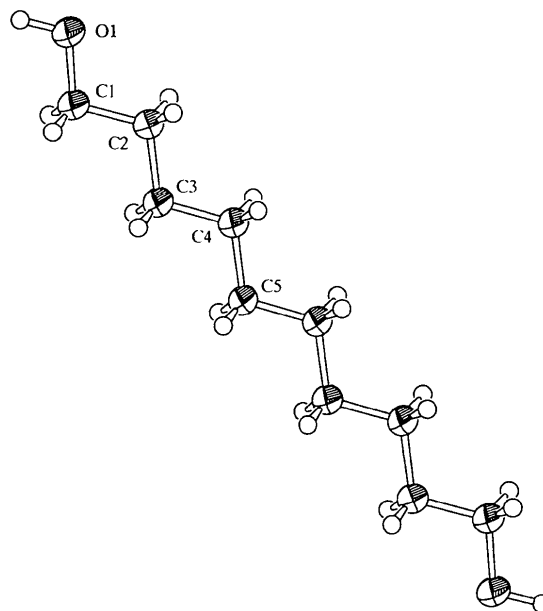


Fig. 1. The title molecule showing the crystallographic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

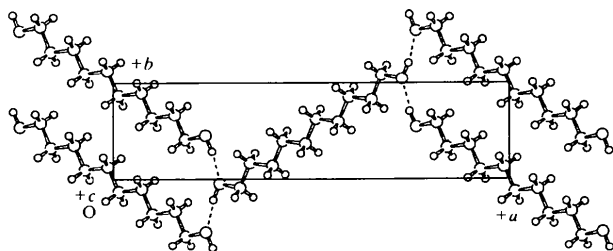


Fig. 2. The projection of the crystal structure along the *c* axis. Dashed lines indicate the hydrogen bonds.