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N-Methyl-2-pyrrolidone hydrochloride

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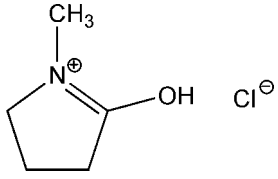
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.004 Å; R factor = 0.030; wR factor = 0.072; data-to-parameter ratio = 10.5.

The cation of the title compound, $C_5H_{10}NO^+ Cl^-$, has the five-membered ring in a very shallow envelope conformation. An interionic O—H···Cl hydrogen bond links the Cl^- anion to the cation.

Related literature

For data on the neutral compound, see: Müller *et al.* (1996). For an alternative method of preparation, see: Utsumi (1981). For related literature, see: Holleman (2007).



Experimental

Crystal data

$C_5H_{10}NO^+ \cdot Cl^-$
M_r = 135.59
Orthorhombic, $P2_12_12_1$
a = 6.1391 (2) Å
b = 8.9537 (2) Å
c = 12.5976 (4) Å
V = 692.46 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.46 mm^{-1}
T = 200 (2) K
0.28 × 0.25 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
8273 measured reflections
1192 independent reflections
1124 reflections with $I > 2\sigma(I)$
R_{int} = 0.094

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.072
S = 1.04
1192 reflections
113 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 471 Friedel pairs
Flack parameter: 0.04 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
O—H···Cl	1.03 (4)	1.80 (4)	2.8330 (14)	176 (3)

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: Windows version (Farrugia, 1997) of *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2495).

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supplementary materials

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N-Methyl-2-pyrrolidone hydrochloride

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Comment

The five-membered ring of *N*-Methyl-2-pyrrolidonium cation adopts a slightly pronounced envelope conformation in the solid state (Fig. 1), with C3 as the flap atom (C1—C2—C3—C4 = 13.4 (3) °). The O—H unit adopts an eclipsed conformation and is nearly coplanar to the ring-plane (C4—N—C1—C2 = −2.2 (2) °). The N—C1 distance [1.296 (3) Å] is substantially shorter than the sum of the covalent radii ($d_{cov.}$: C—N 1.47, C=N 1.27 Å, Holleman, 2007), which indicates partial double-bond character for this bond. A similar situation is found for the O—C1 bond [1.297 (3) Å], which also lies in the range between a single and a double bond ($d_{cov.}$: C—O 1.43, C=O 1.23 Å), indicating a certain degree of charge-delocalization along the N—C1—O unit.

In the crystal structure, the $C_5H_9NO^+$ cation and the Cl^- anion are linked by an O—H···Cl hydrogen-bond with a relatively short donor-acceptor distance. Interestingly, no further hydrogen-bonds were found (Fig. 2).

Experimental

N-Methyl-2-pyrrolidone was obtained from Merck Co. and was used as recieved. $HCl_{conc.}$ (24.64 g, 250 mmol) was added dropwise to a stirred solution of *N*-Methyl-2-pyrrolidone (4.957 g, 50 mmol) in CH_2Cl_2 (40 mL) at 0°C. The clear aqueous layer was separated and was then evaporated to dryness in vacuo. The colourless, crystalline residue was sublimed (10^{-3} mbar, 60°C) for purification. Yield: 5.597 g (41.28 mmol, 83%). Mp. = 94 °C. CHN-analysis: found, C 43.20, N 10.01, H 6.44%; calc. C 44.29, N 10.33, H 7.43%. ^{1}H-NMR (DMSO-d_6, 25 °C, 250 MHz): δ = 1.86 (quint, 2H, $^2J_{H—H}$ = 7.6 Hz), 2.16 (t, 2H, $^2J_{H—H}$ = 7.6 Hz), 2.66 (s, 3H), 3.27 (t, 2H, $^2J_{H—H}$ = 7.6 Hz), 11.5 (s, 1H). $^{13}C\{^1H\}$-NMR (DMSO-d_6, 25 °C, 250 MHz): δ = 17.5, 29.4, 30.4, 48.9, 174.3. Crystallization from a saturated CH_2Cl_2 solution at ambient temperature gave colourless crystals.

Refinement

All hydrogen atoms were found in difference syntheses, and refined freely.

Figures

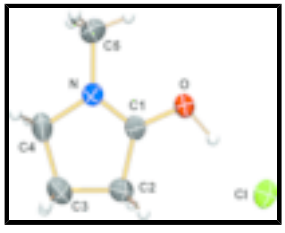

Fig. 1. Molecular structure of (I), showing the atomic numbering scheme and displacement ellipsoids drawn at the 50% probability level.

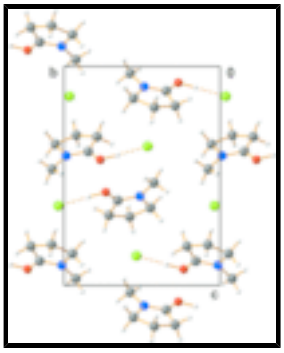

Fig. 2. Packing diagram for (I), viewed down the *a* axis. Hydrogen-bonds are indicated by dashed lines.

N-Methyl-2-pyrrolidone hydrochloride

Crystal data

$C_5H_{10}NO^+ \cdot Cl^-$	$F_{000} = 288$
$M_r = 135.59$	$D_x = 1.301$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 4313 reflections
$a = 6.1391$ (2) Å	$\theta = 3.1$–$24.7°$
$b = 8.9537$ (2) Å	$\mu = 0.46$ mm^{-1}
$c = 12.5976$ (4) Å	$T = 200$ (2) K
$V = 692.46$ (4) Å^3	Block, colourless
$Z = 4$	0.28 × 0.25 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer	1192 independent reflections
Radiation source: rotating anode	1124 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.094$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 24.7°$
$T = 200(2)$ K	$\theta_{min} = 3.2°$
phi/ω–scan	$h = -7 \rightarrow 7$
Absorption correction: none	$k = -10 \rightarrow 10$
8273 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 0.1465P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.072$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.15$ e Å^{-3}

1192 reflections	$\Delta\rho_{min} = -0.20$ e Å^{-3}
113 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 471 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.04 (9)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl	0.98418 (9)	1.03603 (5)	0.63590 (4)	0.04228 (18)
O	1.0110 (3)	0.73233 (15)	0.57419 (11)	0.0400 (3)
N	0.8324 (3)	0.51346 (18)	0.59226 (13)	0.0323 (4)
C1	0.8497 (3)	0.6551 (2)	0.61169 (14)	0.0305 (4)
C2	0.6698 (4)	0.7117 (2)	0.67914 (17)	0.0363 (5)
C3	0.5168 (5)	0.5788 (3)	0.6863 (2)	0.0553 (6)
C4	0.6445 (4)	0.4457 (3)	0.64539 (19)	0.0413 (5)
C5	0.9858 (5)	0.4241 (3)	0.5312 (2)	0.0445 (5)
H41	0.568 (4)	0.387 (3)	0.5962 (18)	0.043 (7)*
H21	0.612 (4)	0.803 (3)	0.647 (2)	0.054 (7)*
H31	0.479 (5)	0.559 (3)	0.752 (3)	0.080 (9)*
H22	0.724 (4)	0.743 (3)	0.745 (2)	0.056 (7)*
H51	1.018 (6)	0.342 (4)	0.571 (3)	0.092 (11)*
H42	0.701 (4)	0.380 (3)	0.702 (2)	0.051 (7)*
H52	0.914 (6)	0.383 (4)	0.472 (3)	0.092 (11)*
H32	0.389 (6)	0.591 (4)	0.641 (3)	0.098 (12)*
H53	1.106 (5)	0.480 (3)	0.511 (2)	0.059 (8)*
H	0.994 (6)	0.843 (4)	0.596 (3)	0.094 (10)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0466 (3)	0.0332 (3)	0.0470 (3)	−0.0062 (2)	−0.0023 (3)	−0.0018 (2)
O	0.0382 (8)	0.0316 (7)	0.0502 (8)	−0.0046 (8)	0.0125 (7)	0.0001 (6)
N	0.0339 (9)	0.0299 (9)	0.0331 (8)	−0.0008 (7)	0.0029 (7)	−0.0009 (7)
C1	0.0298 (10)	0.0314 (10)	0.0302 (10)	−0.0018 (8)	−0.0012 (8)	0.0026 (7)
C2	0.0348 (11)	0.0390 (12)	0.0351 (11)	0.0037 (9)	0.0035 (9)	−0.0005 (9)

C3	0.0445 (15)	0.0556 (14)	0.0657 (16)	−0.0099 (14)	0.0194 (14)	−0.0082 (12)
C4	0.0417 (11)	0.0408 (13)	0.0414 (11)	−0.0127 (10)	0.0023 (11)	0.0017 (11)
C5	0.0469 (13)	0.0338 (11)	0.0526 (13)	0.0048 (12)	0.0086 (13)	−0.0041 (10)

Geometric parameters (Å, °)

Cl—H	1.80 (4)	C2—H22	0.93 (3)
O—C1	1.297 (3)	C3—C4	1.516 (3)
O—H	1.03 (4)	C3—H31	0.87 (3)
N—C1	1.296 (3)	C3—H32	0.98 (4)
N—C5	1.455 (3)	C4—H41	0.94 (2)
N—C4	1.465 (3)	C4—H42	0.99 (2)
C1—C2	1.483 (3)	C5—H51	0.91 (3)
C2—C3	1.519 (3)	C5—H52	0.94 (4)
C2—H21	0.98 (3)	C5—H53	0.93 (3)
C1—O—H	110 (2)	C4—C3—H32	108 (2)
C1—N—C5	125.78 (19)	C2—C3—H32	112 (2)
C1—N—C4	112.53 (16)	H31—C3—H32	111 (3)
C5—N—C4	121.57 (18)	N—C4—C3	103.71 (17)
N—C1—O	121.02 (18)	N—C4—H41	108.8 (14)
N—C1—C2	112.45 (17)	C3—C4—H41	114.0 (15)
O—C1—C2	126.52 (18)	N—C4—H42	107.5 (15)
C1—C2—C3	103.08 (18)	C3—C4—H42	113.9 (14)
C1—C2—H21	108.9 (15)	H41—C4—H42	108.6 (19)
C3—C2—H21	117.0 (15)	N—C5—H51	107 (2)
C1—C2—H22	110.2 (16)	N—C5—H52	109 (2)
C3—C2—H22	113.6 (16)	H51—C5—H52	103 (3)
H21—C2—H22	104 (2)	N—C5—H53	111.6 (18)
C4—C3—C2	106.0 (2)	H51—C5—H53	114 (3)
C4—C3—H31	107 (2)	H52—C5—H53	111 (3)
C2—C3—H31	112 (2)		
C5—N—C1—O	1.6 (3)	O—C1—C2—C3	172.9 (2)
C4—N—C1—O	177.57 (19)	C1—C2—C3—C4	13.4 (3)
C5—N—C1—C2	−178.2 (2)	C1—N—C4—C3	10.8 (3)
C4—N—C1—C2	−2.2 (2)	C5—N—C4—C3	−173.1 (2)
N—C1—C2—C3	−7.4 (3)	C2—C3—C4—N	−14.6 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O—H···Cl	1.03 (4)	1.80 (4)	2.8330 (14)	176 (3)

Fig. 1

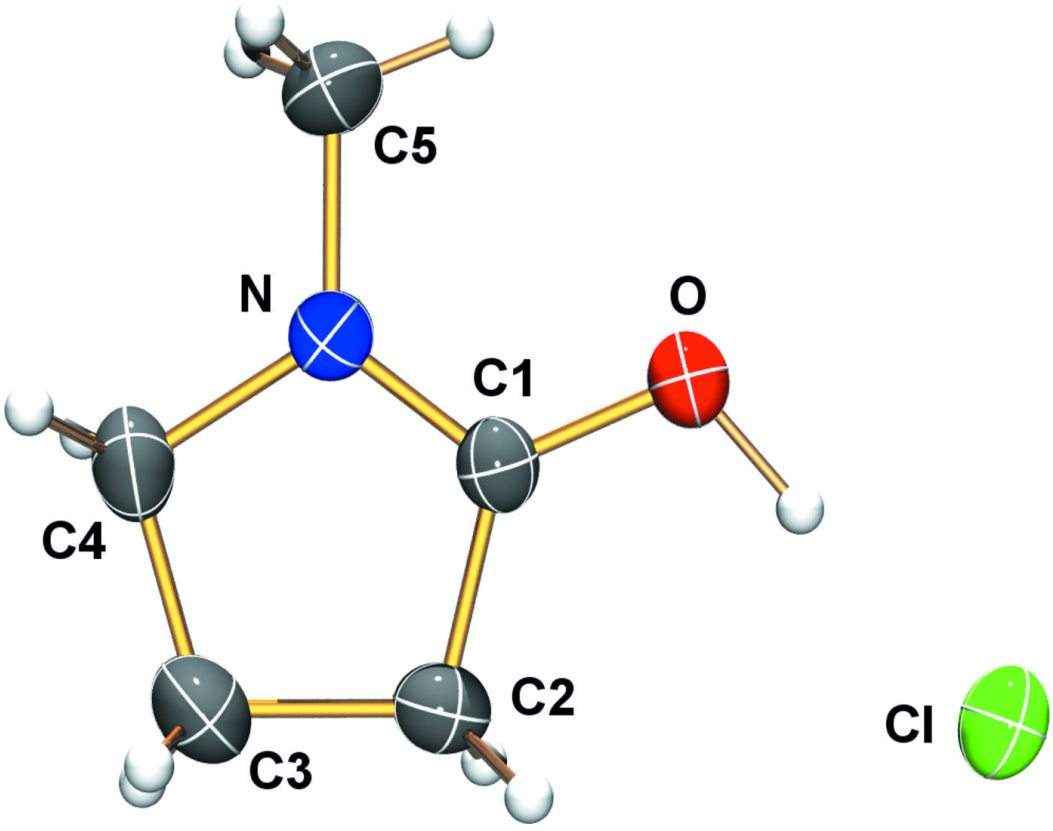

Fig. 2