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## Key indicators

Single-crystal X-ray study

T = 173 K

Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$ 

Disorder in solvent or counterion

R factor = 0.033

wR factor = 0.081

Data-to-parameter ratio = 19.0

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

## 4-Ammonio-1-hydroxy-2,2,6,6-tetramethylpiperidin-1-ium dichloride monohydrate

The crystal structure of the title compound,  $\text{C}_9\text{H}_{22}\text{N}_2\text{O}^{2+} \cdot 2\text{Cl}^- \cdot \text{H}_2\text{O}$ , has been determined at 173 (1) K. Attached to the piperidinyll ring, which adopts a chair conformation, the hydroxyl and the exocyclic ammonium groups are mutually *trans*, both occupying equatorial positions. The water molecule and the two chloride anions are involved in strong hydrogen bonding *via*  $\text{O} \cdots \text{H}$  and  $\text{H} \cdots \text{Cl}$  interactions.

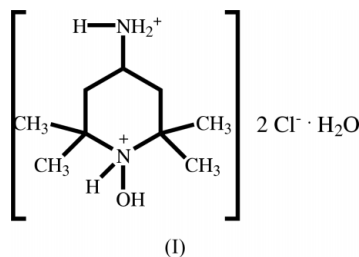
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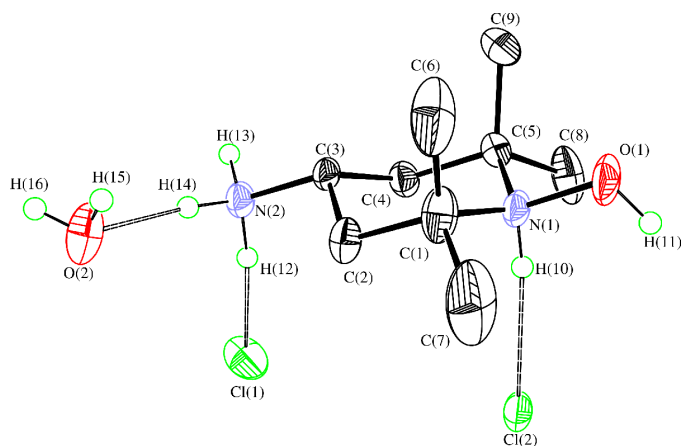
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## Comment

The title compound, (I), was first synthesized in an unhydrated form and studied for its protective effect, in Chinese hamster V79 cells, against oxidative damage caused by exposure to  $\text{H}_2\text{O}_2$  or ionizing radiation (Krishna *et al.*, 1998). Recently, (I) has been found to be a fibre-reactive radical scavenger that binds to lignin-rich wood pulps in aqueous media and inhibits the light-induced oxidation and yellowing of the pulps and papers made from such pulps (Hu, 2003). The molecular structure of this compound is shown in Fig. 1, while selected bond lengths, torsion angles and bond angles are listed in Table 1.



Two different N–H bond lengths and N–C bond lengths are found in the molecule, *viz.*  $\text{N1}-\text{H10} = 0.88 (2) \text{ \AA}$ ,  $\text{N2}-\text{H12}$  or  $-\text{H13}$  or  $-\text{H14} = 0.98-1.01 (2) \text{ \AA}$ ; and  $\text{N1}-\text{C1}$  or  $-\text{C5} = 1.531-1.534 (2) \text{ \AA}$  and  $\text{N2}-\text{C3} = 1.495 (2) \text{ \AA}$ . All C–C–C bond angles fall in the range  $109.0 (2)-113.0 (2)^\circ$ , whereas all N1–C–C bond angles fall in the range  $105.8 (1)-112.3 (1)^\circ$ . The piperidinyll ring has a chair conformation, as shown by the torsion angles in Table 1; those involving N1 deviate slightly from those of a perfect chair conformation because of the marginally longer piperidinyll N–C bonds than the C–C bonds. The hydroxyl and exocyclic ammonium groups are mutually *trans*, in equatorial positions. The  $\text{H}_2\text{O}$  molecule is hydrogen bonded *via* intramolecular interactions; the chloride anions are also hydrogen bonded to H atoms on N2 and N1 (see Table 2).



**Figure 1**

The molecular structure of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. H atoms are shown as spheres of an arbitrary radius.

## Experimental

Compound (I) was synthesized from 490 mg (2.86 mmol) of 4-amino-2,2,6,6-tetramethylpiperidine-1-oxyl (Aldrich) and 0.74 ml of 11.6 M HCl (8.58 mmol) in 3.0 ml of ethanol according to a literature procedure used for the synthesis of other piperidin-1-ol hydrochloride derivatives (Sosnovsky & Cai, 1995). At the end of the reaction (2 h at room temperature), 20 ml of diethyl ether was added and the mixture cooled in a refrigerator (277 K) overnight. The precipitated white solid (498 mg, 66% yield) was isolated by suction filtration, followed by 4 h of vacuum drying. White crystals were produced by dissolving (I) in 3.0 ml ethanol, adding 20 ml of diethyl ether, and cooling the mixture at 277 K overnight.

### Crystal data

$C_9H_{22}N_2O^{2+} \cdot 2Cl^- \cdot H_2O$   
 $M_r = 263.21$   
 Orthorhombic, *Pbca*  
 $a = 13.2668(6) \text{ \AA}$   
 $b = 9.1840(5) \text{ \AA}$   
 $c = 24.471(1) \text{ \AA}$   
 $V = 2981.6(5) \text{ \AA}^3$   
 $Z = 8$   
 $D_x = 1.173 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
 Cell parameters from 13231 reflections  
 $\theta = 2.2\text{--}27.9^\circ$   
 $\mu = 0.42 \text{ mm}^{-1}$   
 $T = 173.2 \text{ K}$   
 Platelet, colourless  
 $0.50 \times 0.30 \times 0.10 \text{ mm}$

### Data collection

Rigaku/ADSC CCD diffractometer  
 $\omega$  and  $\varphi$  scans  
 Absorption correction: multi-scan (Blessing, 1995)  
 $T_{\min} = 0.767$ ,  $T_{\max} = 0.959$   
 27028 measured reflections  
 3286 independent reflections

2001 reflections with  $I > 3\sigma(I)$   
 $R_{\text{int}} = 0.058$   
 $\theta_{\text{max}} = 27.9^\circ$   
 $h = -14 \rightarrow 16$   
 $k = -11 \rightarrow 10$   
 $l = -29 \rightarrow 29$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 3\sigma(F^2)] = 0.033$   
 $wR(F^2) = 0.081$   
 $S = 1.10$   
 3286 reflections  
 173 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2)]$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

O1—N1	1.415 (2)	C1—C7	1.525 (3)
N1—C1	1.531 (2)	C2—C3	1.513 (2)
N1—C5	1.534 (2)	C3—C4	1.518 (2)
N2—C3	1.495 (2)	C4—C5	1.525 (2)
C1—C2	1.526 (2)	C5—C8	1.520 (2)
C1—C6	1.522 (3)	C5—C9	1.522 (2)
O1—N1—C1	108.2 (1)	N2—C3—C2	110.2 (1)
O1—N1—C5	109.6 (1)	N2—C3—C4	109.9 (1)
C1—N1—C5	119.3 (1)		
O1—N1—C1—C2	177.1 (1)	N2—C3—C4—C5	177.8 (1)
O1—N1—C5—C4	−176.2 (1)	C1—N1—C5—C4	−50.8 (2)
N1—C1—C2—C3	−53.5 (2)	C1—C2—C3—C4	60.8 (2)
N1—C5—C4—C3	53.4 (2)	C2—C1—N1—C5	51.0 (2)
N2—C3—C2—C1	−178.2 (1)	C2—C3—C4—C5	−61.1 (2)

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H10 $\cdots$ Cl2	0.88 (2)	2.24 (2)	3.117 (6)	173 (2)
O1—H11 $\cdots$ Cl2 <sup>i</sup>	0.88 (2)	2.06 (2)	2.931 (5)	168 (2)
N2—H12 $\cdots$ Cl1	0.98 (2)	2.12 (2)	3.084 (2)	165 (2)
N2—H13 $\cdots$ Cl1 <sup>ii</sup>	0.99 (2)	2.10 (2)	3.079 (2)	167 (2)
N2—H14 $\cdots$ O2	1.01 (2)	1.78 (2)	2.783 (2)	173 (2)
O2—H15 $\cdots$ Cl1 <sup>iii</sup>	0.90 (3)	2.24 (3)	3.135 (2)	170 (2)
O2—H16 $\cdots$ Cl2 <sup>iii</sup>	0.78 (3)	2.33 (3)	3.111 (5)	171 (3)
C6—H6A $\cdots$ O1	0.98	2.42	2.812 (3)	104
C7—H7A $\cdots$ Cl2B	0.98	2.70	3.531 (5)	142
C9—H9B $\cdots$ O1	0.98	2.45	2.863 (3)	105
C9—H9B $\cdots$ O1 <sup>iv</sup>	0.98	2.49	3.347 (2)	145

Symmetry codes: (i)  $2-x, -y, 1-z$ ; (ii)  $\frac{3}{2}-x, \frac{1}{2}+y, z$ ; (iii)  $2-x, \frac{1}{2}+y, \frac{3}{2}-z$ ; (iv)  $2-x, 1-y, 1-z$ .

One chloride ion appears to be disordered, occupying two sites in a 60:40 ratio. All O—H and N—H H atoms were located in difference maps and refined isotropically, while all other H atoms were placed in calculated positions, with C—H distances of 0.98  $\text{\AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{iso}}(\text{C})$  and were refined as riding.

Data collection: *d\*TREK* (Rigaku/MSC, 2001); cell refinement: *d\*TREK*; data reduction: *d\*TREK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *teXsan* (Molecular Structure Corporation, 1992–1997); molecular graphics: *teXsan*; software used to prepare material for publication: *teXsan*.

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