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## Key indicators

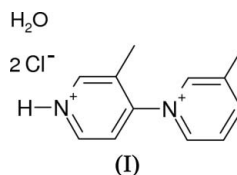
Single-crystal X-ray study  
 $T = 105\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
 $R$  factor = 0.043  
 $wR$  factor = 0.108  
Data-to-parameter ratio = 18.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.3-Methyl-1-(3-methylpyridinium-4-yl)-  
pyridinium dichloride monohydrate

The asymmetric unit of the title compound,  $\text{C}_{12}\text{H}_{14}\text{N}_2^{2+} \cdot 2\text{Cl}^- \cdot \text{H}_2\text{O}$ , comprises a pyridinium dication, two independent  $\text{Cl}^-$  anions and a water molecule. The three components are linked through hydrogen bonding and are packed to form a discrete halide salt structure.

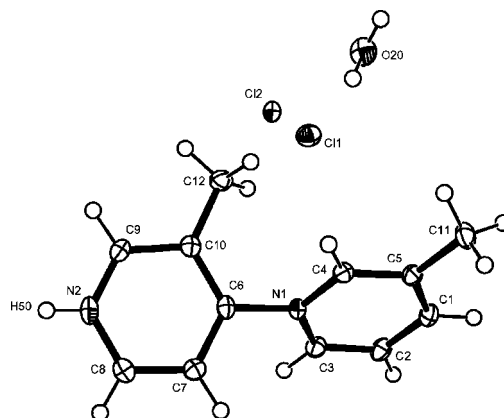
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## Comment

Pyridylpyridinium halides are very susceptible to nucleophilic attack and thereby provide a useful intermediate for the synthesis of desired pyridyl-based organic compounds (Boudszek & Wieczorek, 1980; Jerchel *et al.*, 1956). Our interest in the title compound, (I), and analogous pyridylpyridinium salts, relates to the generation of bidentate bispyridyl ligands with functionalized substituents that will bridge metal ion centres to afford novel coordination materials.



The heterocyclic rings in the cation of (I) (Fig. 1) are connected through a  $\text{C6}-\text{N1}$  bond of  $1.449(3)\text{ \AA}$  and are not coplanar. The dihedral angle between the pyridine rings is  $62.93(3)^\circ$ , and the methyl substituents are in a *syn* conformation. The  $\text{N2}-\text{H50}$  group forms a hydrogen bond to atom  $\text{Cl2}$ , and the water H atoms hydrogen bond to atoms  $\text{Cl1}$  and  $\text{Cl2}$ ; see Table 1 for geometric parameters. The hydrogen bonding links the pyridinium cation,  $\text{Cl}^-$  ions and water molecule to form a discrete unit. Analysis of the crystal



**Figure 1**  
View of the asymmetric unit of (I), showing 50% probability displacements ellipsoids and the atom-numbering scheme.

packing (Fig. 2) reveals no notable interactions, such as further hydrogen bonding or  $\pi$ - $\pi$  stacking, between discrete units.

### Experimental

The pyridinium salt was synthesized according to a modified version of previously reported methods (Boudszek & Wiczorek, 1980). Thionyl chloride (33 ml, 0.452 mol) was added dropwise to dry 3-picoline (12 ml, 0.236 mol) under nitrogen and with cooling. The mixture was stirred at room temperature for 5 d, during which a brown precipitate appeared. The excess thionyl chloride was evaporated under reduced pressure and the crude crystals of the pyridinium salt were purified by warming with charcoal in water. The solvent was evaporated and the residue crystallized from ethanol to afford large single crystals suitable for X-ray analysis.

#### Crystal data

$C_{12}H_{14}N_2^{2+} \cdot 2Cl^- \cdot H_2O$	$Z = 2$
$M_r = 275.17$	$D_x = 1.372 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.3062 (8) \text{ \AA}$	Cell parameters from 2766 reflections
$b = 7.9701 (8) \text{ \AA}$	$\theta = 2.7\text{--}27.8^\circ$
$c = 12.7134 (13) \text{ \AA}$	$\mu = 0.47 \text{ mm}^{-1}$
$\alpha = 78.987 (2)^\circ$	$T = 105 (2) \text{ K}$
$\beta = 74.273 (2)^\circ$	Plate, colourless
$\gamma = 70.079 (2)^\circ$	$0.55 \times 0.26 \times 0.10 \text{ mm}$
$V = 665.97 (12) \text{ \AA}^3$	

#### Data collection

Bruker SMART 1000 CCD diffractometer	3052 independent reflections
$\omega$ scans	2616 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.026$
$T_{\text{min}} = 0.785$ , $T_{\text{max}} = 0.950$	$\theta_{\text{max}} = 28.1^\circ$
6341 measured reflections	$h = -8 \rightarrow 9$
	$k = -10 \rightarrow 10$
	$l = -16 \rightarrow 16$

#### Refinement

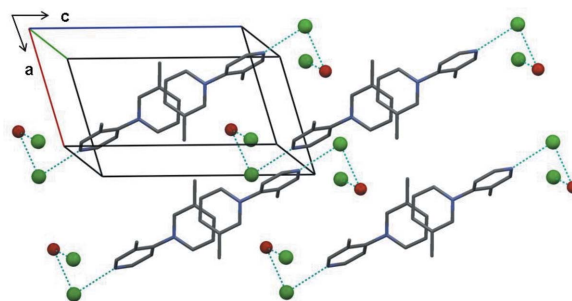
Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0137P)^2 + 1.3181P]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.108$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.20$	$\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
3052 reflections	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
165 parameters	
H atoms treated by a mixture of independent and constrained refinement	

**Table 1**

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O20-H20 \cdots Cl1$	0.89 (4)	2.32 (4)	3.199 (2)	170 (3)
$O20-H21 \cdots Cl2^i$	0.80 (4)	2.40 (4)	3.185 (2)	171 (4)
$N2-H50 \cdots Cl2^{ii}$	0.83 (3)	2.15 (3)	2.975 (2)	174 (3)

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



**Figure 2**

Representation of the crystal packing of (I), viewed down the  $b$  axis, highlighting the discrete hydrogen-bonded units in the structure. H atoms have been omitted for clarity. The Cl and O atoms are shown as green and red balls, respectively, and hydrogen-bonding interactions are indicated as dashed lines.

Aromatic H sites were constrained to their ideal geometric positions, with a C—H distance of 0.95  $\text{\AA}$ . The methyl H atoms were allowed to refine as a rotating group with idealized geometry and were constrained to ride on the parent atom, with a C—H bond length of 0.98  $\text{\AA}$  and H—C—H angles of 109.5 $^\circ$ . Water and NH H atoms were located in a difference map and refined; see Table 1 for distances.  $U_{\text{iso}}(\text{H})$  values were constrained to 1.5 $U_{\text{eq}}$  of the carrier atom for methyl, water and NH H atoms, and to 1.2 $U_{\text{eq}}$  for aromatic H atoms.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT-Plus (Bruker, 1997); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and MERCURY (Version 1.4; Bruno *et al.*, 2002); software used to prepare material for publication: SHELXL97.

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