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1-Ethyl-4-hydroxy-2,6-dimethylpyridinium bromide dihydrate

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Key indicators

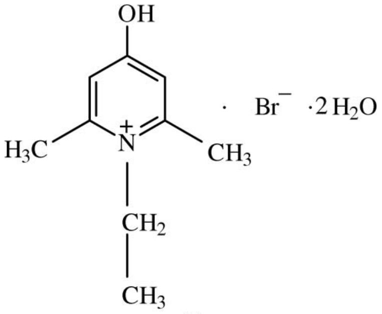
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.021
wR factor = 0.052
Data-to-parameter ratio = 21.1

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

The title compound, $C_9H_{14}NO^+Br^-\cdot 2H_2O$, comprises 1-ethyl-2,6-dimethyl-4-hydroxypyridinium cations and bromide anions, with two solvent water molecules per formula unit. In the crystal structure, the anions, cations and water molecules are linked *via* intermolecular O—H···Br and O—H···O hydrogen bonds, forming layers parallel to the (100) plane.

Comment

2,6-Dimethyl-4-hydroxypyridinone and 4-hydroxypyridinium salts have attracted much attention in the field of non-linear optics (NLO), since the 4-hydroxypyridinium conjugated electronic system could be an interesting hyperpolarizable chromophore for NLO activity (Manivannan *et al.*, 2004; Dhanuskodi *et al.*, 2006). To achieve self-assembly of organic cations in the manner required to exhibit NLO activity (Tamuly *et al.*, 2005), suitable anions must be identified and used effectively. Halide anions have been reported to improve the physicochemical stability of 1-ethyl-2,6-dimethyl-4-(1*H*)-pyridinones (Dhanuskodi *et al.*, 2006). We report here the crystal structure of 1-ethyl-2,6-dimethyl-4-hydroxypyridinium bromide dihydrate (EDMPBr·$2H_2O$), (I).



(I)

The crystal structure of (I) (Fig. 1) comprises 1-ethyl-2,6-dimethyl-4-hydroxypyridinium cations and bromide anions, with two solvent water molecules per formula unit. The C2—N1—C6 bond angle in the cation [120.71 (16)°] is comparable to that in 2,6-dimethylpyridine (Bond & Davies, 2001) and the 2,6-dimethylpyridine-urea complex (Lee & Wallwork, 1965). The organic cations lie in layers parallel to the (100) plane (Fig. 2). The bromide anions and water molecules lie between these layers, forming hydrogen-bonded sheets *via* O—H···O and O—H···Br interactions (Fig. 3 and Table 1). Two distinct ring motifs exist within these sheets, with graph-set descriptors $R_4^6(12)$ and $R_{10}^6(20)$ (Bernstein *et al.*, 1995). O—H···O hydrogen bonds are formed between atom O1 of the hydroxyl

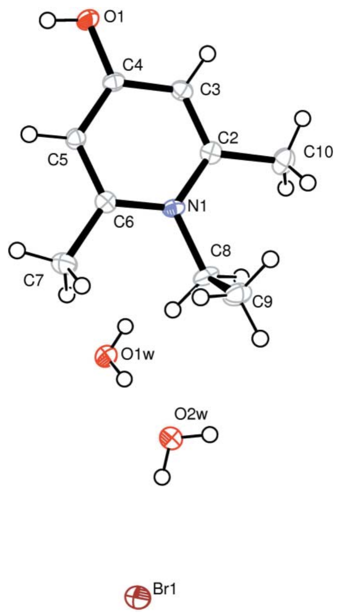


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level. H atoms are represented by circles of arbitrary radius.

group of the organic cation and one of the solvent water molecules (Fig. 2 and Table 1).

Experimental

The title compound was synthesized by dissolving 1-ethyl-2,6-dimethyl-4(1*H*)-pyridinone trihydrate (EDMP·$3H_2O$, 1.51 g) with HBr (2.43 g) in distilled water (5 ml). The solution was stirred well at room temperature for 7 h and the solvent was allowed to evaporate at 323 K. The residual crystalline powder was redissolved in distilled water, and single crystals of (I) were obtained by slow evaporation at 303 K.

Crystal data

$C_9H_{14}NO^+Br^-\cdot 2H_2O$
$M_r = 268.15$
Monoclinic, $P2_1/c$
$a = 10.5747$ (3) Å
$b = 8.0382$ (1) Å
$c = 15.0377$ (4) Å
$\beta = 109.298$ (1)°
$V = 1206.41$ (5) Å^3
$Z = 4$
$D_x = 1.476$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 3.39$ mm^{-1}
$T = 120$ (2) K
Block, colourless
0.54 × 0.48 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.176$, $T_{\max} = 0.666$
15899 measured reflections
2768 independent reflections
2455 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.052$
$S = 1.04$
2768 reflections
131 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0208P)^2 + 0.702P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.52$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0104 (6)

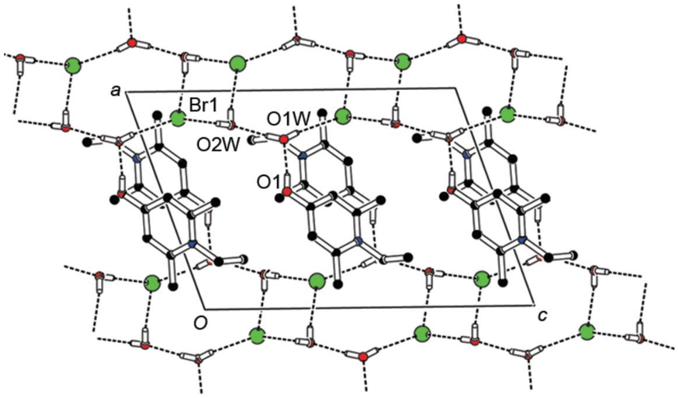


Figure 2
View of (I) along *b*, showing layers of organic cations lying parallel to the (100) plane, with Br^- anions and water molecules lying between them. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

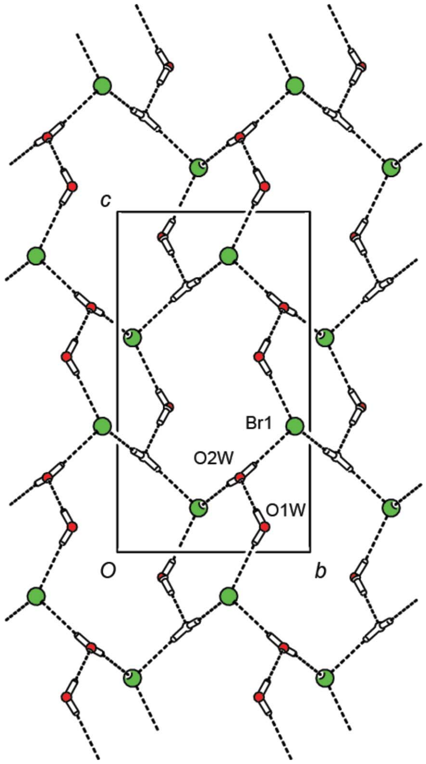


Figure 3
A single Br^-/H_2O sheet viewed along the *a*-axis direction, showing O—H···O and O—H···Br hydrogen bonds (dashed lines).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O1*W*$^{\rm i}$	0.80	1.78	2.5720 (16)	173
O1*W*—H11···O2*W*	0.82	1.90	2.7135 (16)	175
O1*W*—H12···Br1$^{\rm ii}$	0.76	2.50	3.2610 (12)	172
O2*W*—H21···Br1	0.81	2.52	3.3332 (12)	178
O2*W*—H22···Br1$^{\rm iii}$	0.79	2.52	3.3061 (12)	173

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

o600 Seethalakshmi *et al.* • $C_9H_{14}NO^+Br^-\cdot 2H_2O$ *Acta Cryst.* (2007). E**63**, o599–o601

H atoms, except those of the water molecules, were positioned geometrically with C—H = 0.93 (CH), 0.96 (CH_3) or 0.97 Å (CH_2), and with O—H = 0.80 Å. They were then refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C,O)$ or $1.5U_{eq}$(methyl C). H atoms of the water molecules were found in difference Fourier maps and refined initially with a restrained geometry. In the final cycles of refinement, they were made to ride on their parent O atoms, with $U_{iso}(H) = 1.2U_{eq}(O)$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli 1995).

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Acta Cryst. (2007). E**63**, o599–o601 Seethalakshmi *et al.* • $C_9H_{14}NO^+Br^-\cdot 2H_2O$ **o601**