# 2,6-Dimethylpyridinium hydrogen (2*R*,3*R*)-(+)-tartrate sesquihydrate

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**Key indicators**

Single-crystal X-ray study
$T = 292$ K
Mean $\sigma$(C–C) = 0.007 Å
Disorder in main residue
$R$ factor = 0.047
$wR$ factor = 0.107
Data-to-parameter ratio = 7.2

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

The crystal structure of the title compound, $C_7H_{10}N^+$·$C_4H_5O_6^-$·1.5$H_2O$, is based on a three-dimensional supramolecular framework constructed through N—H···O hydrogen bonds and weak $\pi$–$\pi$ interactions.

## Comment

Our groups are currently investigating the supramolecular structures of organic acids and organic bases resulting from hydrogen bonding (Wang & Wei, 2005). The asymmetric unit of the title complex, (I), is composed of one hydrogen (2*R*,3*R*)-(+)-tartrate anion, one 2,6-dimethylpyridinium cation and two water molecules in general positions, one of which has a site-occupation factor of 0.5 (Fig. 1). One carboxyl group of the tartaric acid, at C11, is deprotonated. These ions and water molecules are further linked into a three-dimensional supramolecular framework by N—H···O and O—H···O hydrogen bonds (Fig. 2 and Table 1). In addition, weak $\pi$–$\pi$ interactions are observed between symmetry-related pyridinium rings, with a centroid–centroid distance of 3.999 (3) Å and a dihedral angle of 2.97 (2)° (Fig. 2).



(I)
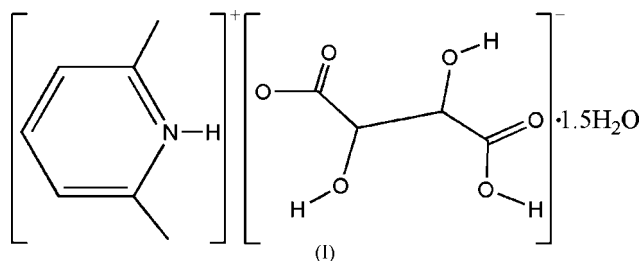
## Experimental

All reagents were commercially available and of analytical grade. (L)-(+)-Tartaric acid (1 mmol, 0.150 g) and 2,6-dimethylpyridine (1 mmol, 0.107 g) were dissolved in a beaker containing 20 ml of distilled water. The solution was stirred for about 20 min at 353 K, avoiding evaporation of 2,6-dimethylpyridine. Colourless crystals of (I) were obtained from the filtrate after four days.

### Crystal data

$C_7H_{10}N^+$·$C_4H_5O_6^-$·1.5$H_2O$
$M_r = 284.27$
Orthorhombic, $P2_12_12_1$
$a = 7.2094$ (11) Å
$b = 12.4483$ (18) Å
$c = 15.628$ (2) Å
$V = 1402.5$ (4) Å$^3$
$Z = 4$
$D_x = 1.346$ Mg m$^{-3}$

Mo $K\alpha$ radiation
Cell parameters from 1276 reflections
$\theta = 2.6$–18.4°
$\mu = 0.11$ mm$^{-1}$
$T = 292$ (2) K
Plate, colourless
$0.36 \times 0.10 \times 0.04$ mm

### Data collection

Bruker SMART APEX CCD area-
  detector diffractometer
$\omega$ scans
Absorption correction: multi-scan
  (*SADABS*; Bruker, 2001)
  $T_{min} = 0.960$, $T_{max} = 0.995$
13627 measured reflections

1447 independent reflections
901 reflections with $I > 2\sigma(I)$
$R_{int} = 0.197$
$\theta_{max} = 25.0°$
$h = -8 \rightarrow 8$
$k = -14 \rightarrow 14$
$l = -18 \rightarrow 18$

### Refinement

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.107$
$S = 0.90$
1447 reflections
202 parameters

H atoms treated by a mixture of
  independent and constrained
  refinement
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.022$
$\Delta\rho_{max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{min} = -0.31$ e Å$^{-3}$

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| N1—H13$\cdots$O6$^i$ | 0.86 | 1.90 | 2.705 (4) | 156 |
| N1—H13$\cdots$O4$^i$ | 0.86 | 2.29 | 2.889 (4) | 127 |
| O1—H1$\cdots$O5$^{ii}$ | 0.82 | 1.78 | 2.565 (4) | 160 |
| O3—H11$\cdots$O2$W^{iii}$ | 0.82 | 2.03 | 2.840 (13) | 168 |
| O3—H11$\cdots$O2 | 0.82 | 2.27 | 2.677 (5) | 111 |
| O4—H12$\cdots$O1$W$ | 0.82 | 1.89 | 2.706 (5) | 179 |
| O1$W$—H1$WA\cdots$O6$^{ii}$ | 0.823 (10) | 2.006 (16) | 2.792 (5) | 160 (4) |
| O1$W$—H1$WB\cdots$O5$^i$ | 0.826 (10) | 2.094 (17) | 2.899 (4) | 165 (5) |
| O2$W$—H2$WB\cdots$O1$W^{iv}$ | 0.831 (11) | 2.23 (5) | 3.016 (12) | 158 (12) |
| O2$W$—H2$WA\cdots$O2$^v$ | 0.830 (11) | 2.28 (11) | 2.807 (12) | 122 (10) |
| O3—H11$\cdots$O2 | 0.82 | 2.27 | 2.677 (5) | 111 |

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

Several unsuccessful attempts were made to obtain a better diffraction pattern. However, (I) invariably crystallizes as thin plates, giving poorly diffracting samples. In the absence of significant anomalous scattering effects, Friedel pairs were merged and the absolute configuration assigned assuming the configuration of the chiral starting material. All H atoms, except for those of water molecules, were placed in calculated positions and refined with constrained distances: O—H = 0.82 Å, N—H = 0.86 Å, and C—H = 0.93, 0.96 and 0.98 Å for methyl, aromatic and methine groups, respectively. Isotropic displacement parameters were fixed at $U_{iso} = 1.5U_{eq}$(carrier atom) for methyl and OH groups and $U_{iso} = 1.2U_{eq}$(carrier atom) for other H atoms. H atoms of water molecules were found in difference maps and refined with a restrained geometry [O—H = 0.83 (1) Å and H$\cdots$H = 1.34 (1) Å]. Initial refinement yielded unusually high displacement parameters for the water atom O2$W$. Also, the residul factors and negative residual peak were bigger than on the introduction of half-occupancy. Introduction of half-occupancy into the refinement produced a significant improvement in the overall precision of the structure.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.
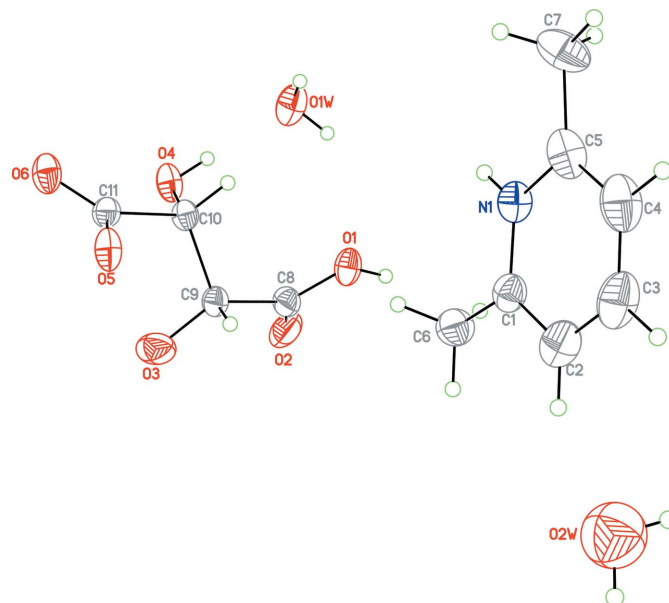
**Figure 1**
The asymmetric unit of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.
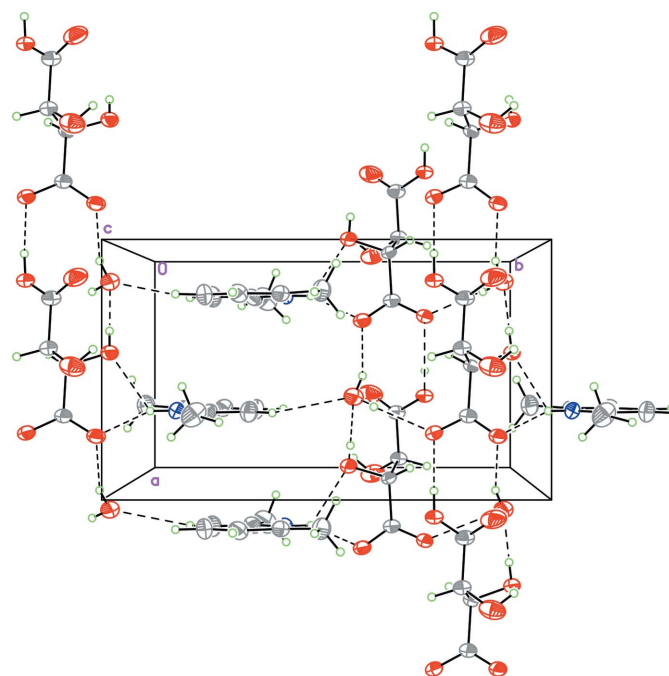


**Figure 2**
Perspective view of the crystal packing along the $c$ axis, showing the linkage of the ions and water molecules by hydrogen-bonding interactions (dashed lines).

## References

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