Acta Crystallographica Section E
**Structure Reports
Online**

ISSN 1600-5368

2-Carboxy-4-(hydroxymethyl)pyridinium chloride hemihydrate

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Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.001 Å
R factor = 0.035
wR factor = 0.101
Data-to-parameter ratio = 25.7

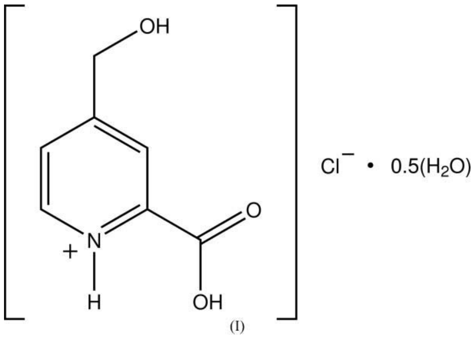
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 29 January 2007
Accepted 30 January 2007

The title compound, $C_7H_8NO_3{}^+\cdot Cl^-\cdot 0.5H_2O$, features inter- and intramolecular O—H···Cl and N—H···O hydrogen bonds, linking the molecules into a three-dimensional network. The water molecule is located on a crystallographic twofold rotation axis.

Comment

Our studies of organometallic immobilization on various supports, for use as catalysts and photochemical devices, have led us to use picolinic acid as a ligand. We have therefore synthesized *para*-methanol-picolinic acid and present here the crystal structure of the title chloride hemihydrate, (I).



(I)

In compound, (I), the pyridine ring is planar and the methanol function is in the plane of the pyridine ring (Fig. 1). The carboxylic acid function is also in the plane of the ring and is stabilized by an intramolecular hydrogen bond involving atom H1*N*.

Atom H1*N* is involved in a bifurcated hydrogen bond to atoms O1 and O3^{i} [symmetry code: (i) $x, 1 - y, z - \frac{1}{2}$], with the sum of the angles at H being 359°. The interaction H1N···O1 = 2.309 (16) Å is an intramolecular contact. In contrast, the interaction H1N···O3^{i} = 1.900 (17) Å is intermolecular and links two molecules into a chain along the crystallographic c axis. The Cl atom bridges the chains along the crystallographic a axis, with H1O···Cl1^{ii} = 2.06 (2) Å and H3O···Cl1 = 2.181 (18) Å [symmetry code: (ii) $x - \frac{1}{2}, \frac{3}{2} - y, z - \frac{1}{2}$]. The water molecule is also involved (H4O···Cl1 = 2.31 Å), forming hydrogen bonds in the c direction, resulting in a three-dimensional network (Fig. 2).

Experimental

2-Cyano-4-[{(*tert*-butyldimethylsilyl)oxy}methyl]pyridine (1.71 g), prepared as reported by El Hadri & Leclerc (1993), was placed in a 4 *M* HCl solution (30 ml). This mixture was heated at reflux for 24 h and then evaporated to dryness. The solid residue was washed three times with acetone. A white crystalline material was obtained in quantitative yield. Colourless crystals of (I) were obtained after recrystallization from acetone–water (9:1).

Crystal data

$C_7H_8NO_3^+\cdot Cl^-\cdot 0.5H_2O$
$M_r = 198.60$
Monoclinic, $C2/c$
$a = 8.1935$ (4) Å
$b = 13.5699$ (6) Å
$c = 15.5198$ (15) Å
$\beta = 98.990$ (3)°
$V = 1704.36$ (19) Å^3
$Z = 8$
$D_x = 1.548$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.42$ mm^{-1}
$T = 110$ (2) K
Block, colourless
0.42 × 0.30 × 0.24 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.83$, $T_{\max} = 0.90$
40008 measured reflections
3753 independent reflections
3274 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 35.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.101$
$S = 1.05$
3753 reflections
146 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 1.7395P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.13$ e Å^{-3}
$\Delta\rho_{\min} = -0.70$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1	0.893 (17)	2.309 (16)	2.6318 (10)	101.1 (12)
N1—H1N···O3$^{\rm i}$	0.893 (17)	1.900 (17)	2.7697 (9)	164.2 (15)
O1—H1O···Cl1$^{\rm ii}$	0.87 (2)	2.06 (2)	2.9309 (7)	173 (2)
O3—H3O···Cl1	0.805 (18)	2.181 (18)	2.9836 (7)	174.7 (17)
O4—H4O···Cl1	0.88	2.31	3.1692 (8)	165

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

All H atoms could be located in a difference Fourier map. The crystallographically independent water H atom was kept fixed in the located position during the refinement. with $U = 0.05$ Å^2. All other C—H, N—H and O—H hydrogens were refined freely with individual isotropic displacement parameters. The final difference Fourier map shows residual density of 1.13 e Å^{-3} was located 0.63 Å from Cl, suggesting minor disorder of Cl. This disorder could not be refined satisfactorily. The O atom of the water molecule is located on a twofold axis. Its displacement parameters are slightly high, indicating a lower occupancy, which can be explained by evaporation of water from the crystal.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *PEAKREF* (Schreurs, 2005); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); 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: *SHELXL97*.

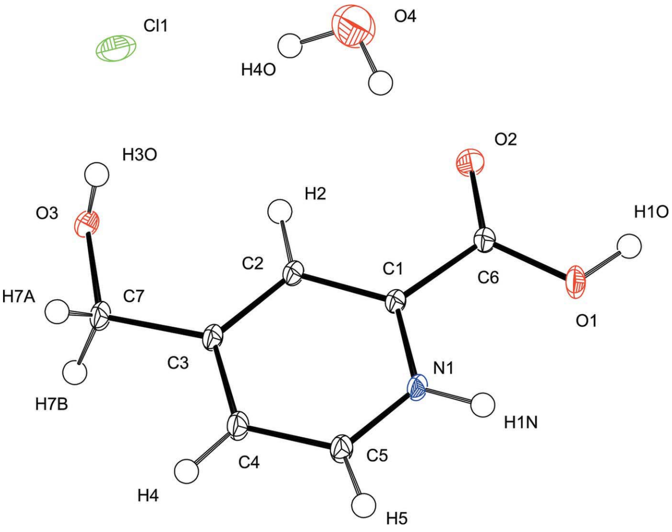


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

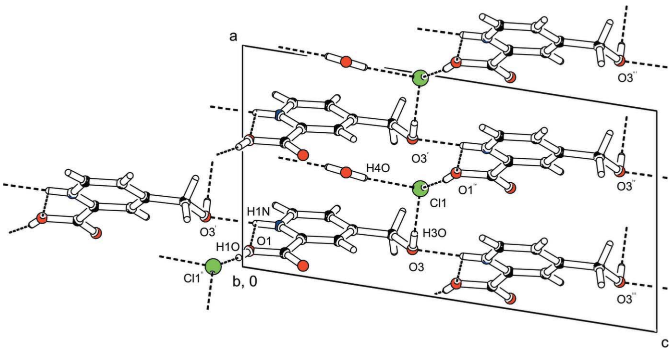


Figure 2
The hydrogen-bond network in (I), viewed along the crystallographic b axis. Hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) $x, 1-y, z-\frac{1}{2}$; (ii) $x-\frac{1}{2}, \frac{3}{2}-y, z-\frac{1}{2}$; (iii) $x, 1-y, z+\frac{1}{2}$; (iv) $x+\frac{1}{2}, \frac{3}{2}-y, z+\frac{1}{2}$; (v) $x+\frac{1}{2}, y+\frac{1}{2}, z$; (vi) $x+1, 2-y, z+\frac{1}{2}$.]

This work was supported by the Council for Chemical Sciences of the Netherlands Organization for Scientific Research (CW-NWO) and the Dutch Technology Foundation (STW-NWO).

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