3-Carboxypyridinium chloride

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Key indicators

Single-crystal X-ray study

T = 293 K

Mean σ(C–C) = 0.004 Å

R factor = 0.031

wR factor = 0.077

Data-to-parameter ratio = 8.5

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

The synthesis, crystallization and structure determination of the title compound, nicotinic acid hydrochloride, $C_6H_6NO_2^+ \cdot Cl^-$, is part of a project on charge densities in crystals. The good quality crystals of the compound composed of light elements and in centrosymmetric space group $P2_1/m$ indicate their suitability for charge–density study.

Comment

The crystal structure of the title compound, (I), is built of protonated nicotinic acid cations $C_6H_6NO_2^+$ and chloride anions. As expected, the nicotinic acid is protonated on the aromatic ring N atom and forms a 3-carboxypyridinium cation (Fig. 1). Interatomic distances and angles are within usual limits (Table 1).

(I)
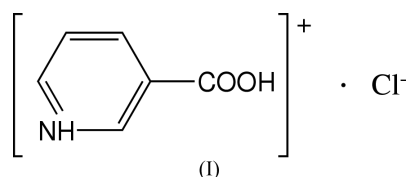
Electrostatic interactions among the ions in the crystal are supplemented with hydrogen bonds (Table 1). The 3-carboxypyridinium O1 atom acts as donor to Cl1 within the asymmetric unit, the 3-carboxypyridinium N1 atom acts as donor to the 3-carboxypyridinium O2(x, y, $z + 1$) atom and, simultaneously, to Cl1(x, y, $z + 1$). In total, there are one two-centre and one three-centre hydrogen bond per cation (Table 2).

The compound crystallizes in the space group $P2_1/m$. Both ions present in the crystal are situated in the special Wyckoff positions e of multiplicity 2 and site symmetry m. This means that both ions lie on the mirror plane (x, $\frac{1}{4}$, z) and the 3-carboxypyridinium cation is strictly planar. As a result, there are parallel planar layers in the crystal structure, on the mirror planes of space group $P2_1/m$. The distance between the layers is 3.3343 (3) Å, half of the lattice parameter b.

Within the layer, there are chains of larger 3-carboxypyridinium cations alternating with smaller Cl^- anions; they are linked through hydrogen bonds (Fig. 2).
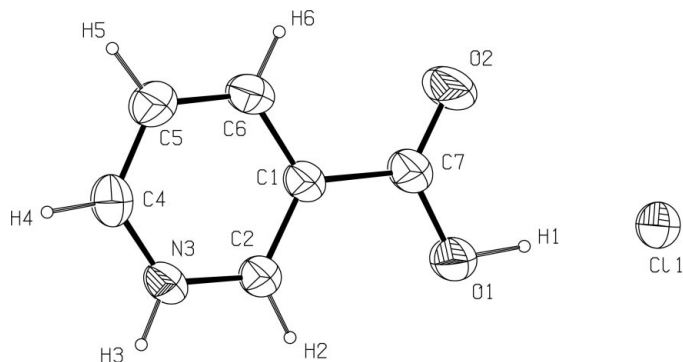
There are no hydrogen-bonding interactions between the layers; examination of the structure with *PLATON* (Spek, 1990) shows that there are no solvent-accessible voids in the unit cell.

This work is a preliminary study, part of a project on charge densities in crystals (Slouf, 2000). The structure of nicotinic acid itself has already been determined (Wright & King, 1953)
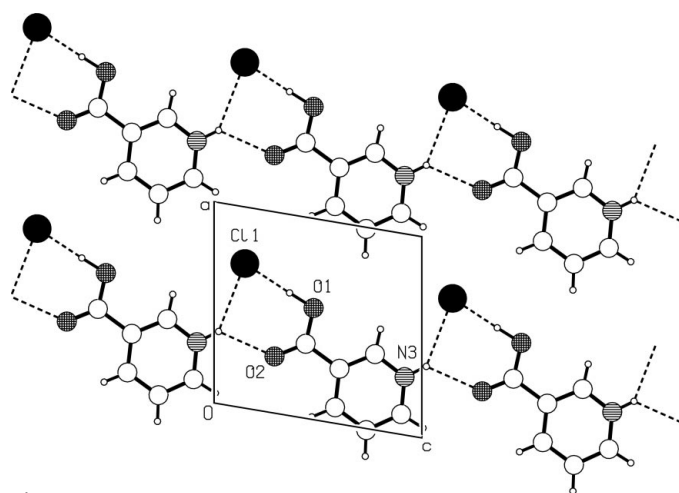
Figure 1
A view of the title compound with displacement ellipsoids at the 50% probability level.

Figure 2
A view of part of the crystal structure of (I), showing the layer identical with mirror plane $(x, \frac{1}{4}, z)$ of space group $P2_1/m$. Hydrogen bonds are indicated by dashed lines.

and redetermined (Gupta & Kumar, 1975; Kutoglu & Scheringer, 1983). In the Cambridge Structural Database (Allen & Kennard, 1993), neither the structure of 3-carboxypyridinium, which is reported here, nor any charge–density study on nicotinic acid was found.

Experimental

An 0.1 *M* methanol solution of nicotinic acid (99% pure) was mixed with an equimolar amount of an 0.1 *M* methanol solution of hydrochloric acid (36% water solution). Crystals suitable for structure determination were grown by very slow evaporation of the mixture.

Crystal data

$C_6H_6O_2N^+ \cdot Cl^-$	$D_x = 1.500$ Mg m^{-3}
$M_r = 159.57$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/m$	Cell parameters from 2406
$a = 7.1704$ (3) Å	reflections
$b = 6.6685$ (6) Å	$\theta = 1$–25°
$c = 7.4937$ (6) Å	$\mu = 0.47$ mm^{-1}
$\beta = 99.555$ (5)°	$T = 293$ (2) K
$V = 353.35$ (5) Å^3	Prism, translucent colourless
$Z = 2$	$0.3 \times 0.2 \times 0.2$ mm

Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.015$
φ and ω scans	$\theta_{max} = 25.0°$
2406 measured reflections	$h = -8 \rightarrow 8$
679 independent reflections	$k = 0 \rightarrow 7$
608 reflections with $I > 2\sigma(I)$	$l = 0 \rightarrow 8$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0263P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.031$	$+ 0.1615P]$
$wR(F^2) = 0.077$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} < 0.001$
679 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
80 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
All H-atom parameters refined	Extinction correction: *SHELXL*97
	Extinction coefficient: 0.090 (17)

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.308 (3)	C2—N3	1.333 (3)
O2—C7	1.197 (3)	N3—C4	1.326 (4)
C7—C1	1.496 (3)	C4—C5	1.369 (4)
C1—C2	1.367 (3)	C5—C6	1.373 (4)
C1—C6	1.383 (4)		
O2—C7—O1	124.5 (2)	O1—C7—C1	114.0 (2)
O2—C7—C1	121.5 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D \cdots A$	D—H$\cdots A$
O1—H1$\cdots$Cl1	1.01 (4)	1.90 (4)	2.9154 (19)	179 (1)
N3—H3$\cdots$O2^i	0.86 (3)	2.18 (3)	2.808 (3)	130 (3)
N3—H3$\cdots$Cl1^i	0.86 (3)	2.60 (3)	3.283 (2)	137 (3)

Symmetry code: (i) $x, y, 1 + z$.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997) and *JANA*2000 (Petricek & Dusek, 2000); structure solution: *SIR*97 (Altomare *et al.*, 1999); structure refinement: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97.

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