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# 2-Oxo-1,2-dihydropyridine-3-carboxylic acid

**Sihui Long,[a] Maxime Siegler[b] and Tonglei Li[a]***

[a]Department of Pharmaceutical Sciences, University of Kentucky, Lexington, KY 40506-0082, USA, and [b]Department of Chemistry, University of Kentucky, Lexington, KY 40506-0055, USA

Correspondence e-mail: tonglei@uky.edu

**Key indicators**

Single-crystal X-ray study
$T$ = 90 K
Mean $\sigma$(C–C) = 0.002 Å
$R$ factor = 0.045
$wR$ factor = 0.130
Data-to-parameter ratio = 14.2

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



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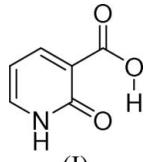
The title compound, $C_6H_5NO_2$, a tautomer of 2-hydroxynicotinic acid, adopts a planar conformation. An intramolecular O—H···O hydrogen bond of 2.504 (2) Å is found. The compound forms one-dimensional hydrogen-bonded chains along the [$\bar{1}$01] direction *via* an intermolecular N—H···O hydrogen bond of 2.810 (2) Å.

## Comment

The title compound, (I), an important intermediate for the synthesis of pharmaceuticals and agrochemicals, is currently commercially available. However, its synthesis is not straightforward. As an alternative, its microbial production from nicotinic acid has been investigated (Tinschert *et al.*, 1997). Its spectroscopic characteristics, and the effect of solvents and pH have been studied with different spectroscopic methods, and quantum chemistry calculations (Dogra, 2005). To date, there has been no report on the solid state structure of this compound. The compound was synthesized by hydrolysis of 2-chloronicotinic acid according to a new procedure (Ting *et al.*, 1990) and single crystals were obtained from an aqueous solution.




(I)

The molecule of (I) (Fig. 1) has an almost perfectly planar conformation. The hydrogen-bonding network in the crystal structure can be described as a one-dimensional hydrogen-

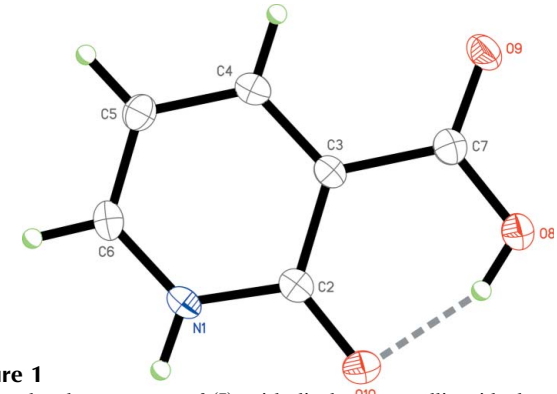



**Figure 1**
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms). The dashed line indicates a hydrogen bond.

bonded chain along the [$\bar{1}$01] direction created by an intermolecular N—H···O hydrogen bond (Table 1 and Fig. 2). An intramolecular $S(6)$ O—H···O hydrogen-bonded motif (Etter, 1990) is also present.

## Experimental

2-Chloronicotinic acid (3.5 g, 22.2 mmol) and 2,4-dichloro-6-methylaniline (3.9 g, 22.2 mmol) were suspended in water (20 ml), followed by addition of pyridine (1.8 ml, 22.2 mmol) and *p*-toluenesulfonic acid (0.8 g, 4.6 mmol). The resulting solution was refluxed overnight. Upon cooling of the solution, colourless crystals precipitated and were characterized by NMR as compound (I) (yield 35%; m.p. 532–535 K).

*Crystal data*

$C_6H_5NO_3$
$M_r$ = 139.11
Monoclinic, $P2_1/n$
$a$ = 3.640 (1) Å
$b$ = 11.584 (3) Å
$c$ = 13.565 (3) Å
$\beta$ = 94.64 (1)°
$V$ = 570.1 (2) Å$^3$
$Z$ = 4
$D_x$ = 1.621 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu$ = 0.13 mm$^{-1}$
$T$ = 90.0 (2) K
Prism, colourless
0.30 × 0.20 × 0.15 mm

*Data collection*

Nonius KappaCCD diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min}$ = 0.961, $T_{max}$ = 0.980
2539 measured reflections
1307 independent reflections
971 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026
$\theta_{max}$ = 27.5°

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.130
$S$ = 1.04
1307 reflections
92 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0761P)^2 + 0.1679P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O9$^{i}$ | 0.88 | 1.95 | 2.8100 (18) | 164 |
| O8—H8···O10 | 0.84 | 1.72 | 2.5040 (17) | 154 |

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

H atoms were positioned geometrically (O—H = 0.84 Å, N—H = 0.88 Å and C—H = 0.93 Å) and refined as riding, with $U_{iso}$(H) = 1.2$U_{eq}$(C,N) or 1.5$U_{eq}$(O).

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97* and local procedures.

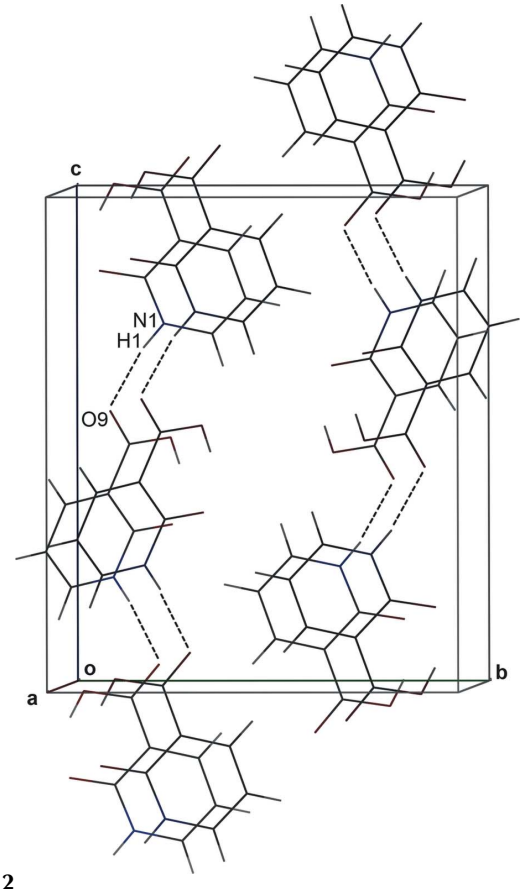



**Figure 2**
A packing diagram of (I), with hydrogen bonds shown as dashed lines.

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