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6-Oxo-1,6-dihydropyridine-3-carboxylic acid

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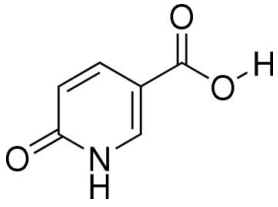
Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.156; data-to-parameter ratio = 14.6.

The title compound, $C_6H_5NO_3$, commonly known as 6-hydroxynicotinic acid, is found to be a tautomer of it. Four molecules, all adopting a planar conformation, are found in the asymmetric unit. The compound forms hydrogen-bonded sheets parallel to the [001] direction *via* intermolecular N—H···O and O—H···O hydrogen bonds. Each sheet consists of interconnected dimers created by $R_2^2(8)$ N—H···O hydrogen-bonded motifs and infinite chains formed by $C(7)$ hydrogen-bonded motifs. Alternatively, these sheets can be viewed as infinitely fused 32-membered hydrogen-bonded rings.

Related literature

See Tinschert *et al.* (1997) for background; Dogra (2005) for spectral studies; Etter (1990) for hydrogen bonding motifs; Long *et al.* (2006) for a similar structure.



Experimental

Crystal data

$C_6H_5NO_3$
M_r = 139.11
Triclinic, $P\overline{1}$
a = 6.8130 (1) Å
b = 11.1340 (3) Å
c = 16.2780 (4) Å
α = 82.5570 (9)°
β = 78.106 (1)°
γ = 76.251 (1)°
V = 1169.43 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 90 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.962, T_{max} = 0.987
10567 measured reflections
5330 independent reflections
3206 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.156
S = 1.00
5330 reflections
365 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2A—H2A···O3D^{i}	0.84	1.76	2.5971 (18)	171
N1D—H1D···O3C	0.88	1.96	2.813 (2)	163
O2D—H2D···O3A^{ii}	0.84	1.76	2.5925 (19)	171
O2B—H2B···O3C	0.84	1.74	2.5681 (19)	171
N1B—H1B···O3A^{iii}	0.88	1.88	2.761 (2)	175
N1A—H1A···O3B^{iii}	0.88	1.93	2.800 (2)	169
O2C—H2C···O3B^{iv}	0.84	1.73	2.5661 (19)	172
N1C—H1C···O3D	0.88	1.90	2.772 (2)	173

Symmetry codes: (i) $-x, -y, -z$; (ii) $-x, -y, -z+1$; (iii) $-x+1, -y-1, -z+1$; (iv) $x, y, z-1$.

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1995); software used to prepare material for publication: *SHELXTL/PC* (Sheldrick, 1995) and local procedures.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2129).

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supplementary materials

Acta Cryst. (2007). E**63**, o2784 [**doi:10.1107/S1600536807020958**]

6-Oxo-1,6-dihydropyridine-3-carboxylic acid

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Comment

The title compound (I), along with 2-hydroxynicotinic acid (II), are useful intermediates for the synthesis of pharmaceuticals and agrochemicals. Microbial production of both compounds from just nicotinic acid has been investigated by different groups. Bacteria which can regio-selectively hydrolyze nicotinic acid at positions 2 and 6 have been isolated successfully (Tinschert *et al.*, 1997). The spectral characteristics of II, and the factors affecting it, have been studied by Dogra (2005). The presence of II was found to be insignificant in various types of solvents because tautomerization led to 1,2-dihydro-2-oxo-3-pyridinecarboxylic acid, the tautomer of II. We reported the first crystal structure of II previously (Long *et al.*, 2006). The solid state structure of II turned out to be 1,2-dihydro-2-oxo-3-pyridinecarboxylic acid. That was in agreement with Dogra's findings. In addition, the compound formed one-dimensional hydrogen-bonded chains along the [-1 0 1] direction *via* intermolecular N—H···O hydrogen bonds. Intramolecular $R_1^1(6)$ O—H···O hydrogen bonds were also found (Etter, 1990).

To systematically study the solid state structures of hydroxynicotinic acids, we further examined the crystal structure of 6-hydroxynicotinic acid, a structural isomer of II in this report.

The asymmetric unit of (I), (Fig. 1), contains four molecules and all the molecules have an almost perfectly planar conformation. Like II, the molecule was measured as 1,6-dihydro-6-oxo-3-pyridinecarboxylic acid, the tautomer of 6-hydroxynicotinic acid. Unlike 1,2-dihydro-2-oxo-3-pyridinecarboxylic acid, whose carboxyl group has an anti conformation, the carboxyl group of I has a *syn* conformation.

The hydrogen bonding network in the crystal (I) is more complicated compared with that of 1,2-dihydro-2-oxo-3-pyridinecarboxylic acid. In the title compound, the hydrogen bonding network can be described as hydrogen-bonded sheets along the [0 0 1] direction *via* intermolecular N—H···O and O—H···O hydrogen bonds (Table 1, Fig. 2). Careful examination of the sheets finds that each sheet consists of inter-connected dimers created by $R^2_2(8)$ N—H···O hydrogen-bonded motifs and infinite chains formed by C(7) hydrogen-bonded motifs according to Etter's notation. Alternately, these sheets can be viewed as infinitely fused 32-membered hydrogen bonded rings.

Experimental

6-Hydroxynicotinic acid was purchased from Alfa Aesar. Crystals of the title compound were grown from dimethylsulfoxide solution by slow evaporation.

Figures

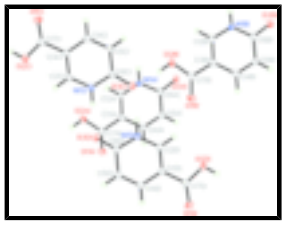


Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms).

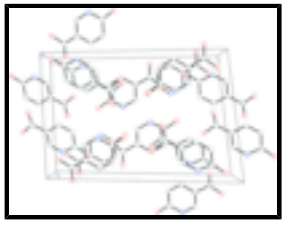


Fig. 2. A packing diagram of (I) along *a* axis.

6-Oxo-1,6-dihydropyridine-3-carboxylic acid

Crystal data

$C_6H_5NO_3$	$Z = 8$
$M_r = 139.11$	$F_{000} = 576$
Triclinic, $P\bar{1}$	$D_x = 1.580$ Mg m^{-3}
$a = 6.8130$ (1) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 11.1340$ (3) Å	Cell parameters from 5285 reflections
$c = 16.2780$ (4) Å	$\theta = 1.0$–27.5°
$\alpha = 82.5570$ (9)°	$\mu = 0.13$ mm^{-1}
$\beta = 78.106$ (1)°	$T = 90$ (2) K
$\gamma = 76.251$ (1)°	Irregular block, colourless
$V = 1169.43$ (5) Å^3	0.30 × 0.20 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer	5330 independent reflections
Radiation source: fine-focus sealed tube	3206 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.042$
Detector resolution: 18 pixels mm^{-1}	$\theta_{max} = 27.5^\circ$
$T = 90(2)$ K	$\theta_{min} = 1.3^\circ$
ω scans at fixed $\chi = 55°$	$h = -8 \rightarrow 8$
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)	$k = -14 \rightarrow 14$
$T_{min} = 0.962$, $T_{max} = 0.987$	$l = -21 \rightarrow 21$
10567 measured reflections	

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.052$

$wR(F^2) = 0.156$

$S = 1.00$

5330 reflections

365 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0889P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.44$ e Å^{-3}

$\Delta\rho_{min} = -0.33$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1A	−0.2663 (2)	0.02267 (12)	0.12836 (9)	0.0246 (4)
N1A	0.0776 (2)	−0.31841 (14)	0.28713 (10)	0.0172 (4)
H1A	0.1450	−0.3964	0.2874	0.021*
O2A	−0.0747 (2)	−0.14740 (12)	0.06663 (9)	0.0228 (4)
H2A	−0.1243	−0.1118	0.0244	0.034*
C2A	0.0217 (3)	−0.26190 (18)	0.21448 (12)	0.0170 (4)
H2A1	0.0576	−0.3062	0.1655	0.020*
O3A	0.0935 (2)	−0.32412 (12)	0.42539 (8)	0.0208 (3)
C3A	−0.0858 (3)	−0.14206 (18)	0.21055 (12)	0.0167 (4)
C4A	−0.1339 (3)	−0.07843 (18)	0.28476 (13)	0.0190 (5)
H4A	−0.2095	0.0051	0.2837	0.023*
C5A	−0.0734 (3)	−0.13532 (18)	0.35741 (13)	0.0202 (5)
H5A	−0.1038	−0.0908	0.4061	0.024*
C6A	0.0358 (3)	−0.26180 (18)	0.36048 (12)	0.0178 (5)
C7A	−0.1531 (3)	−0.07933 (18)	0.13204 (12)	0.0184 (5)
O1B	0.3848 (2)	−0.08869 (13)	0.40114 (9)	0.0266 (4)

N1B	0.6711 (2)	−0.43693 (14)	0.57089 (10)	0.0178 (4)
H1B	0.7396	−0.5146	0.5717	0.021*
O2B	0.5393 (2)	−0.27162 (12)	0.34799 (9)	0.0223 (3)
H2B	0.5115	−0.2324	0.3027	0.033*
C2B	0.6289 (3)	−0.37921 (18)	0.49640 (12)	0.0177 (5)
H2B1	0.6717	−0.4232	0.4473	0.021*
O3B	0.6605 (2)	−0.44604 (12)	0.71141 (9)	0.0225 (4)
C3B	0.5257 (3)	−0.25881 (18)	0.49095 (12)	0.0173 (4)
C4B	0.4673 (3)	−0.19573 (19)	0.56541 (13)	0.0190 (5)
H4B	0.3986	−0.1108	0.5629	0.023*
C5B	0.5084 (3)	−0.25521 (17)	0.64043 (13)	0.0179 (4)
H5B	0.4665	−0.2120	0.6899	0.021*
C6B	0.6141 (3)	−0.38239 (19)	0.64511 (13)	0.0184 (5)
C7B	0.4748 (3)	−0.19628 (18)	0.40984 (13)	0.0194 (5)
N1C	0.4331 (2)	−0.18372 (15)	0.06937 (10)	0.0177 (4)
H1C	0.3515	−0.1095	0.0698	0.021*
O1C	0.7973 (2)	−0.51108 (12)	−0.09714 (9)	0.0256 (4)
O2C	0.5683 (2)	−0.35163 (12)	−0.14625 (9)	0.0249 (4)
H2C	0.6060	−0.3885	−0.1906	0.037*
C2C	0.4836 (3)	−0.23954 (18)	−0.00307 (12)	0.0169 (4)
H2C1	0.4297	−0.1989	−0.0512	0.020*
O3C	0.4444 (2)	−0.17246 (12)	0.20599 (8)	0.0204 (3)
C3C	0.6111 (3)	−0.35363 (17)	−0.00764 (12)	0.0167 (4)
C4C	0.6874 (3)	−0.41203 (18)	0.06576 (13)	0.0198 (5)
H4C	0.7786	−0.4912	0.0638	0.024*
C5C	0.6310 (3)	−0.35561 (17)	0.13909 (13)	0.0189 (5)
H5C	0.6791	−0.3970	0.1883	0.023*
C6C	0.5007 (3)	−0.23520 (18)	0.14232 (12)	0.0165 (4)
C7C	0.6702 (3)	−0.41509 (18)	−0.08724 (13)	0.0186 (5)
O1D	−0.1487 (2)	0.40702 (12)	0.36990 (9)	0.0247 (4)
N1D	0.2167 (2)	0.07385 (15)	0.20876 (10)	0.0185 (4)
H1D	0.3099	0.0039	0.2075	0.022*
O2D	0.0118 (2)	0.22789 (12)	0.43099 (9)	0.0243 (4)
H2D	−0.0248	0.2668	0.4744	0.036*
C2D	0.1569 (3)	0.12889 (17)	0.28185 (12)	0.0174 (5)
H2D1	0.2118	0.0904	0.3302	0.021*
O3D	0.2057 (2)	0.05834 (12)	0.07258 (8)	0.0202 (3)
C3D	0.0185 (3)	0.23908 (18)	0.28634 (12)	0.0170 (4)
C4D	−0.0627 (3)	0.29300 (18)	0.21338 (13)	0.0202 (5)
H4D	−0.1589	0.3704	0.2152	0.024*
C5D	−0.0047 (3)	0.23539 (18)	0.14063 (13)	0.0197 (5)
H5D	−0.0617	0.2721	0.0924	0.024*
C6D	0.1415 (3)	0.12008 (18)	0.13673 (12)	0.0170 (4)
C7D	−0.0489 (3)	0.30178 (18)	0.36576 (13)	0.0182 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

O1A	0.0280 (8)	0.0195 (8)	0.0241 (9)	0.0008 (6)	−0.0087 (7)	0.0007 (7)
N1A	0.0215 (9)	0.0129 (9)	0.0154 (10)	0.0005 (7)	−0.0047 (7)	−0.0013 (7)
O2A	0.0314 (9)	0.0193 (8)	0.0170 (8)	0.0006 (6)	−0.0106 (7)	−0.0011 (6)
C2A	0.0202 (11)	0.0195 (11)	0.0112 (11)	−0.0026 (9)	−0.0043 (8)	−0.0021 (8)
O3A	0.0283 (8)	0.0190 (8)	0.0135 (8)	0.0012 (6)	−0.0068 (6)	−0.0025 (6)
C3A	0.0181 (10)	0.0159 (10)	0.0161 (11)	−0.0040 (8)	−0.0040 (8)	0.0004 (8)
C4A	0.0197 (11)	0.0140 (10)	0.0223 (12)	−0.0004 (8)	−0.0042 (9)	−0.0036 (9)
C5A	0.0237 (11)	0.0180 (11)	0.0185 (12)	−0.0021 (9)	−0.0028 (9)	−0.0058 (9)
C6A	0.0184 (10)	0.0215 (11)	0.0140 (11)	−0.0038 (8)	−0.0045 (9)	−0.0015 (9)
C7A	0.0180 (11)	0.0201 (12)	0.0189 (12)	−0.0058 (9)	−0.0047 (9)	−0.0030 (9)
O1B	0.0340 (9)	0.0199 (8)	0.0220 (9)	0.0033 (7)	−0.0083 (7)	0.0003 (6)
N1B	0.0218 (9)	0.0135 (9)	0.0168 (10)	0.0006 (7)	−0.0053 (7)	−0.0024 (7)
O2B	0.0308 (8)	0.0209 (8)	0.0142 (8)	−0.0014 (7)	−0.0076 (7)	−0.0003 (6)
C2B	0.0214 (11)	0.0187 (11)	0.0134 (11)	−0.0033 (9)	−0.0052 (9)	−0.0016 (8)
O3B	0.0296 (8)	0.0199 (8)	0.0159 (8)	0.0021 (6)	−0.0081 (6)	−0.0017 (6)
C3B	0.0181 (10)	0.0174 (11)	0.0164 (11)	−0.0036 (8)	−0.0049 (9)	0.0005 (8)
C4B	0.0190 (11)	0.0154 (10)	0.0218 (12)	−0.0008 (8)	−0.0053 (9)	−0.0017 (9)
C5B	0.0193 (11)	0.0178 (11)	0.0153 (11)	−0.0004 (8)	−0.0019 (8)	−0.0056 (8)
C6B	0.0184 (11)	0.0238 (11)	0.0133 (11)	−0.0042 (9)	−0.0032 (9)	−0.0039 (9)
C7B	0.0203 (11)	0.0188 (12)	0.0187 (12)	−0.0030 (9)	−0.0045 (9)	−0.0005 (9)
N1C	0.0211 (9)	0.0155 (9)	0.0145 (9)	0.0016 (7)	−0.0055 (7)	−0.0011 (7)
O1C	0.0300 (9)	0.0208 (8)	0.0240 (9)	0.0026 (7)	−0.0065 (7)	−0.0073 (7)
O2C	0.0326 (9)	0.0223 (8)	0.0174 (8)	0.0035 (7)	−0.0086 (7)	−0.0045 (6)
C2C	0.0206 (11)	0.0182 (11)	0.0119 (11)	−0.0038 (8)	−0.0043 (8)	−0.0003 (8)
O3C	0.0259 (8)	0.0187 (8)	0.0154 (8)	−0.0010 (6)	−0.0044 (6)	−0.0037 (6)
C3C	0.0193 (11)	0.0166 (11)	0.0140 (11)	−0.0034 (8)	−0.0026 (8)	−0.0026 (8)
C4C	0.0215 (11)	0.0130 (10)	0.0236 (12)	0.0003 (8)	−0.0057 (9)	−0.0015 (9)
C5C	0.0238 (11)	0.0143 (11)	0.0178 (11)	0.0006 (8)	−0.0092 (9)	0.0014 (8)
C6C	0.0200 (11)	0.0188 (11)	0.0115 (11)	−0.0063 (8)	−0.0031 (8)	−0.0001 (9)
C7C	0.0217 (11)	0.0169 (11)	0.0181 (11)	−0.0049 (9)	−0.0042 (9)	−0.0022 (9)
O1D	0.0304 (8)	0.0187 (8)	0.0230 (9)	0.0010 (7)	−0.0040 (7)	−0.0072 (6)
N1D	0.0226 (9)	0.0160 (9)	0.0152 (9)	0.0015 (7)	−0.0049 (7)	−0.0033 (7)
O2D	0.0311 (9)	0.0237 (8)	0.0161 (8)	0.0019 (7)	−0.0060 (7)	−0.0065 (6)
C2D	0.0236 (11)	0.0163 (11)	0.0133 (11)	−0.0050 (9)	−0.0047 (9)	−0.0011 (8)
O3D	0.0270 (8)	0.0171 (8)	0.0156 (8)	−0.0006 (6)	−0.0052 (6)	−0.0044 (6)
C3D	0.0190 (11)	0.0166 (11)	0.0157 (11)	−0.0042 (8)	−0.0020 (9)	−0.0039 (8)
C4D	0.0208 (11)	0.0158 (11)	0.0226 (12)	0.0003 (8)	−0.0061 (9)	−0.0017 (9)
C5D	0.0236 (11)	0.0184 (11)	0.0170 (11)	−0.0035 (9)	−0.0073 (9)	0.0027 (9)
C6D	0.0224 (11)	0.0154 (10)	0.0146 (11)	−0.0059 (8)	−0.0046 (9)	−0.0008 (8)
C7D	0.0177 (10)	0.0183 (11)	0.0190 (12)	−0.0025 (9)	−0.0042 (9)	−0.0036 (9)

Geometric parameters (Å, °)

O1A—C7A	1.214 (2)	N1C—C2C	1.348 (2)
N1A—C2A	1.348 (2)	N1C—C6C	1.369 (2)
N1A—C6A	1.369 (2)	N1C—H1C	0.8800
N1A—H1A	0.8800	O1C—C7C	1.211 (2)
O2A—C7A	1.333 (2)	O2C—C7C	1.330 (2)
O2A—H2A	0.8400	O2C—H2C	0.8400

C2A—C3A	1.361 (3)	C2C—C3C	1.359 (3)
C2A—H2A1	0.9500	C2C—H2C1	0.9500
O3A—C6A	1.267 (2)	O3C—C6C	1.267 (2)
C3A—C4A	1.420 (3)	C3C—C4C	1.419 (3)
C3A—C7A	1.480 (3)	C3C—C7C	1.481 (3)
C4A—C5A	1.361 (3)	C4C—C5C	1.363 (3)
C4A—H4A	0.9500	C4C—H4C	0.9500
C5A—C6A	1.428 (3)	C5C—C6C	1.422 (3)
C5A—H5A	0.9500	C5C—H5C	0.9500
O1B—C7B	1.214 (2)	O1D—C7D	1.209 (2)
N1B—C2B	1.349 (2)	N1D—C2D	1.352 (2)
N1B—C6B	1.365 (2)	N1D—C6D	1.367 (2)
N1B—H1B	0.8800	N1D—H1D	0.8800
O2B—C7B	1.331 (2)	O2D—C7D	1.330 (2)
O2B—H2B	0.8400	O2D—H2D	0.8400
C2B—C3B	1.359 (3)	C2D—C3D	1.358 (3)
C2B—H2B1	0.9500	C2D—H2D1	0.9500
O3B—C6B	1.266 (2)	O3D—C6D	1.269 (2)
C3B—C4B	1.418 (3)	C3D—C4D	1.416 (3)
C3B—C7B	1.478 (3)	C3D—C7D	1.485 (3)
C4B—C5B	1.361 (3)	C4D—C5D	1.361 (3)
C4B—H4B	0.9500	C4D—H4D	0.9500
C5B—C6B	1.428 (3)	C5D—C6D	1.425 (3)
C5B—H5B	0.9500	C5D—H5D	0.9500
C2A—N1A—C6A	123.93 (17)	C2C—N1C—C6C	124.17 (17)
C2A—N1A—H1A	118.0	C2C—N1C—H1C	117.9
C6A—N1A—H1A	118.0	C6C—N1C—H1C	117.9
C7A—O2A—H2A	109.5	C7C—O2C—H2C	109.5
N1A—C2A—C3A	120.71 (18)	N1C—C2C—C3C	120.42 (18)
N1A—C2A—H2A1	119.6	N1C—C2C—H2C1	119.8
C3A—C2A—H2A1	119.6	C3C—C2C—H2C1	119.8
C2A—C3A—C4A	117.94 (18)	C2C—C3C—C4C	118.25 (18)
C2A—C3A—C7A	121.57 (18)	C2C—C3C—C7C	120.91 (18)
C4A—C3A—C7A	120.48 (17)	C4C—C3C—C7C	120.84 (17)
C5A—C4A—C3A	121.00 (18)	C5C—C4C—C3C	120.62 (18)
C5A—C4A—H4A	119.5	C5C—C4C—H4C	119.7
C3A—C4A—H4A	119.5	C3C—C4C—H4C	119.7
C4A—C5A—C6A	120.07 (18)	C4C—C5C—C6C	120.38 (18)
C4A—C5A—H5A	120.0	C4C—C5C—H5C	119.8
C6A—C5A—H5A	120.0	C6C—C5C—H5C	119.8
O3A—C6A—N1A	118.68 (17)	O3C—C6C—N1C	118.10 (17)
O3A—C6A—C5A	124.99 (18)	O3C—C6C—C5C	125.77 (18)
N1A—C6A—C5A	116.33 (18)	N1C—C6C—C5C	116.12 (17)
O1A—C7A—O2A	124.41 (18)	O1C—C7C—O2C	124.41 (18)
O1A—C7A—C3A	123.34 (19)	O1C—C7C—C3C	123.70 (18)
O2A—C7A—C3A	112.25 (16)	O2C—C7C—C3C	111.89 (16)
C2B—N1B—C6B	124.13 (17)	C2D—N1D—C6D	124.12 (16)
C2B—N1B—H1B	117.9	C2D—N1D—H1D	117.9
C6B—N1B—H1B	117.9	C6D—N1D—H1D	117.9

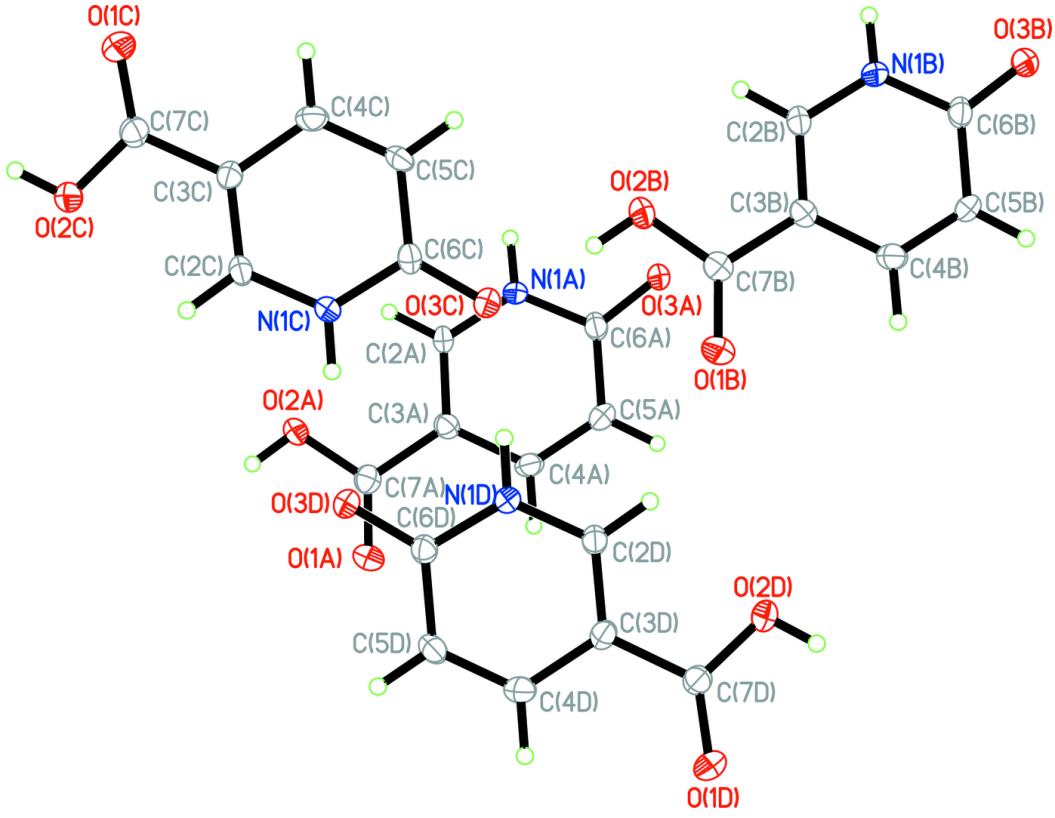
C7B—O2B—H2B	109.5	C7D—O2D—H2D	109.5
N1B—C2B—C3B	120.68 (18)	N1D—C2D—C3D	120.23 (18)
N1B—C2B—H2B1	119.7	N1D—C2D—H2D1	119.9
C3B—C2B—H2B1	119.7	C3D—C2D—H2D1	119.9
C2B—C3B—C4B	117.98 (18)	C2D—C3D—C4D	118.41 (18)
C2B—C3B—C7B	120.79 (18)	C2D—C3D—C7D	121.28 (18)
C4B—C3B—C7B	121.23 (17)	C4D—C3D—C7D	120.30 (17)
C5B—C4B—C3B	120.88 (18)	C5D—C4D—C3D	120.83 (18)
C5B—C4B—H4B	119.6	C5D—C4D—H4D	119.6
C3B—C4B—H4B	119.6	C3D—C4D—H4D	119.6
C4B—C5B—C6B	120.28 (18)	C4D—C5D—C6D	120.13 (19)
C4B—C5B—H5B	119.9	C4D—C5D—H5D	119.9
C6B—C5B—H5B	119.9	C6D—C5D—H5D	119.9
O3B—C6B—N1B	118.63 (18)	O3D—C6D—N1D	118.35 (17)
O3B—C6B—C5B	125.34 (18)	O3D—C6D—C5D	125.40 (18)
N1B—C6B—C5B	116.02 (18)	N1D—C6D—C5D	116.25 (17)
O1B—C7B—O2B	124.30 (19)	O1D—C7D—O2D	124.66 (18)
O1B—C7B—C3B	123.60 (19)	O1D—C7D—C3D	123.49 (18)
O2B—C7B—C3B	112.10 (16)	O2D—C7D—C3D	111.84 (16)
C6A—N1A—C2A—C3A	−1.1 (3)	C6C—N1C—C2C—C3C	−0.8 (3)
N1A—C2A—C3A—C4A	0.9 (3)	N1C—C2C—C3C—C4C	0.5 (3)
N1A—C2A—C3A—C7A	−178.44 (17)	N1C—C2C—C3C—C7C	−179.07 (17)
C2A—C3A—C4A—C5A	0.4 (3)	C2C—C3C—C4C—C5C	1.0 (3)
C7A—C3A—C4A—C5A	179.77 (17)	C7C—C3C—C4C—C5C	−179.38 (17)
C3A—C4A—C5A—C6A	−1.6 (3)	C3C—C4C—C5C—C6C	−2.3 (3)
C2A—N1A—C6A—O3A	179.19 (17)	C2C—N1C—C6C—O3C	179.05 (16)
C2A—N1A—C6A—C5A	−0.1 (3)	C2C—N1C—C6C—C5C	−0.4 (3)
C4A—C5A—C6A—O3A	−177.83 (19)	C4C—C5C—C6C—O3C	−177.47 (19)
C4A—C5A—C6A—N1A	1.4 (3)	C4C—C5C—C6C—N1C	2.0 (3)
C2A—C3A—C7A—O1A	172.31 (19)	C2C—C3C—C7C—O1C	173.01 (19)
C4A—C3A—C7A—O1A	−7.0 (3)	C4C—C3C—C7C—O1C	−6.6 (3)
C2A—C3A—C7A—O2A	−7.9 (3)	C2C—C3C—C7C—O2C	−6.5 (3)
C4A—C3A—C7A—O2A	172.71 (16)	C4C—C3C—C7C—O2C	173.96 (17)
C6B—N1B—C2B—C3B	0.8 (3)	C6D—N1D—C2D—C3D	−2.2 (3)
N1B—C2B—C3B—C4B	1.0 (3)	N1D—C2D—C3D—C4D	1.0 (3)
N1B—C2B—C3B—C7B	−178.48 (17)	N1D—C2D—C3D—C7D	−179.68 (17)
C2B—C3B—C4B—C5B	−1.8 (3)	C2D—C3D—C4D—C5D	0.5 (3)
C7B—C3B—C4B—C5B	177.66 (17)	C7D—C3D—C4D—C5D	−178.90 (17)
C3B—C4B—C5B—C6B	0.9 (3)	C3D—C4D—C5D—C6D	−0.8 (3)
C2B—N1B—C6B—O3B	179.36 (17)	C2D—N1D—C6D—O3D	−178.20 (16)
C2B—N1B—C6B—C5B	−1.8 (3)	C2D—N1D—C6D—C5D	1.8 (3)
C4B—C5B—C6B—O3B	179.67 (19)	C4D—C5D—C6D—O3D	179.71 (18)
C4B—C5B—C6B—N1B	0.9 (3)	C4D—C5D—C6D—N1D	−0.3 (3)
C2B—C3B—C7B—O1B	−179.27 (19)	C2D—C3D—C7D—O1D	169.38 (19)
C4B—C3B—C7B—O1B	1.3 (3)	C4D—C3D—C7D—O1D	−11.3 (3)
C2B—C3B—C7B—O2B	0.7 (3)	C2D—C3D—C7D—O2D	−11.3 (3)
C4B—C3B—C7B—O2B	−178.76 (17)	C4D—C3D—C7D—O2D	168.02 (17)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2A—H2A···O3D^{i}	0.84	1.76	2.5971 (18)	171
N1D—H1D···O3C	0.88	1.96	2.813 (2)	163
O2D—H2D···O3A^{ii}	0.84	1.76	2.5925 (19)	171
O2B—H2B···O3C	0.84	1.74	2.5681 (19)	171
N1B—H1B···O3A^{iii}	0.88	1.88	2.761 (2)	175
N1A—H1A···O3B^{iii}	0.88	1.93	2.800 (2)	169
O2C—H2C···O3B^{iv}	0.84	1.73	2.5661 (19)	172
N1C—H1C···O3D	0.88	1.90	2.772 (2)	173

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) −*x*, −*y*, −*z*+1; (iii) −*x*+1, −*y*−1, −*z*+1; (iv) *x*, *y*, *z*−1.

Fig. 1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C(4C)
C(5C)
C(3C)
O(2C)
C(2C)
C(6C)
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N(1A)
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C(6B)
C(5B)
C(4B)

Fig. 2