Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Pyridine-2,4-dicarboxylic acid *N*-oxide

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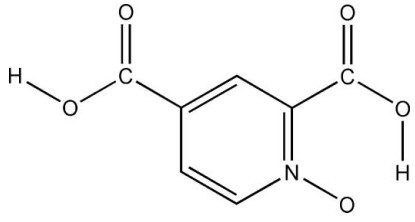
Received 4 June 2007; accepted 11 August 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.096; data-to-parameter ratio = 13.3.

The title compound, $C_7H_5NO_5$, was obtained as rectangular prism-like crystals by the hydroponic method. It displays a planar one-dimensional chain structure stabilized *via* inter- and intramolecular O—H···O hydrogen bonds.

Related literature

For related literature, see: Allen *et al.* (1987); Dideberg & Dupont (1975); Li *et al.* (1987); Rychlewska & Gdaniec (1977); Steiner *et al.* (2000).



Experimental

Crystal data

$C_7H_5NO_5$
M_r = 183.12
Monoclinic, $P2_1/n$
a = 7.323 (3) Å
b = 6.985 (3) Å
c = 13.982 (6) Å
β = 95.614 (4)°
V = 711.8 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 293 (2) K
0.45 × 0.40 × 0.25 mm

Data collection

Bruker P4 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
T_{min} = 0.920, T_{max} = 0.963
4998 measured reflections
1619 independent reflections
1472 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.096
S = 1.04
1619 reflections
122 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2B···O1	0.82	1.69	2.4508 (15)	154
O4—H4B···O3^{i}	0.866 (19)	1.80 (2)	2.6481 (14)	165.6 (18)

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

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

The project was supported by the National Science Foundation of Fujian Province of China (Nos. 2006J164 and 2003F006) and the National Science Foundation of the Overseas Chinese Affairs Office of the State Council, China (No. 05QZR01).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RT2012).

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  doi:10.1107/S160053680703992X

supplementary materials

Acta Cryst. (2007). E**63**, o3950 [**doi:10.1107/S160053680703992X**]

Pyridine-2,4-dicarboxylic acid *N*-oxide

H.-P. Li, W.-S. Wu, G.-D. Yang and G.-X. Liu

Comment

The structure of the title compound, (I) reveals a virtually planar molecule with r.m.s. of 0.0061 Å. All bond distances and angles are normal and the structure is stabilized by inter- and intramolecular hydrogen bonds. The molecules are linked in one-dimensional zigzag chains *via* intermolecular carboxylic hydrogen bonds (Figure 2; Table 2) between O4 and O3. On the other hand, there is a weaker but prominent intermolecular hydrogen bond between H5A and O5 which provides inter chain stabilization, yielding a planar two-dimensional arrangement. Furthermore, the structure displays a strong, almost ideal intramolecular carboxylic hydrogen bond (Figure 1; Table 2) between O2 and O1 (Dideberg & Dupont, 1975; Rychlewska & Gdaniec, 1977; Steiner *et al.*, 2000). The carboxyl C—O and C═O bonds are normal (Table 1), while the N—O distance of 1.324 (1)Å is slightly elongated compared to the average value of 1.304Å in pyridine N-oxides (Allen *et al.*, 1992). The carboxylic hydrogen bonds display O—H···O angles around 160° (Table 2) due to stereochemical restriction of the C—O—H angle.

Experimental

Glacial acetic acid (30 ml) and cold hydrogen peroxide (15 ml) were shaken in a round-bottomed flask, followed by the addition of pyridine-2,4- dicarboxylic acid monohydrate (2 g; 11 mmol), and the mixture was heated (Li *et al.*, 1987). After the excess acetic acid and water were removed under reduced pressure, the title compound was obtained as a white powder (0.95 g, yield 48%). Slow evaporation at room temperature, following dissolution of the white powder (0.2 g, 1 mmol) in a mixture of boiling mixture of methanol (10 ml) and dichloromethane(15 ml) and careful filtration, yielded colorless crystals after 4 days.

Refinement

The C-bound H atoms were included in the riding model approximation with C—H = 0.93Å and U_{iso}(H) = 1.2U_{eq}(C) and the O2-bound H atom was included in the riding model approximation with O—H = 0.82 Å. The O4-bound H atom was located from a difference Fourier map and refined isotropically.

Figures

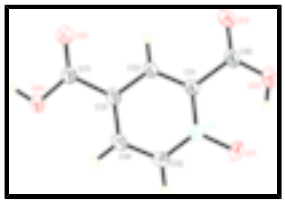

Fig. 1. *ORTEP* drawing (at 50% probability) of (I); hydrogen atoms at arbitrary size.

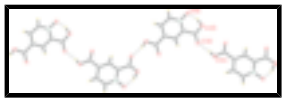

Fig. 2. Illustration of the hydrogen bonding in the one-dimensional chain.

Pyridine-2,4-dicarboxylic acid N-oxide

Crystal data

$C_7H_5NO_5$	F_{000} = 376
M_r = 183.12	D_x = 1.709 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1853 reflections
a = 7.323 (3) Å	θ = 2.8–27.5º
b = 6.985 (3) Å	μ = 0.15 mm^{-1}
c = 13.982 (6) Å	T = 293 (2) K
β = 95.614 (4)º	Prism, colorless
V = 711.8 (5) Å^3	0.45 × 0.40 × 0.25 mm
Z = 4	

Data collection

Bruker P4 diffractometer	1472 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.011
Monochromator: graphite	θ_{max} = 27.5º
T = 293(2) K	θ_{min} = 3.0º
CCD_Profile_fitting scans	h = −4→9
Absorption correction: multi-scan (CrystalClear; Rigaku, 2000)	k = −9→9
T_{min} = 0.920, T_{max} = 0.963	l = −18→18
4998 measured reflections	Standard reflections: ?
1619 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.035	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.096	$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.2243P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
1619 reflections	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
122 parameters	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.48719 (13)	0.26168 (14)	0.73729 (7)	0.0278 (2)
O1	0.41895 (14)	0.39139 (13)	0.79298 (7)	0.0398 (3)
O2	0.31826 (15)	0.13474 (15)	0.89520 (7)	0.0448 (3)
H2B	0.3347	0.2406	0.8721	0.067*
O3	0.40768 (15)	−0.16328 (14)	0.86771 (7)	0.0426 (3)
O4	0.79194 (13)	−0.07382 (13)	0.48418 (6)	0.0353 (2)
H4B	0.838 (2)	−0.169 (3)	0.4549 (13)	0.052 (5)*
O5	0.72985 (14)	−0.31254 (13)	0.58244 (7)	0.0405 (3)
C1	0.48397 (15)	0.07171 (16)	0.75937 (8)	0.0257 (2)
C2	0.56127 (15)	−0.05789 (16)	0.70098 (8)	0.0257 (2)
H2A	0.5580	−0.1878	0.7152	0.031*
C3	0.64396 (14)	0.00290 (16)	0.62119 (7)	0.0245 (2)
C4	0.64456 (16)	0.19734 (17)	0.60040 (8)	0.0291 (3)
H4A	0.6993	0.2416	0.5473	0.035*
C5	0.56388 (17)	0.32411 (17)	0.65864 (9)	0.0315 (3)
H5A	0.5619	0.4540	0.6439	0.038*
C6	0.39838 (16)	0.00556 (18)	0.84741 (8)	0.0317 (3)
C7	0.72671 (15)	−0.14509 (16)	0.56115 (8)	0.0261 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0339 (5)	0.0248 (5)	0.0261 (5)	0.0013 (4)	0.0099 (4)	−0.0030 (4)
O1	0.0562 (6)	0.0290 (5)	0.0371 (5)	0.0081 (4)	0.0188 (4)	−0.0071 (4)
O2	0.0598 (6)	0.0413 (5)	0.0384 (5)	0.0010 (4)	0.0303 (5)	−0.0032 (4)
O3	0.0616 (6)	0.0355 (5)	0.0346 (5)	−0.0033 (4)	0.0248 (4)	0.0067 (4)
O4	0.0485 (5)	0.0322 (5)	0.0288 (4)	0.0024 (4)	0.0213 (4)	−0.0002 (3)
O5	0.0585 (6)	0.0273 (5)	0.0391 (5)	0.0033 (4)	0.0216 (4)	0.0014 (4)
C1	0.0301 (5)	0.0257 (6)	0.0226 (5)	−0.0022 (4)	0.0089 (4)	0.0003 (4)
C2	0.0322 (6)	0.0227 (5)	0.0234 (5)	−0.0017 (4)	0.0085 (4)	0.0003 (4)
C3	0.0265 (5)	0.0269 (6)	0.0210 (5)	−0.0006 (4)	0.0069 (4)	−0.0007 (4)
C4	0.0349 (6)	0.0289 (6)	0.0250 (5)	−0.0037 (5)	0.0114 (4)	0.0027 (4)

C5	0.0425 (6)	0.0230 (5)	0.0305 (6)	−0.0019 (5)	0.0111 (5)	0.0033 (4)
C6	0.0372 (6)	0.0346 (6)	0.0256 (5)	−0.0040 (5)	0.0142 (5)	−0.0010 (5)
C7	0.0286 (5)	0.0275 (5)	0.0232 (5)	−0.0009 (4)	0.0080 (4)	−0.0011 (4)

Geometric parameters (Å, °)

N1—O1	1.3240 (13)	C1—C2	1.3773 (15)
N1—C5	1.3549 (15)	C1—C6	1.5076 (15)
N1—C1	1.3630 (16)	C2—C3	1.3869 (15)
O2—C6	1.2968 (15)	C2—H2A	0.9300
O2—H2B	0.8200	C3—C4	1.3890 (17)
O3—C6	1.2134 (17)	C3—C7	1.4967 (15)
O4—C7	1.3170 (14)	C4—C5	1.3750 (17)
O4—H4B	0.866 (19)	C4—H4A	0.9300
O5—C7	1.2066 (15)	C5—H5A	0.9300
O1—N1—C5	117.86 (10)	C4—C3—C7	123.24 (10)
O1—N1—C1	121.14 (9)	C5—C4—C3	119.69 (10)
C5—N1—C1	120.99 (10)	C5—C4—H4A	120.2
C6—O2—H2B	109.5	C3—C4—H4A	120.2
C7—O4—H4B	106.4 (12)	N1—C5—C4	120.65 (11)
N1—C1—C2	119.24 (10)	N1—C5—H5A	119.7
N1—C1—C6	120.07 (10)	C4—C5—H5A	119.7
C2—C1—C6	120.69 (10)	O3—C6—O2	125.01 (11)
C1—C2—C3	120.84 (10)	O3—C6—C1	118.07 (11)
C1—C2—H2A	119.6	O2—C6—C1	116.92 (11)
C3—C2—H2A	119.6	O5—C7—O4	124.64 (11)
C2—C3—C4	118.58 (10)	O5—C7—C3	122.00 (10)
C2—C3—C7	118.19 (10)	O4—C7—C3	113.35 (10)
O1—N1—C1—C2	−178.11 (10)	C1—N1—C5—C4	−1.63 (18)
C5—N1—C1—C2	0.49 (17)	C3—C4—C5—N1	1.41 (19)
O1—N1—C1—C6	1.23 (17)	N1—C1—C6—O3	−175.79 (12)
C5—N1—C1—C6	179.83 (11)	C2—C1—C6—O3	3.54 (18)
N1—C1—C2—C3	0.86 (17)	N1—C1—C6—O2	4.94 (17)
C6—C1—C2—C3	−178.47 (11)	C2—C1—C6—O2	−175.74 (11)
C1—C2—C3—C4	−1.06 (17)	C2—C3—C7—O5	−3.10 (17)
C1—C2—C3—C7	179.52 (10)	C4—C3—C7—O5	177.50 (12)
C2—C3—C4—C5	−0.07 (17)	C2—C3—C7—O4	176.24 (10)
C7—C3—C4—C5	179.32 (11)	C4—C3—C7—O4	−3.16 (16)
O1—N1—C5—C4	177.01 (11)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2B···O1	0.82	1.69	2.4508 (15)	154
O4—H4B···O3^{i}	0.866 (19)	1.80 (2)	2.6481 (14)	165.6 (18)

Symmetry codes: (i) *x*+1/2, −*y*−1/2, *z*−1/2.

Fig. 1

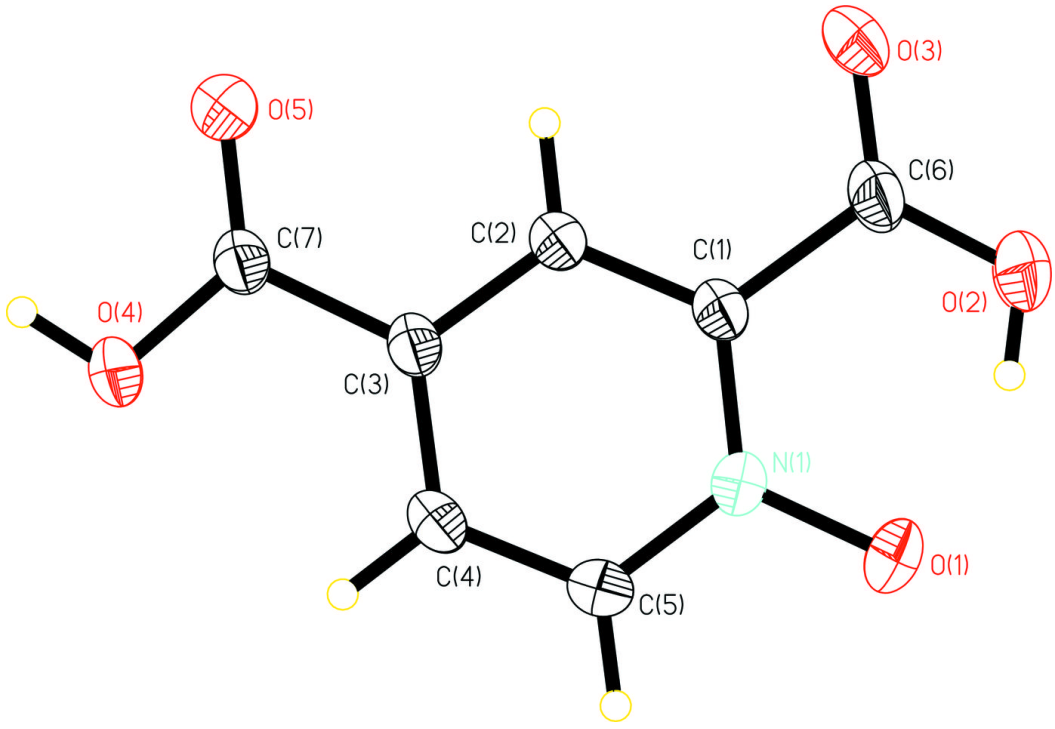

Fig. 2