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N′-Hydroxypyridine-2-carboximidamide–succinic acid (2/1)

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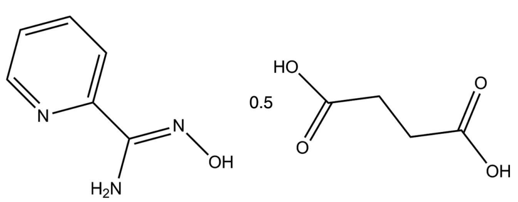
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.106; data-to-parameter ratio = 12.4.

The asymmetric unit of the title co-crystal, $C_6H_7N_3O\cdot 0.5C_4H_6O_4$, comprises one *N*′-hydroxypyridine-2-carboximidamide molecule and half a succinic acid molecule (the whole molecule is generated by inversion symmetry). In the crystal, molecules are assembled into columns along [110], *via* strong N—H⋯O, O—H⋯O and O—H⋯N hydrogen bonds.

Related literature

For background to cocrystals and their applications, see: Biradha *et al.* (2009); Desiraju (1995, 2003).



Experimental

Crystal data

$C_6H_7N_3O\cdot 0.5C_4H_6O_4$
M_r = 196.19
Monoclinic, $P2_1/c$
a = 8.6707 (8) Å
b = 5.2628 (4) Å
c = 20.6693 (15) Å
β = 93.014 (7)°
V = 941.87 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.32 × 0.28 × 0.15 mm

Data collection

Oxford Diffraction Xcalibur (Atlas, Gemini ultra) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.966, T_{max} = 0.984
4220 measured reflections
1733 independent reflections
1255 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.106
S = 1.05
1733 reflections
140 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O3—H3A⋯N3	0.84 (1)	1.80 (1)	2.6362 (18)	175 (2)
O1—H1⋯O2	0.83 (1)	1.96 (1)	2.7608 (18)	164 (2)
N2—H2B⋯O1^{i}	0.86 (1)	2.26 (1)	3.025 (2)	149 (2)

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2507).

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supporting information

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N′-Hydroxypyridine-2-carboximidamide–succinic acid (2/1)

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S1. Comment

There has been an instense interest in the preparation of cocrystals which is evident from the increasing number of research publications on this topic in recent years. With reliable strategies, cocrystals could offer a modular approach to delvelping materials with desirable properties.(Desiraju, 1995, 2003; Biradha *et al.*, 2009) Cocrystals are created by utilizing weak noncovalent interactions such as hydrogen bonds. Herein we report the structure of the first cocrystal of the pyC(NH_2)NOH molecule.

The asymmetric unit of the title compound (Fig.1) contains one pyC(NH_2)NOH molecule and one half succinic acid molecule (the entire molecule is completed by the application of a centre of inversion). The pyridine rings and the N2—C6—N3—O1 rings are nearly coplanar, and the C7—C8—C8^{ii}—C7^{ii} torsion angle [Symmetry codes: (ii)-*x*, -*y*, -*z* + 1] of succinic acid is 180° restricted by crystallographic centrosymmetry. The proton of the carboxylate O atom (O3) of the succinic acid molecule forms a strong hydrogen bond with atom N3 of the pyC(NH_2)NOH molecule, at the same time, hydrogen bonding exist between hydroxyl O1 and carboxylater O2 atoms.(see Table 1 for hydrogen bond geometry). In addition, strong intermolecular N2—H2B···O1^{i} [Symmetry codes: (i)-*x* + 1, -*y* + 2, -*z* + 1] hydrogen bonding supplement intermolecular O—H···O and O—H···N hydrogen bonding to form columns running parallel to the [110] direction. (Fig 2)

S2. Experimental

A stoichiometric amount in the ratio of 2:1 of pyC(CH_2)NOH and succinic acid were dissolved in 20 ml e thanol, and the solution slowly left to evaporate to afford colourless block-like crystals after one week.

S3. Refinement

H atoms bonded to C atoms were placed in geometrically calculated positions and were refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C). The N-bound and O-bound H atoms were located in the difference map and coordinates refined freely together with their isotropic displacement parameters.

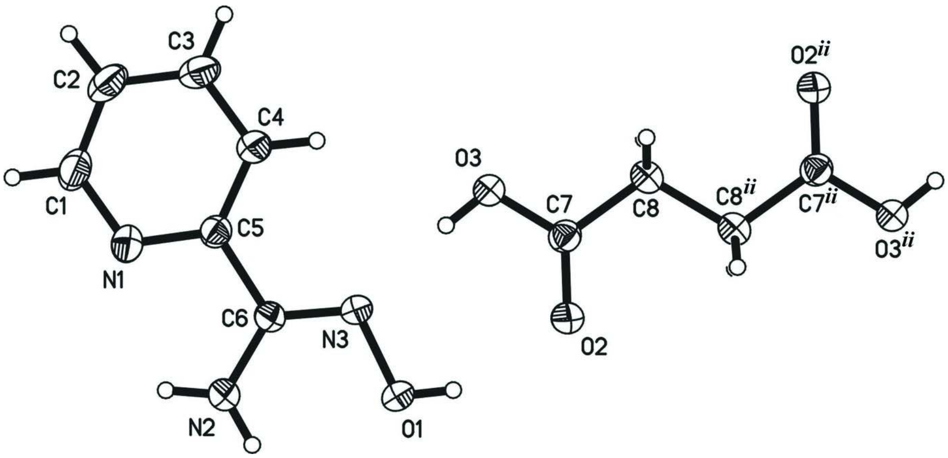

Figure 1

ORTEP view of the title compound. The displacement ellipsoids are drawn at 30% probability level. Symmetry code: (ii) -x, -y, -z+1.

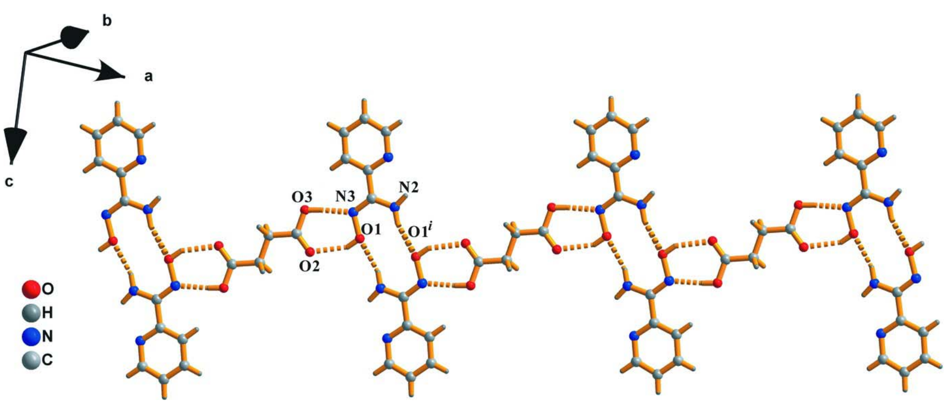

Figure 2

The one-dimensional chain of the compound along [110] direction. Symmetry code: (i) -x+1, -y+2, -z+1

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Crystal data

$C_6H_7N_3O \cdot 0.5C_4H_6O_4$
M_r = 196.19
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.6707 (8) Å
b = 5.2628 (4) Å
c = 20.6693 (15) Å
β = 93.014 (7)°
V = 941.87 (13) Å^3
Z = 4
$F(000)$ = 412
D_x = 1.384 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1508 reflections
θ = 3.0–29.6°
μ = 0.11 mm^{-1}
T = 293 K
Block, colourless
0.32 × 0.28 × 0.15 mm

Data collection

Oxford Diffraction Xcalibur (Atlas, Gemini ultra) diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.3592 pixels mm^{-1}
ω scans

Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.966, T_{max} = 0.984
4220 measured reflections
1733 independent reflections
1255 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.4°, θ_{min} = 3.0°
$h = -10 \rightarrow 10$
$k = -5 \rightarrow 6$
$l = -24 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.106$
S = 1.05
1733 reflections
140 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.0833P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.106 (6)

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 $F^2 > \sigma(F^2)$ 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}
O1	0.39704 (16)	0.7215 (3)	0.47132 (6)	0.0554 (4)
H1	0.352 (3)	0.597 (3)	0.4861 (11)	0.083*
O2	0.19671 (16)	0.3449 (3)	0.50695 (6)	0.0595 (4)
O3	0.11775 (15)	0.3628 (3)	0.40307 (6)	0.0527 (4)
H3A	0.182 (2)	0.482 (3)	0.4024 (11)	0.079*
N1	0.36547 (19)	1.1544 (3)	0.27768 (7)	0.0536 (5)
N2	0.4806 (2)	1.0874 (3)	0.39785 (8)	0.0546 (5)
H2A	0.497 (2)	1.209 (3)	0.3713 (8)	0.066*
H2B	0.517 (2)	1.079 (4)	0.4370 (6)	0.066*
N3	0.32452 (17)	0.7305 (3)	0.40829 (6)	0.0429 (4)
C1	0.3146 (3)	1.1925 (4)	0.21617 (10)	0.0630 (6)
H1A	0.3535	1.3306	0.1942	0.076*
C2	0.2086 (3)	1.0397 (4)	0.18374 (10)	0.0623 (6)
H2	0.1772	1.0720	0.1408	0.075*
C3	0.1503 (3)	0.8383 (4)	0.21632 (10)	0.0691 (7)
H3	0.0768	0.7322	0.1961	0.083*
C4	0.2017 (3)	0.7947 (4)	0.27915 (10)	0.0589 (6)
H4	0.1639	0.6578	0.3020	0.071*

C5	0.30971 (19)	0.9554 (3)	0.30816 (8)	0.0394 (4)
C6	0.37353 (19)	0.9207 (3)	0.37593 (8)	0.0375 (4)
C7	0.1144 (2)	0.2691 (3)	0.46188 (8)	0.0404 (4)
C8	−0.0020 (2)	0.0614 (3)	0.46712 (8)	0.0450 (5)
H8A	0.0166	−0.0675	0.4349	0.054*
H8B	−0.1043	0.1305	0.4575	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0678 (9)	0.0588 (9)	0.0376 (7)	−0.0209 (7)	−0.0147 (6)	0.0097 (6)
O2	0.0687 (9)	0.0703 (9)	0.0387 (7)	−0.0310 (8)	−0.0055 (7)	0.0058 (7)
O3	0.0573 (9)	0.0596 (9)	0.0402 (7)	−0.0211 (7)	−0.0069 (6)	0.0095 (7)
N1	0.0545 (10)	0.0596 (10)	0.0466 (10)	−0.0103 (8)	0.0023 (8)	0.0131 (8)
N2	0.0664 (11)	0.0567 (11)	0.0400 (9)	−0.0249 (9)	−0.0048 (8)	0.0050 (8)
N3	0.0482 (9)	0.0450 (9)	0.0347 (8)	−0.0081 (7)	−0.0070 (6)	0.0036 (7)
C1	0.0607 (13)	0.0774 (15)	0.0508 (13)	−0.0053 (12)	0.0014 (10)	0.0252 (11)
C2	0.0659 (14)	0.0788 (15)	0.0410 (11)	0.0078 (12)	−0.0075 (10)	0.0142 (11)
C3	0.0829 (16)	0.0678 (14)	0.0534 (13)	−0.0107 (12)	−0.0279 (12)	0.0085 (11)
C4	0.0737 (14)	0.0512 (12)	0.0496 (12)	−0.0162 (10)	−0.0176 (10)	0.0112 (10)
C5	0.0412 (10)	0.0391 (10)	0.0378 (10)	0.0007 (8)	0.0011 (8)	0.0016 (8)
C6	0.0390 (10)	0.0371 (9)	0.0365 (9)	−0.0042 (8)	0.0016 (7)	−0.0013 (8)
C7	0.0411 (10)	0.0433 (10)	0.0370 (10)	0.0000 (8)	0.0024 (8)	0.0003 (8)
C8	0.0432 (10)	0.0467 (11)	0.0448 (10)	−0.0094 (8)	0.0002 (8)	0.0020 (8)

Geometric parameters (Å, º)

O1—N3	1.4173 (17)	C1—H1A	0.9300
O1—H1	0.828 (10)	C2—C3	1.367 (3)
O2—C7	1.211 (2)	C2—H2	0.9300
O3—C7	1.314 (2)	C3—C4	1.370 (3)
O3—H3A	0.840 (10)	C3—H3	0.9300
N1—C5	1.327 (2)	C4—C5	1.376 (3)
N1—C1	1.339 (2)	C4—H4	0.9300
N2—C6	1.339 (2)	C5—C6	1.490 (2)
N2—H2A	0.859 (9)	C7—C8	1.495 (2)
N2—H2B	0.856 (9)	C8—C8^{i}	1.503 (3)
N3—C6	1.288 (2)	C8—H8A	0.9700
C1—C2	1.370 (3)	C8—H8B	0.9700
N3—O1—H1	99.8 (16)	C3—C4—H4	120.3
C7—O3—H3A	110.1 (16)	C5—C4—H4	120.3
C5—N1—C1	117.31 (17)	N1—C5—C4	122.36 (16)
C6—N2—H2A	114.0 (14)	N1—C5—C6	114.61 (15)
C6—N2—H2B	120.2 (14)	C4—C5—C6	123.04 (16)
H2A—N2—H2B	125 (2)	N3—C6—N2	125.12 (16)
C6—N3—O1	111.14 (13)	N3—C6—C5	117.83 (14)
N1—C1—C2	123.79 (19)	N2—C6—C5	117.03 (15)

N1—C1—H1A	118.1	O2—C7—O3	123.17 (16)
C2—C1—H1A	118.1	O2—C7—C8	123.90 (16)
C3—C2—C1	118.03 (18)	O3—C7—C8	112.93 (15)
C3—C2—H2	121.0	C7—C8—C8^{i}	113.34 (18)
C1—C2—H2	121.0	C7—C8—H8A	108.9
C2—C3—C4	119.1 (2)	C8^{i}—C8—H8A	108.9
C2—C3—H3	120.4	C7—C8—H8B	108.9
C4—C3—H3	120.4	C8^{i}—C8—H8B	108.9
C3—C4—C5	119.35 (19)	H8A—C8—H8B	107.7

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···N3	0.84 (1)	1.80 (1)	2.6362 (18)	175 (2)
O1—H1···O2	0.83 (1)	1.96 (1)	2.7608 (18)	164 (2)
N2—H2*B*···O1^{ii}	0.86 (1)	2.26 (1)	3.025 (2)	149 (2)

Symmetry code: (ii) −*x*+1, −*y*+2, −*z*+1.