organic compounds

3-(2-Pyridylaminocarbonyl)propanoic acid

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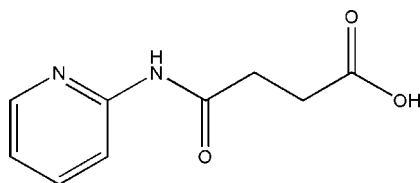Correspondence e-mail: wang2009chengfeng@126.com

In the crystal structure of the title compound, $C_9H_{10}N_2O_3$, the molecules are linked by intermolecular O—H···N and N—H···O hydrogen bonds, resulting in chains propagating in [010]. Weak intramolecular and intermolecular C—H···O interactions are also observed.

Related literature

For background on the pharmaceutical applications of this family of compounds, see: Narendar *et al.* (2003); Ravlee *et al.* (2003).

Experimental

Crystal data

$C_9H_{10}N_2O_3$	a = 12.7384 (10) Å
M_r = 194.19	b = 5.0485 (5) Å
Monoclinic, $P2_1/n$	c = 13.8463 (12) Å
β = 92.924 (8)°	μ = 0.11 mm^{-1}
V = 889.29 (14) Å^3	T = 113 K
Z = 4	$0.22 \times 0.04 \times 0.03$ mm
Mo $K\alpha$ radiation	

Data collection

Rigaku Saturn diffractometer	8079 measured reflections
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)	1972 independent reflections
T_{min} = 0.995, T_{max} = 0.996	1297 reflections with $I > 2\sigma(I)$
	R_{int} = 0.074

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.104	
S = 0.97	
1972 reflections	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
135 parameters	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
1 restraint	

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2^i	0.954 (19)	1.744 (19)	2.690 (2)	170.4 (17)
N1—H1A···O2^{ii}	0.96 (2)	1.86 (2)	2.824 (2)	176.6 (19)
C6—H6···O3	0.95	2.31	2.893 (2)	119
C3—H3A···O2^{ii}	0.99	2.60	3.445 (2)	143
C3—H3B···O3^{iii}	0.99	2.58	3.408 (2)	141
C7—H7···O3^{iv}	0.95	2.56	3.319 (2)	137

Symmetry codes: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x, y+1, z$; (iv) $-x, -y, -z+1$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *CrystalStructure* (Rigaku, 2005).

References

Narendar, P., Parthiban, J. & Anbalagan, N. (2003). *Biol. Pharm. Bull.* **26**, 182–187.
Ravlee, I., Sivakumar, R. & Muruganantham, N. (2003). *Chem. Pharm. Bull.* **51**, 162–170.
Rigaku (2005). *CrystalClear* and *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

3-(2-Pyridylaminocarbonyl)propanoic acid

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S1. Comment

Pyridine derivatives substituted by *N*-alkylation show useful pharmaceutical properties (Narendar *et al.*, 2003; Ravlee *et al.*, 2003). In this paper, the structure of 4-oxo-4-(pyridin-2-ylamino)butanoic acid (I), is reported which was synthesized by acylating reation of pyridin-2-amine with pyrrolidine-2,5-dione. The pyridin ring system is essentially planar with mean deviations of 0.0013 Å. In addition, there are C—H⋯O interactions, as shown in Fig. 2 and detailed in Table 1.

S2. Experimental

A solution of pyrrolidine-2,5-dione (1.0 g,10 mmol) in dimethylformamide (15 ml) was stirred at room temperature for 10 min. Pyridin-2-amine (0.94 g, 10 mmol) was added and the mixture was stirred for a further 3 h at 353 K. The resulting mixture was then poured into water (100 ml), yielding a white precipitate. The solid product was filtered off, washed with cold water and recrystallized from methnol, giving crystals of the title compound [yield: 1.17 g (61.4%)]. These were dissolved in mixture of methnol (10 ml) and water (3 ml) and the solution was kept at room temperature for 18 d. Natural evaporation of the solution gave colourless prisms of (I) (m.p. 454–455 K).

S3. Refinement

The O- and N-bound H atoms were located in a difference map and their positions and U_{iso} values were freely refined. The C-bound H atoms were geometrically placed (C—H = 0.95–0.99Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.
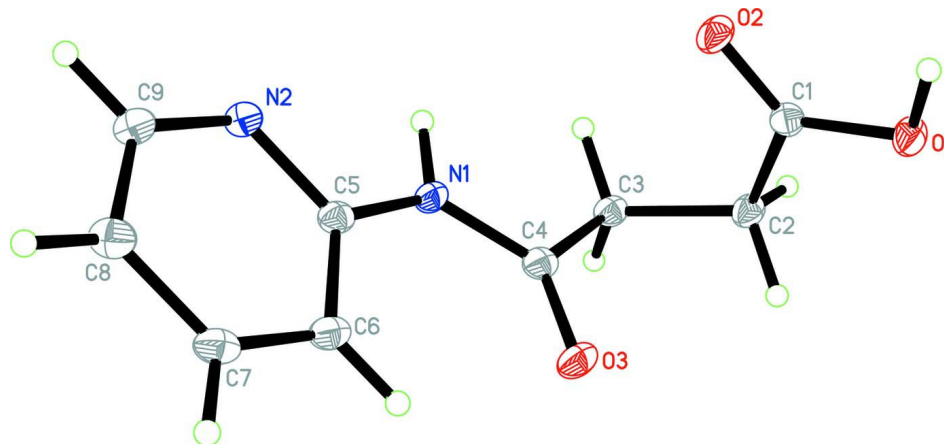

Figure 1

A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level (arbitrary spheres for H atoms).
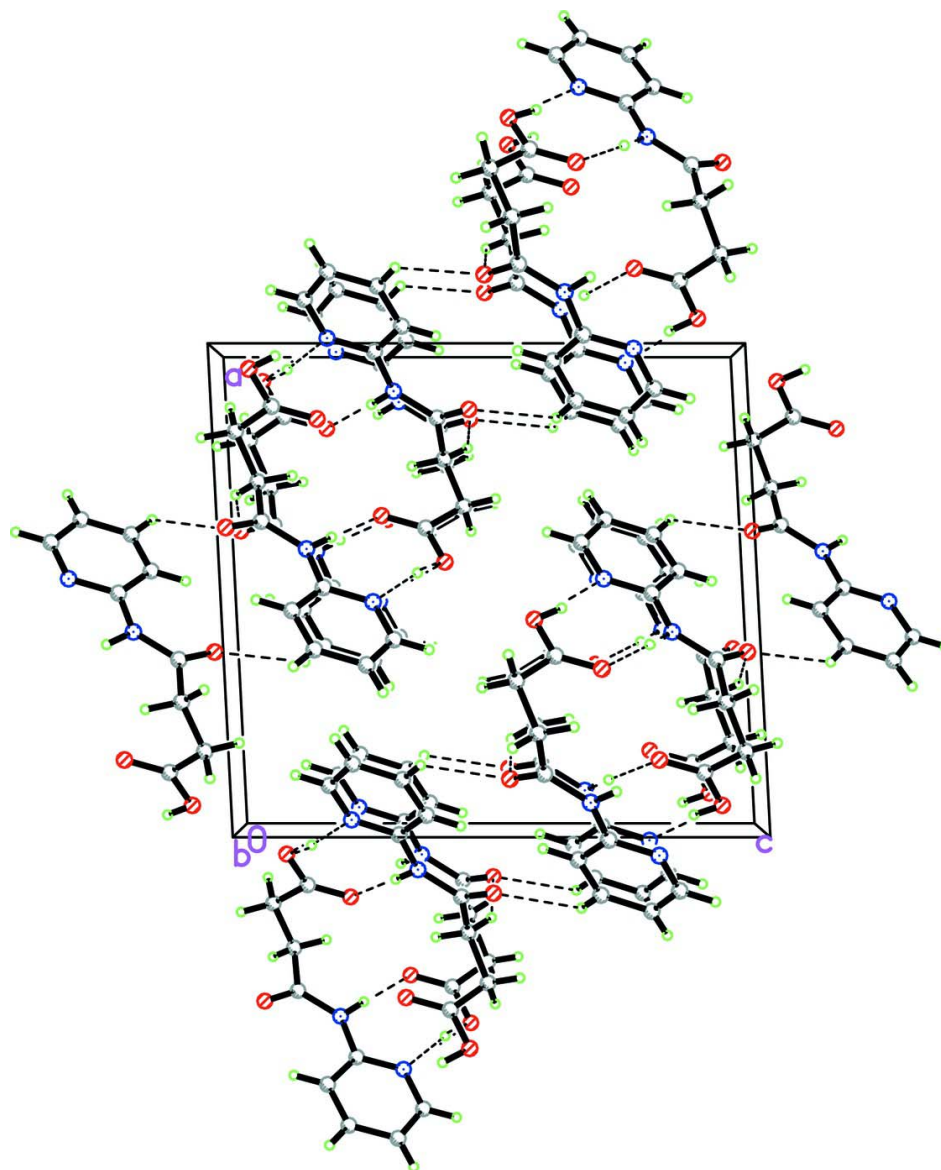
Figure 2

Part of the crystal structure of (I), with hydrogen bonds shown as dashed lines.

3-(2-Pyridylaminocarbonyl)propanoic acid

Crystal data

$C_9H_{10}N_2O_3$	$F(000) = 408$
$M_r = 194.19$	$D_x = 1.450$ Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point = 454–455 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
$a = 12.7384$ (10) Å	Cell parameters from 2970 reflections
$b = 5.0485$ (5) Å	$\theta = 2.1–27.2°$
$c = 13.8463$ (12) Å	$\mu = 0.11$ mm^{-1}
$\beta = 92.924$ (8)°	$T = 113$ K
$V = 889.29$ (14) Å^3	Prism, colourless
$Z = 4$	$0.22 \times 0.04 \times 0.03$ mm

Data collection

Rigaku Saturn diffractometer	8079 measured reflections
Radiation source: rotating anode	1972 independent reflections
Confocal monochromator	1297 reflections with $I > 2\sigma(I)$
Detector resolution: 14.63 pixels mm^{-1}	$R_{\text{int}} = 0.074$
ω and φ scans	$\theta_{\max} = 27.2°$, $\theta_{\min} = 2.1°$
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)	$h = -16\rightarrow16$
	$k = -6\rightarrow6$
$T_{\min} = 0.995$, $T_{\max} = 0.996$	$l = -17\rightarrow17$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.048$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.104$	
$S = 0.97$	$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2]$
1972 reflections	where $P = (F_o^2 + 2F_c^2)/3$
135 parameters	$(\Delta/\sigma)_{\max} = 0.001$
1 restraint	$\Delta\rho_{\max} = 0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.22$ e Å^{-3}
	Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.041 (3)

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	0.44178 (10)	0.2594 (3)	0.58044 (9)	0.0272 (4)
H1	0.4642 (15)	0.145 (4)	0.6323 (13)	0.041*
O2	0.34511 (10)	0.4177 (3)	0.69759 (9)	0.0290 (4)
O3	0.12611 (10)	0.3483 (3)	0.52083 (9)	0.0284 (4)
N1	0.08391 (12)	0.5393 (3)	0.66376 (11)	0.0216 (4)
N2	−0.02347 (12)	0.4254 (3)	0.78432 (11)	0.0231 (4)
C1	0.37059 (14)	0.4218 (4)	0.61433 (13)	0.0221 (4)
C2	0.32399 (15)	0.6117 (4)	0.54040 (13)	0.0239 (5)
H2A	0.3090	0.5171	0.4786	0.029*
H2B	0.3753	0.7541	0.5288	0.029*
C3	0.22283 (14)	0.7334 (4)	0.57436 (13)	0.0227 (5)
H3A	0.2367	0.8194	0.6380	0.027*
H3B	0.1972	0.8703	0.5277	0.027*
C4	0.14010 (14)	0.5211 (4)	0.58271 (13)	0.0223 (5)

C5	0.00578 (14)	0.3716 (4)	0.69450 (13)	0.0214 (5)
C6	−0.04012 (15)	0.1661 (4)	0.63919 (13)	0.0244 (5)
H6	−0.0190	0.1317	0.5756	0.029*
C7	−0.11649 (15)	0.0153 (4)	0.67911 (14)	0.0265 (5)
H7	−0.1484	−0.1261	0.6431	0.032*
C8	−0.14707 (15)	0.0686 (4)	0.77148 (13)	0.0276 (5)
H8	−0.1999	−0.0338	0.7999	0.033*
C9	−0.09839 (15)	0.2750 (4)	0.82096 (13)	0.0256 (5)
H9	−0.1190	0.3126	0.8845	0.031*
H1A	0.1087 (16)	0.673 (4)	0.7090 (14)	0.043 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0313 (8)	0.0289 (8)	0.0218 (8)	0.0121 (7)	0.0044 (6)	0.0043 (6)
O2	0.0352 (8)	0.0350 (9)	0.0172 (7)	0.0102 (7)	0.0057 (6)	0.0054 (6)
O3	0.0363 (9)	0.0269 (8)	0.0221 (8)	0.0033 (6)	0.0026 (6)	−0.0067 (6)
N1	0.0237 (9)	0.0228 (10)	0.0185 (8)	−0.0006 (7)	0.0019 (7)	−0.0045 (7)
N2	0.0259 (9)	0.0232 (9)	0.0202 (8)	−0.0010 (8)	0.0006 (7)	−0.0007 (7)
C1	0.0223 (10)	0.0217 (11)	0.0222 (10)	−0.0005 (9)	0.0007 (8)	−0.0010 (9)
C2	0.0282 (11)	0.0246 (11)	0.0191 (10)	0.0051 (9)	0.0028 (8)	0.0020 (8)
C3	0.0274 (11)	0.0213 (11)	0.0191 (10)	0.0043 (9)	0.0000 (8)	0.0009 (8)
C4	0.0257 (11)	0.0208 (11)	0.0200 (10)	0.0074 (9)	−0.0031 (8)	0.0008 (8)
C5	0.0234 (11)	0.0214 (11)	0.0191 (10)	0.0037 (9)	−0.0021 (8)	0.0005 (8)
C6	0.0283 (12)	0.0237 (11)	0.0207 (10)	0.0042 (9)	−0.0033 (8)	−0.0034 (8)
C7	0.0299 (11)	0.0235 (12)	0.0252 (11)	0.0008 (9)	−0.0063 (9)	−0.0041 (9)
C8	0.0320 (12)	0.0238 (11)	0.0267 (11)	−0.0057 (9)	−0.0023 (9)	0.0030 (9)
C9	0.0311 (12)	0.0253 (12)	0.0203 (10)	−0.0022 (9)	0.0008 (8)	−0.0008 (9)

Geometric parameters (Å, º)

O1—C1	1.326 (2)	C2—H2B	0.9900
O1—H1	0.954 (17)	C3—C4	1.512 (3)
O2—C1	1.214 (2)	C3—H3A	0.9900
O3—C4	1.229 (2)	C3—H3B	0.9900
N1—C4	1.365 (2)	C5—C6	1.400 (2)
N1—C5	1.390 (2)	C6—C7	1.373 (3)
N1—H1A	0.96 (2)	C6—H6	0.9500
N2—C9	1.340 (2)	C7—C8	1.382 (2)
N2—C5	1.344 (2)	C7—H7	0.9500
C1—C2	1.503 (2)	C8—C9	1.377 (2)
C2—C3	1.523 (2)	C8—H8	0.9500
C2—H2A	0.9900	C9—H9	0.9500
C1—O1—H1	107.1 (12)	H3A—C3—H3B	108.2
C4—N1—C5	128.65 (17)	O3—C4—N1	123.90 (19)
C4—N1—H1A	114.5 (12)	O3—C4—C3	121.78 (18)
C5—N1—H1A	116.3 (12)	N1—C4—C3	114.33 (16)

C9—N2—C5	118.17 (16)	N2—C5—N1	113.37 (16)
O2—C1—O1	123.11 (17)	N2—C5—C6	121.90 (18)
O2—C1—C2	122.87 (17)	N1—C5—C6	124.73 (17)
O1—C1—C2	114.02 (16)	C7—C6—C5	118.38 (18)
C1—C2—C3	110.98 (16)	C7—C6—H6	120.8
C1—C2—H2A	109.4	C5—C6—H6	120.8
C3—C2—H2A	109.4	C6—C7—C8	120.21 (18)
C1—C2—H2B	109.4	C6—C7—H7	119.9
C3—C2—H2B	109.4	C8—C7—H7	119.9
H2A—C2—H2B	108.0	C9—C8—C7	117.82 (18)
C4—C3—C2	109.99 (15)	C9—C8—H8	121.1
C4—C3—H3A	109.7	C7—C8—H8	121.1
C2—C3—H3A	109.7	N2—C9—C8	123.51 (18)
C4—C3—H3B	109.7	N2—C9—H9	118.2
C2—C3—H3B	109.7	C8—C9—H9	118.2
O2—C1—C2—C3	−16.5 (3)	C4—N1—C5—N2	171.25 (17)
O1—C1—C2—C3	163.40 (16)	C4—N1—C5—C6	−9.1 (3)
C1—C2—C3—C4	−64.77 (19)	N2—C5—C6—C7	−0.6 (3)
C5—N1—C4—O3	2.5 (3)	N1—C5—C6—C7	179.81 (16)
C5—N1—C4—C3	−177.43 (15)	C5—C6—C7—C8	0.5 (3)
C2—C3—C4—O3	−42.8 (2)	C6—C7—C8—C9	−0.2 (3)
C2—C3—C4—N1	137.14 (15)	C5—N2—C9—C8	−0.1 (3)
C9—N2—C5—N1	−179.95 (15)	C7—C8—C9—N2	0.0 (3)
C9—N2—C5—C6	0.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2[i]	0.95 (2)	1.74 (2)	2.690 (2)	170 (2)
N1—H1A···O2[ii]	0.96 (2)	1.86 (2)	2.824 (2)	176.6 (19)
C6—H6···O3	0.95	2.31	2.893 (2)	119
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