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(*E*)-4-(Nicotinoylhydrazono)pentanoic acid

Yong-Tao Wang,[a]* Gui-Mei Tang,[a] Jian-Chao Yu,[a] Wen-Tao Yu[b] and Jian-Dong Fan[b]

[a]Department of Chemical Engineering, Shandong Institute of Light Industry, Jinan, Shandong 250353, People's Republic of China, and [b]Institute of Crystalline Materials, Shandong University, Jinan, Shandong 250100, People's Republic of China
Correspondence e-mail: ceswyt@sohu.com

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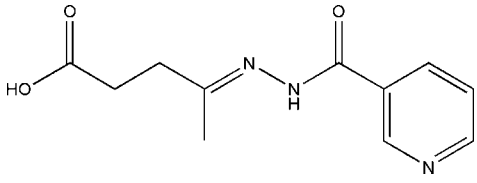
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.118; data-to-parameter ratio = 10.6.

The molecules of the title compound, $C_{11}H_{13}N_3O_3$, are linked by O—H···N and N—H···O interactions into a linear hydrogen-bonded chain.

Related literature

For the biological activity of hydrazones, see: Maccari *et al.* (2005); Vicini *et al.* (2002). For the use of hydrazones as intermediates in synthesis, see Rollas *et al.* (2002). For the synthesis of the title compound, see Wang & Tang (2006).



Experimental

Crystal data

$C_{11}H_{13}N_3O_3$
M_r = 235.24
Triclinic, $P\overline{1}$
a = 7.0290 (3) Å
b = 7.0968 (2) Å
c = 12.1706 (4) Å
α = 85.419 (2)°
β = 75.096 (2)°
γ = 84.064 (2)°
V = 582.64 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 (2) K
0.40 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
5604 measured reflections
2185 independent reflections
1626 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.118
S = 1.05
2185 reflections
206 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N3^{i}	0.96 (2)	1.75 (2)	2.689 (2)	165 (3)
N2—H2···O3^{ii}	0.87 (2)	2.11 (2)	2.959 (2)	167 (1)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2363).

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supplementary materials

Acta Cryst. (2007). E**63**, o4655 [**doi:10.1107/S1600536807056449**]

(*E*)-4-(Nicotinoylhydrazono)pentanoic acid

Y.-T. Wang, G.-M. Tang, J.-C. Yu, W.-T. Yu and J.-D. Fan

Comment

Hydrazone and its derivatives exhibit biological and pharmacological activities (Vicini *et al.*, 2002; Maccari *et al.*, 2005). These compounds are also used as synthetic intermediates (Rollas *et al.*, 2002). The title compound− a monohydrate of (*E*)-4-(nicotinoylaminoimino)pentanoic acid (napa) - was synthesized in our laboratory by the reaction of acetopropanic acid and nicotinohydrazide in ethanol medium. Here we present its crystal structure (Fig. 1).

The bond lengths (Table 1) and angles of the napa molecule show normal values. Intermolecular O—H···N and N—H···O hydrogen bonds play an important role in the formation of three-dimensional network structure (Table 2). In addition, C—H···O weak intermolecular interaction furthure stabilize the crystal packing (Fig. 2).

Experimental

The title compound was synthesized according to the literature (Wang and Tang, 2006). The title compound was recrystallized from the mixed solvent of methanol and water. Block colorless single crystals suitable for *x*-ray diffraction were obtained. Analysis found (%): C, 56.46; H, 5.55; N, 17.79; requires (%): C, 56.16; H, 5.57; N, 17.86; IR(KBr, cm^{-1}): 3436 (w), 3391(w), 3294(w), 3063(w), 3019(w), 2941(w), 2913(w), 2806(w), 2663(w), 2534(w), 1943(br), 1718(*s*), 1633(*m*), 1594(*m*), 1540(*versus*), 1476(w), 1418(*s*), 1376(*m*), 1321(*s*), 1282(*s*), 1232(*s*), 1187(*s*), 1143(*m*), 1097(w), 1055(w), 1027(*s*), 960(w), 933(*s*), 910(w), 831(*versus*), 770(w), 733(*m*), 701(*s*), 672(*m*), 640(*m*), 617(w), 528(w), 490(w), 419(w).

Refinement

All H atoms were initially located in a difference Fourier map. The positions of the H atoms for water and carboxylate were refined freely along with an isotropic displacement parameter. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with N—H and C—H distances in the range 0.86–0.97Å and U_{iso}(H) = 1.2U_{eq}(C),respectively.

Figures

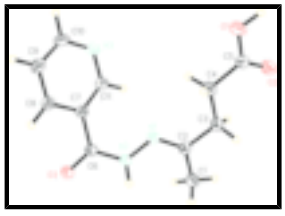

Fig. 1. View of the (I) showing displacement ellipsoids at the 30% probability level. H atoms are represented by circles of arbitrary size.

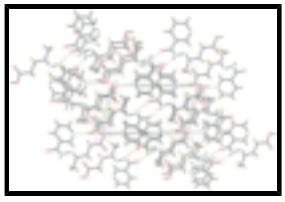

Fig. 2. Packing diagram showing hydrogen bonds as dashed lines.

(*E*)-4-(Nicotinoylhydrazono)pentanoic acid

Crystal data

$C_{11}H_{13}N_3O_3$	$Z = 2$
$M_r = 235.24$	$F_{000} = 248$
Triclinic, $P\overline{1}$	$D_x = 1.341$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.0290$ (3) Å	Cell parameters from 2322 reflections
$b = 7.0968$ (2) Å	$\theta = 2.9$–27.1°
$c = 12.1706$ (4) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 85.419$ (2)°	$T = 295$ (2) K
$\beta = 75.096$ (2)°	Plate, colourless
$\gamma = 84.064$ (2)°	0.40 × 0.20 × 0.20 mm
$V = 582.64$ (4) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	1626 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.018$
Monochromator: graphite	$\theta_{max} = 26.0^\circ$
$T = 295(2)$ K	$\theta_{min} = 1.7^\circ$
φ and ω scans	$h = -5 \rightarrow 8$
Absorption correction: none	$k = -8 \rightarrow 8$
5604 measured reflections	$l = -14 \rightarrow 14$
2185 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
$R[F^2 > 2\sigma(F^2)] = 0.038$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.118$	$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.1419P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2185 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}

206 parameters
6 restraints
Primary atom site location: structure-invariant direct methods

$\Delta\rho_{min} = -0.14$ e Å^{-3}
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	0.3679 (2)	0.0659 (2)	0.26413 (12)	0.0395 (4)
N2	0.4224 (2)	−0.0185 (2)	0.36022 (13)	0.0411 (4)
N3	−0.0020 (2)	−0.3318 (2)	0.26439 (14)	0.0491 (4)
O1	0.0342 (2)	0.2835 (2)	−0.04831 (13)	0.0618 (4)
O2	0.3513 (2)	0.3254 (2)	−0.12117 (12)	0.0655 (5)
O3	0.34167 (19)	−0.1862 (2)	0.52647 (11)	0.0500 (4)
C1	0.7050 (3)	0.1727 (4)	0.20292 (19)	0.0608 (6)
C2	0.4995 (3)	0.1553 (3)	0.19311 (15)	0.0406 (4)
C3	0.4487 (3)	0.2466 (4)	0.08767 (18)	0.0520 (5)
C4	0.2433 (3)	0.2259 (4)	0.07626 (18)	0.0507 (5)
C5	0.2189 (3)	0.2841 (3)	−0.04111 (16)	0.0451 (5)
C6	0.3087 (2)	−0.1382 (2)	0.43323 (14)	0.0370 (4)
C7	0.1418 (2)	−0.2157 (2)	0.40016 (14)	0.0363 (4)
C8	−0.0256 (3)	−0.2502 (2)	0.48646 (16)	0.0402 (4)
C9	−0.1819 (3)	−0.3223 (3)	0.45892 (18)	0.0466 (5)
C10	−0.1651 (3)	−0.3590 (3)	0.34801 (19)	0.0500 (5)
C11	0.1486 (3)	−0.2621 (3)	0.29097 (16)	0.0429 (4)
H8	−0.030 (3)	−0.219 (3)	0.5665 (17)	0.049 (5)*
H3B	0.468 (3)	0.385 (4)	0.0853 (19)	0.073 (7)*
H2	0.508 (3)	0.031 (3)	0.3871 (18)	0.056 (6)*
H11	0.266 (3)	−0.243 (3)	0.2277 (18)	0.054 (6)*
H10	−0.275 (3)	−0.408 (3)	0.3253 (19)	0.068 (6)*
H4B	0.214 (4)	0.095 (4)	0.091 (2)	0.075 (7)*
H3A	0.548 (4)	0.197 (3)	0.022 (2)	0.077 (8)*
H9	−0.296 (3)	−0.348 (3)	0.5191 (18)	0.062 (6)*
H4A	0.142 (4)	0.303 (4)	0.129 (2)	0.082 (8)*
H1	0.029 (4)	0.322 (4)	−0.125 (2)	0.097 (9)*
H1A	0.786 (3)	0.213 (3)	0.1317 (12)	0.109 (10)*
H1B	0.760 (4)	0.054 (2)	0.230 (2)	0.127 (12)*

H1C	0.699 (5)	0.264 (3)	0.2583 (17)	0.152 (15)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0393 (8)	0.0467 (8)	0.0364 (8)	−0.0118 (7)	−0.0162 (6)	0.0060 (6)
N2	0.0386 (8)	0.0520 (9)	0.0383 (8)	−0.0154 (7)	−0.0181 (7)	0.0075 (7)
N3	0.0527 (10)	0.0546 (10)	0.0487 (9)	−0.0162 (8)	−0.0247 (8)	−0.0002 (8)
O1	0.0508 (9)	0.0949 (12)	0.0462 (9)	−0.0187 (8)	−0.0224 (7)	0.0071 (8)
O2	0.0564 (9)	0.1015 (13)	0.0425 (8)	−0.0249 (9)	−0.0173 (7)	0.0115 (8)
O3	0.0500 (8)	0.0653 (9)	0.0428 (7)	−0.0215 (7)	−0.0250 (6)	0.0137 (6)
C1	0.0450 (12)	0.0929 (18)	0.0477 (12)	−0.0267 (12)	−0.0153 (10)	0.0161 (12)
C2	0.0387 (10)	0.0496 (10)	0.0361 (9)	−0.0141 (8)	−0.0114 (7)	0.0019 (8)
C3	0.0511 (13)	0.0656 (14)	0.0441 (11)	−0.0229 (11)	−0.0188 (10)	0.0148 (10)
C4	0.0475 (12)	0.0654 (14)	0.0428 (11)	−0.0137 (11)	−0.0182 (9)	0.0093 (10)
C5	0.0500 (12)	0.0496 (11)	0.0409 (10)	−0.0132 (9)	−0.0181 (9)	0.0006 (8)
C6	0.0331 (9)	0.0432 (9)	0.0376 (9)	−0.0062 (7)	−0.0141 (7)	0.0027 (8)
C7	0.0344 (9)	0.0374 (9)	0.0400 (9)	−0.0057 (7)	−0.0152 (7)	0.0032 (7)
C8	0.0380 (10)	0.0402 (10)	0.0438 (10)	−0.0047 (8)	−0.0136 (8)	0.0015 (8)
C9	0.0354 (11)	0.0476 (11)	0.0572 (12)	−0.0077 (8)	−0.0132 (9)	0.0051 (9)
C10	0.0443 (11)	0.0495 (11)	0.0654 (13)	−0.0138 (9)	−0.0283 (10)	0.0025 (10)
C11	0.0438 (11)	0.0479 (11)	0.0409 (10)	−0.0116 (9)	−0.0162 (8)	0.0013 (8)

Geometric parameters (Å, °)

N1—C2	1.276 (2)	C3—C4	1.508 (3)
N1—N2	1.394 (2)	C3—H3B	1.00 (2)
N2—C6	1.343 (2)	C3—H3A	0.98 (3)
N2—H2	0.87 (2)	C4—C5	1.503 (3)
N3—C11	1.334 (2)	C4—H4B	0.97 (3)
N3—C10	1.342 (3)	C4—H4A	0.98 (3)
O1—C5	1.324 (2)	C6—C7	1.501 (2)
O1—H1	0.96 (3)	C7—C11	1.382 (2)
O2—C5	1.203 (2)	C7—C8	1.389 (2)
O3—C6	1.231 (2)	C8—C9	1.384 (3)
C1—C2	1.498 (3)	C8—H8	1.01 (2)
C1—H1A	0.949 (9)	C9—C10	1.368 (3)
C1—H1B	0.960 (19)	C9—H9	0.96 (2)
C1—H1C	0.96 (2)	C10—H10	0.99 (2)
C2—C3	1.500 (3)	C11—H11	0.98 (2)
C2—N1—N2	115.85 (14)	C5—C4—H4A	106.2 (15)
C6—N2—N1	121.66 (14)	C3—C4—H4A	112.7 (16)
C6—N2—H2	115.2 (14)	H4B—C4—H4A	108 (2)
N1—N2—H2	120.2 (14)	O2—C5—O1	122.75 (17)
C11—N3—C10	118.06 (17)	O2—C5—C4	124.49 (18)
C5—O1—H1	108.3 (16)	O1—C5—C4	112.76 (17)
C2—C1—H1A	110.5 (17)	O3—C6—N2	120.49 (15)
C2—C1—H1B	110.4 (18)	O3—C6—C7	119.81 (15)

H1A—C1—H1B	110.0 (13)	N2—C6—C7	119.70 (15)
C2—C1—H1C	109 (2)	C11—C7—C8	118.60 (16)
H1A—C1—H1C	109.6 (13)	C11—C7—C6	124.06 (16)
H1B—C1—H1C	107.5 (13)	C8—C7—C6	117.27 (16)
N1—C2—C1	125.97 (17)	C9—C8—C7	118.74 (18)
N1—C2—C3	117.82 (16)	C9—C8—H8	122.5 (11)
C1—C2—C3	116.18 (16)	C7—C8—H8	118.8 (11)
C2—C3—C4	116.36 (16)	C10—C9—C8	118.81 (19)
C2—C3—H3B	107.5 (13)	C10—C9—H9	123.1 (13)
C4—C3—H3B	109.0 (13)	C8—C9—H9	118.1 (13)
C2—C3—H3A	107.9 (15)	N3—C10—C9	123.09 (17)
C4—C3—H3A	110.4 (15)	N3—C10—H10	116.1 (13)
H3B—C3—H3A	105 (2)	C9—C10—H10	120.8 (13)
C5—C4—C3	112.72 (17)	N3—C11—C7	122.64 (18)
C5—C4—H4B	106.5 (14)	N3—C11—H11	116.2 (12)
C3—C4—H4B	110.2 (15)	C7—C11—H11	121.2 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3^{i}	0.96 (2)	1.75 (2)	2.689 (2)	165 (3)
N2—H2···O3^{ii}	0.87 (2)	2.11 (2)	2.959 (2)	167 (1)
C1—H1A···O1^{iii}	0.95 (2)	2.48 (2)	3.417 (3)	171 (1)
C1—H1C···O3^{ii}	0.96 (2)	2.58 (2)	3.233 (3)	125 (2)

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

Fig. 1

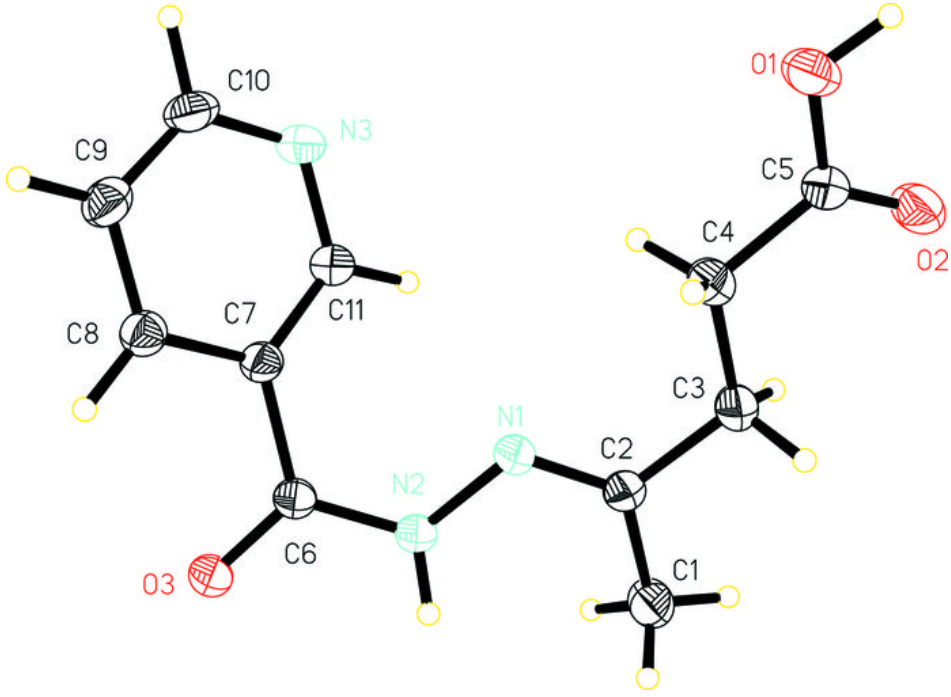

Fig. 2