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(*E*)-4-[2-(2-Hydroxybenzoyl)hydrazinylidene]pentanoic acid

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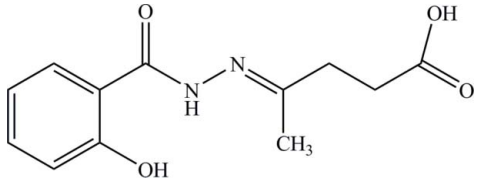
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.048; wR factor = 0.114; data-to-parameter ratio = 12.8.

The title molecule, $C_{12}H_{14}N_2O_4$, adopts a *trans* configuration with respect to the C=N double bond. The amino group is involved an in intramolecular N—H···O hydrogen bond. In the crystal structure, intermolecular O—H···O hydrogen bonds link the molecules into doubled sheets parallel to the (101) plane.

Related literature

For the synthesis and structures of some organotin(IV) complexes of related tridentate hydrazone ligands, see: Yin *et al.* (2008).



Experimental

Crystal data

$C_{12}H_{14}N_2O_4$ M_r = 250.25
Monoclinic, $C2/c$
a = 24.445 (2) Å
b = 8.4683 (8) Å
c = 13.1204 (12) Å
β = 118.560 (1)°
V = 2385.6 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 K
0.45 × 0.20 × 0.17 mm

Data collection

Bruker SMART 1000 diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.954, T_{max} = 0.982
5738 measured reflections
2092 independent reflections
1013 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.114
S = 1.00
2092 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O2^{i}	0.82	1.91	2.679 (3)	155
O1—H1···O3^{ii}	0.82	1.80	2.570 (3)	155
N2—H2···O4	0.86	1.95	2.635 (3)	136

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5127).

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S1. Comment

Recently, we have reported some organotin(IV) complexes with hydrazone ligands (Yin *et al.*, 2008). As an extension of our work on the structural characterization of hydrazone compounds, the title compound, (I), is reported here.

In the title compound, (I), the N1═C4 bond length of 1.276 (3) Å is a typical double bond value, while the N2—C6 [1.337 (3) Å] and N1—N2 [1.390 (3) Å] bonds are intermediate between double and single bonds because of conjugation effects in the molecule.

In the crystal structure, intermolecular O—H···O hydrogen bonds (Table 1) link the molecules into doubled sheets parallel to (101) plane.

S2. Experimental

Compound (I) was synthesized by the reaction of 2-hydroxybenzohydrazide (10 mmol) with 4-oxopentanoic acid (10 mmol). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

S3. Refinement

The H atoms were positioned geometrically, with methyl C—H distances of 0.96 Å, methylene C—H distances of 0.93 Å, aromatic C—H distances of 0.93 Å, N—H distances of 0.86 Å and O—H distances of 0.82 Å, and refined as riding on their parent atoms, with U_{iso}(H) = 1.2-1.5 U_{eq} of the parent atom.

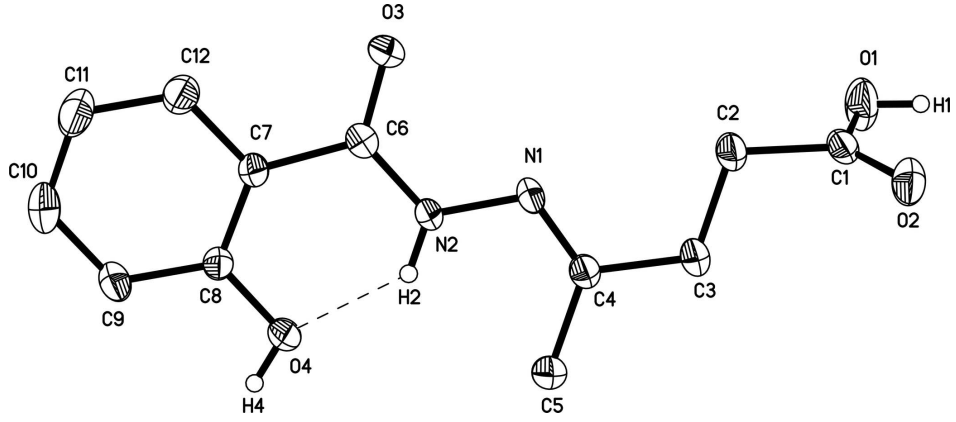

Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids. Dashed line denotes hydrogen bond.

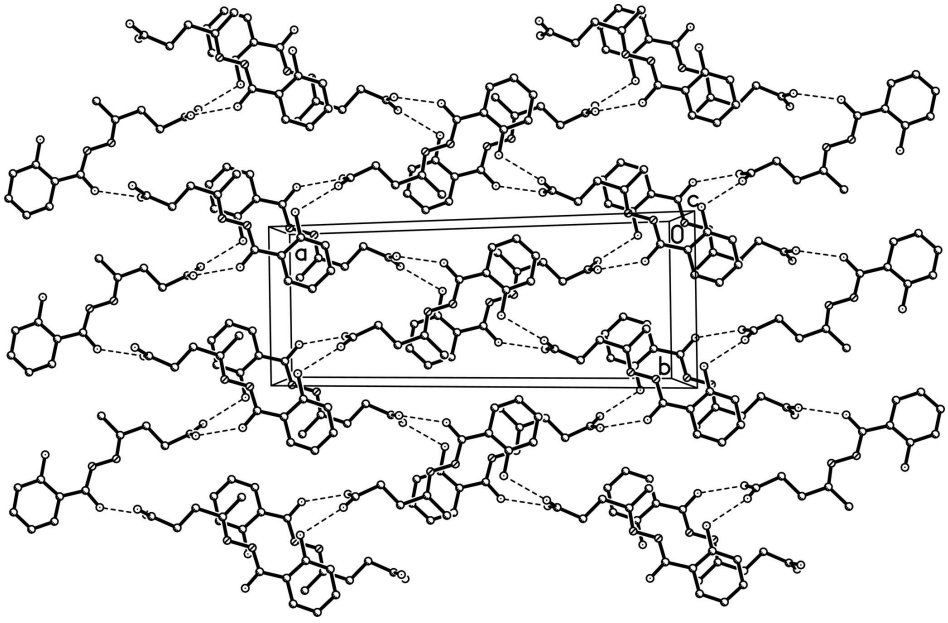

Figure 2

A portion of the crystal packing, showing hydrogen-bonded (dashed lines) two-dimensional network. H atoms have been omitted for clarity.

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Crystal data

$C_{12}H_{14}N_2O_4$
$M_r = 250.25$
Monoclinic, $C2/c$
$a = 24.445$ (2) Å
$b = 8.4683$ (8) Å
$c = 13.1204$ (12) Å
$\beta = 118.560$ (1)°
$V = 2385.6$ (4) Å^3
$Z = 8$
$F(000) = 1056$
$D_x = 1.394$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 770 reflections
$\theta = 2.6$–$20.7°$
$\mu = 0.11$ mm^{-1}
$T = 298$ K
Block, colourless
0.45 × 0.20 × 0.17 mm

Data collection

Bruker SMART 1000 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.954$, $T_{max} = 0.982$
5738 measured reflections
2092 independent reflections
1013 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.9°$
$h = -28 \rightarrow 14$
$k = -10 \rightarrow 9$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.114$
$S = 1.00$
2092 reflections
164 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0385P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} < 0.001$
H-atom parameters constrained	$\Delta\rho_{max} = 0.23$ e Å^{-3}
	$\Delta\rho_{min} = -0.26$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.39247 (9)	0.4503 (3)	0.29554 (19)	0.0352 (6)
N2	0.45430 (9)	0.4964 (3)	0.34082 (18)	0.0351 (6)
H2	0.4823	0.4263	0.3536	0.042*
O1	0.18399 (10)	0.3011 (3)	0.2303 (2)	0.0700 (7)
H1	0.1496	0.2591	0.2013	0.105*
O2	0.17818 (9)	0.2415 (2)	0.0602 (2)	0.0586 (7)
O3	0.43347 (9)	0.7535 (2)	0.35295 (18)	0.0528 (6)
O4	0.57296 (8)	0.4200 (2)	0.43211 (16)	0.0447 (6)
H4	0.6050	0.3692	0.4522	0.067*
C1	0.20548 (13)	0.2976 (3)	0.1566 (3)	0.0429 (8)
C2	0.26898 (12)	0.3703 (3)	0.2044 (3)	0.0485 (9)
H2A	0.2796	0.4225	0.2774	0.058*
H2B	0.2689	0.4491	0.1506	0.058*
C3	0.31687 (12)	0.2454 (3)	0.2235 (3)	0.0445 (8)
H3A	0.3143	0.1641	0.2732	0.053*
H3B	0.3062	0.1969	0.1494	0.053*
C4	0.38293 (12)	0.3024 (3)	0.2768 (2)	0.0367 (8)
C5	0.43127 (12)	0.1791 (3)	0.3004 (3)	0.0488 (9)
H5A	0.4466	0.1895	0.2456	0.073*
H5B	0.4133	0.0763	0.2931	0.073*
H5C	0.4651	0.1924	0.3776	0.073*
C6	0.47095 (13)	0.6478 (4)	0.3649 (2)	0.0352 (7)
C7	0.53770 (12)	0.6864 (3)	0.4048 (2)	0.0333 (7)
C8	0.58579 (12)	0.5776 (3)	0.4357 (2)	0.0337 (7)
C9	0.64610 (13)	0.6295 (4)	0.4698 (2)	0.0461 (8)
H9	0.6779	0.5562	0.4897	0.055*
C10	0.65900 (15)	0.7874 (4)	0.4743 (3)	0.0570 (10)
H10	0.6994	0.8208	0.4969	0.068*
C11	0.61268 (15)	0.8956 (4)	0.4456 (3)	0.0583 (10)
H11	0.6215	1.0029	0.4492	0.070*
C12	0.55293 (13)	0.8455 (3)	0.4115 (2)	0.0457 (8)
H12	0.5217	0.9205	0.3922	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0219 (14)	0.0424 (16)	0.0391 (16)	−0.0016 (11)	0.0127 (11)	0.0005 (12)
N2	0.0233 (13)	0.0326 (14)	0.0479 (16)	−0.0001 (11)	0.0160 (12)	−0.0001 (12)
O1	0.0452 (14)	0.0932 (19)	0.0764 (18)	−0.0259 (12)	0.0329 (13)	−0.0195 (15)
O2	0.0395 (13)	0.0633 (16)	0.0669 (17)	−0.0156 (11)	0.0206 (12)	−0.0133 (13)

O3	0.0373 (12)	0.0370 (13)	0.0851 (17)	0.0070 (10)	0.0301 (12)	0.0027 (12)
O4	0.0293 (11)	0.0377 (13)	0.0612 (15)	0.0056 (9)	0.0167 (10)	0.0010 (11)
C1	0.0274 (18)	0.0371 (19)	0.061 (3)	0.0012 (14)	0.0184 (18)	0.0041 (18)
C2	0.0297 (17)	0.0404 (18)	0.070 (2)	−0.0044 (14)	0.0190 (16)	0.0016 (17)
C3	0.0279 (17)	0.0460 (19)	0.057 (2)	−0.0046 (14)	0.0180 (16)	−0.0035 (16)
C4	0.0302 (17)	0.0376 (19)	0.042 (2)	−0.0009 (14)	0.0172 (15)	0.0004 (15)
C5	0.0409 (19)	0.0369 (18)	0.070 (2)	−0.0001 (14)	0.0278 (17)	−0.0053 (16)
C6	0.0304 (17)	0.040 (2)	0.0349 (19)	0.0011 (15)	0.0157 (14)	0.0011 (15)
C7	0.0273 (16)	0.0350 (18)	0.0343 (18)	−0.0020 (13)	0.0122 (14)	−0.0014 (14)
C8	0.0319 (17)	0.0339 (18)	0.0371 (19)	−0.0036 (14)	0.0179 (15)	−0.0028 (14)
C9	0.0299 (18)	0.054 (2)	0.054 (2)	−0.0030 (15)	0.0196 (17)	−0.0033 (17)
C10	0.038 (2)	0.069 (3)	0.058 (2)	−0.0189 (19)	0.0188 (18)	−0.005 (2)
C11	0.053 (2)	0.047 (2)	0.067 (3)	−0.0174 (18)	0.022 (2)	−0.0002 (18)
C12	0.042 (2)	0.039 (2)	0.049 (2)	−0.0028 (15)	0.0163 (17)	0.0008 (16)

Geometric parameters (Å, º)

N1—C4	1.276 (3)	C3—H3B	0.9700
N1—N2	1.390 (3)	C4—C5	1.494 (3)
N2—C6	1.337 (3)	C5—H5A	0.9600
N2—H2	0.8600	C5—H5B	0.9600
O1—C1	1.303 (3)	C5—H5C	0.9600
O1—H1	0.8200	C6—C7	1.492 (3)
O2—C1	1.209 (3)	C7—C12	1.390 (3)
O3—C6	1.236 (3)	C7—C8	1.393 (3)
O4—C8	1.366 (3)	C8—C9	1.392 (3)
O4—H4	0.8200	C9—C10	1.368 (4)
C1—C2	1.501 (4)	C9—H9	0.9300
C2—C3	1.507 (3)	C10—C11	1.363 (4)
C2—H2A	0.9700	C10—H10	0.9300
C2—H2B	0.9700	C11—C12	1.374 (4)
C3—C4	1.500 (3)	C11—H11	0.9300
C3—H3A	0.9700	C12—H12	0.9300
C4—N1—N2	114.8 (2)	H5A—C5—H5B	109.5
C6—N2—N1	121.1 (2)	C4—C5—H5C	109.5
C6—N2—H2	119.5	H5A—C5—H5C	109.5
N1—N2—H2	119.5	H5B—C5—H5C	109.5
C1—O1—H1	109.5	O3—C6—N2	122.8 (3)
C8—O4—H4	109.5	O3—C6—C7	120.4 (3)
O2—C1—O1	124.6 (3)	N2—C6—C7	116.8 (2)
O2—C1—C2	122.8 (3)	C12—C7—C8	117.4 (3)
O1—C1—C2	112.5 (3)	C12—C7—C6	116.7 (2)
C1—C2—C3	110.3 (2)	C8—C7—C6	125.9 (3)
C1—C2—H2A	109.6	O4—C8—C9	120.7 (2)
C3—C2—H2A	109.6	O4—C8—C7	119.2 (2)
C1—C2—H2B	109.6	C9—C8—C7	120.1 (3)
C3—C2—H2B	109.6	C10—C9—C8	120.6 (3)

H2A—C2—H2B	108.1	C10—C9—H9	119.7
C4—C3—C2	115.4 (2)	C8—C9—H9	119.7
C4—C3—H3A	108.4	C11—C10—C9	120.1 (3)
C2—C3—H3A	108.4	C11—C10—H10	120.0
C4—C3—H3B	108.4	C9—C10—H10	120.0
C2—C3—H3B	108.4	C10—C11—C12	119.7 (3)
H3A—C3—H3B	107.5	C10—C11—H11	120.1
N1—C4—C5	126.3 (2)	C12—C11—H11	120.1
N1—C4—C3	117.5 (2)	C11—C12—C7	122.1 (3)
C5—C4—C3	116.2 (2)	C11—C12—H12	119.0
C4—C5—H5A	109.5	C7—C12—H12	119.0
C4—C5—H5B	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O2^{i}	0.82	1.91	2.679 (3)	155
O1—H1···O3^{ii}	0.82	1.80	2.570 (3)	155
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