

trans-4-(1-Naphthyl)-2-oxo-1,3-oxazolidine-5-carboxylic acid

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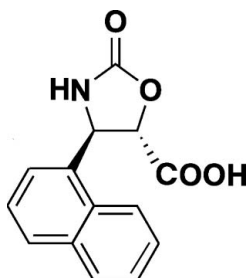
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Key indicators: single-crystal X-ray study; $T = 292$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.037; wR factor = 0.093; data-to-parameter ratio = 12.7.

The crystal structure of the title compound, $\text{C}_{14}\text{H}_{11}\text{NO}_4$, is influenced by $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, linking molecules into one-dimensional tapes running along the [010] direction.

Related literature

For general background regarding the title compound, see: Lu *et al.* (2008). For patterns in hydrogen bonding, see: Bernstein *et al.* (1995). For related literature, see: Barbachyn & Ford (2003); Evans (1982); Mukhtar & Wright (2005).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{11}\text{NO}_4$	$V = 2316.6$ (8) Å ³
$M_r = 257.24$	$Z = 8$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
$a = 8.7159$ (17) Å	$\mu = 0.11$ mm ⁻¹
$b = 12.817$ (3) Å	$T = 292$ (2) K
$c = 20.737$ (4) Å	$0.47 \times 0.38 \times 0.35$ mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer	2266 independent reflections
Absorption correction: none	2008 reflections with $I > 2\sigma(I)$
12484 measured reflections	$R_{\text{int}} = 0.016$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.093$	$\Delta\rho_{\text{max}} = 0.19$ e Å ⁻³
$S = 1.04$	$\Delta\rho_{\text{min}} = -0.20$ e Å ⁻³
2266 reflections	
178 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1A}\cdots\text{O4}^i$	0.94 (2)	1.72 (2)	2.6591 (15)	174 (2)
$\text{N1}-\text{H1}\cdots\text{O2}^{ii}$	0.834 (17)	2.247 (17)	3.0097 (18)	152.0 (15)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

We thank Dr Xiang-Gao Meng for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2191).

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supplementary materials

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***trans*-4-(1-Naphthyl)-2-oxo-1,3-oxazolidine-5-carboxylic acid**

J.-Y. Yang, Z.-H. Ming, J. An, Q.-L. Hua and L.-Q. Lu

Comment

Oxazolidin-2-ones are prevalent in biologically active molecules (Mukhtar *et al.*, 2005; Barbachyn, *et al.*, 2003), as well as versatile synthons in organic synthesis (Evans, 1982). Recently we reported a new cascade reaction to synthesize these compounds from stable sulfur ylides and nitroolefins (Lu *et al.*, 2008). In order to demonstrate the utility of this method we continued to hydrolyze *trans*-ethyl 4-(naphthalen-1-yl)-2-oxooxazolidine-5-carboxylate with LiOH to obtain *trans*-4-(naphthalen-1-yl)-2-oxooxazolidine-5-carboxylic acid, and we are presenting herein the X-ray crystallographic analysis of the title compound, thus obtained (Fig. 1),

The crystal structure is determined by N—H \cdots O and O—H \cdots O hydrogen bonds, defining R₂²(8) rings (Bernstein *et al.* (1995), which link molecules into one-dimensional hydrogen-bonded tapes along [010] (Fig. 2).

Experimental

The starting material, 4-(naphthalen-1-yl)-2-oxooxazolidine-5-carboxylate (100.0 mg, 0.35 mmol) was added to the aqueous solution of LiOH (88.1 mg, 2.10 mmol, 2.5 ml H₂O) and the reaction mixture was stirred for 2.5 h. By adjusting the pH = 6–7 with concentrated HCl and 1M diluted HCl solution, the white solid precipitated and was filtrated. The residue was washed with cold water and diethyl ester, the desired product was collected as white power after dryness with 93% yield. Recrystallization from CH₃OH—H₂O provided the crystalline solid.

Refinement

All H atoms bonded to C atoms were initially located in difference Fourier maps and then constrained to their ideal geometry positions with C—H=0.96Å (methyl), 0.97Å (methylene). H atoms bonded to N and O were found in difference maps and refined with N/O—H distances free. In all cases U_{iso} ((H) values were set to x times U_{eq} (host), $x=1.5$ (methyl), $x=1.2$ (methylene), $x=1.2$ (N), $x=1.5$ (O).

Figures

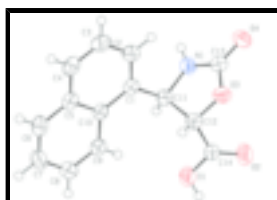


Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

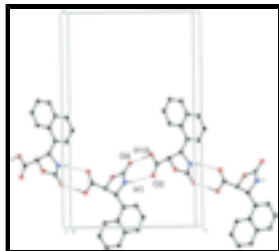


Fig. 2. Packing of (I) with hydrogen bonds drawn as dashed lines showing the formation of hydrogen-bonded $R_2^2(8)$ loops involving $\text{NH}\cdots\text{O}$ and $\text{OH}\cdots\text{O}$ hydrogen bonds. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) $-x, y - 1/2, -z + 1/2$; (ii) $-x, y + 1/2, z + 1/2$]

***trans*-4-(1-Naphthyl)-2-oxo-1,3-oxazolidine-5-carboxylic acid**

Crystal data

$\text{C}_{14}\text{H}_{11}\text{NO}_4$

$M_r = 257.24$

Orthorhombic, *Pbca*

Hall symbol: $-P\ 2ac\ 2ab$

$a = 8.7159\ (17)\ \text{\AA}$

$b = 12.817\ (3)\ \text{\AA}$

$c = 20.737\ (4)\ \text{\AA}$

$V = 2316.6\ (8)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 1072$

$D_x = 1.475\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 5223 reflections

$\theta = 2.8\text{--}28.9^\circ$

$\mu = 0.11\ \text{mm}^{-1}$

$T = 292\ (2)\ \text{K}$

Block, colorless

$0.47 \times 0.38 \times 0.35\ \text{mm}$

Data collection

Bruker SMART 4K CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 292\ (2)\ \text{K}$

φ and ω scans

Absorption correction: none

12484 measured reflections

2266 independent reflections

2008 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$

$\theta_{\text{max}} = 26.0^\circ$

$\theta_{\text{min}} = 3.0^\circ$

$h = -9 \rightarrow 10$

$k = -15 \rightarrow 15$

$l = -25 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.093$

$S = 1.05$

2266 reflections

178 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.7823P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\text{max}} < 0.001$$

$$\Delta\rho_{\text{max}} = 0.19\ \text{e \AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.20\ \text{e \AA}^{-3}$$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.10060 (15)	0.89669 (10)	0.40612 (6)	0.0322 (3)
C2	0.18399 (17)	0.98662 (11)	0.40013 (7)	0.0415 (3)
H2	0.1971	1.0159	0.3595	0.050*
C3	0.25014 (19)	1.03567 (11)	0.45381 (7)	0.0474 (4)
H3	0.3063	1.0967	0.4484	0.057*
C4	0.23264 (17)	0.99464 (12)	0.51357 (7)	0.0438 (4)
H4	0.2764	1.0280	0.5489	0.053*
C5	0.14847 (15)	0.90147 (11)	0.52271 (6)	0.0363 (3)
C6	0.13034 (17)	0.85684 (12)	0.58449 (7)	0.0435 (4)
H6	0.1731	0.8900	0.6201	0.052*
C7	0.05170 (18)	0.76650 (13)	0.59291 (7)	0.0468 (4)
H7	0.0424	0.7377	0.6339	0.056*
C8	-0.01545 (17)	0.71659 (12)	0.53978 (7)	0.0435 (3)
H8	-0.0694	0.6548	0.5458	0.052*
C9	-0.00239 (15)	0.75784 (10)	0.47936 (6)	0.0359 (3)
H9	-0.0490	0.7242	0.4448	0.043*
C10	0.08091 (14)	0.85104 (10)	0.46843 (6)	0.0315 (3)
C11	0.03291 (15)	0.84401 (10)	0.34692 (6)	0.0326 (3)
H11	-0.0684	0.8149	0.3567	0.039*
C12	0.13968 (15)	0.75872 (10)	0.31821 (6)	0.0356 (3)
H12	0.2150	0.7374	0.3507	0.043*
C13	0.13627 (16)	0.89576 (10)	0.24897 (7)	0.0370 (3)
C14	0.05709 (17)	0.66298 (10)	0.29259 (6)	0.0390 (3)
N1	0.02505 (13)	0.91215 (9)	0.29122 (5)	0.0363 (3)
H1	-0.0281 (18)	0.9662 (13)	0.2897 (7)	0.044*
O1	-0.01994 (14)	0.61525 (9)	0.33777 (5)	0.0538 (3)
H1A	-0.068 (3)	0.5549 (17)	0.3222 (10)	0.081*
O2	0.0653 (2)	0.63355 (10)	0.23870 (5)	0.0869 (5)
O3	0.21709 (11)	0.80945 (7)	0.26568 (5)	0.0429 (3)
O4	0.17042 (13)	0.94603 (8)	0.20100 (5)	0.0497 (3)

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0341 (6)	0.0298 (6)	0.0328 (7)	0.0010 (5)	0.0002 (5)	-0.0034 (5)
C2	0.0503 (8)	0.0350 (7)	0.0394 (7)	-0.0056 (6)	0.0002 (6)	0.0012 (6)
C3	0.0529 (9)	0.0346 (8)	0.0545 (9)	-0.0125 (6)	-0.0010 (7)	-0.0056 (6)
C4	0.0452 (8)	0.0422 (8)	0.0439 (8)	-0.0054 (6)	-0.0044 (6)	-0.0138 (6)
C5	0.0358 (7)	0.0378 (7)	0.0353 (7)	0.0037 (6)	-0.0010 (5)	-0.0071 (6)
C6	0.0452 (8)	0.0538 (9)	0.0316 (7)	0.0033 (7)	-0.0036 (6)	-0.0071 (6)
C7	0.0544 (9)	0.0542 (9)	0.0319 (7)	0.0063 (7)	0.0019 (6)	0.0065 (6)
C8	0.0505 (8)	0.0383 (8)	0.0415 (8)	-0.0020 (6)	0.0059 (6)	0.0035 (6)
C9	0.0402 (7)	0.0339 (7)	0.0337 (7)	-0.0017 (6)	0.0008 (5)	-0.0038 (5)
C10	0.0322 (6)	0.0306 (6)	0.0317 (6)	0.0032 (5)	0.0004 (5)	-0.0042 (5)
C11	0.0363 (7)	0.0314 (7)	0.0302 (6)	-0.0008 (5)	-0.0008 (5)	-0.0002 (5)
C12	0.0421 (7)	0.0314 (7)	0.0333 (7)	0.0010 (6)	0.0005 (6)	0.0009 (5)
C13	0.0407 (7)	0.0313 (7)	0.0389 (7)	0.0004 (6)	0.0004 (6)	0.0004 (6)
C14	0.0563 (9)	0.0307 (7)	0.0301 (7)	-0.0004 (6)	0.0037 (6)	-0.0003 (5)
N1	0.0398 (6)	0.0369 (6)	0.0322 (6)	0.0083 (5)	0.0001 (5)	0.0023 (5)
O1	0.0767 (8)	0.0478 (6)	0.0370 (6)	-0.0233 (6)	0.0127 (5)	-0.0085 (5)
O2	0.1626 (15)	0.0612 (8)	0.0371 (6)	-0.0509 (9)	0.0266 (8)	-0.0154 (6)
O3	0.0437 (5)	0.0328 (5)	0.0521 (6)	0.0043 (4)	0.0128 (5)	0.0052 (4)
O4	0.0620 (7)	0.0415 (6)	0.0457 (6)	0.0059 (5)	0.0143 (5)	0.0106 (5)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.3682 (19)	C8—H8	0.9300
C1—C10	1.4289 (17)	C9—C10	1.4160 (18)
C1—C11	1.5202 (17)	C9—H9	0.9300
C2—C3	1.403 (2)	C11—N1	1.4497 (16)
C2—H2	0.9300	C11—C12	1.5542 (18)
C3—C4	1.355 (2)	C11—H11	0.9800
C3—H3	0.9300	C12—O3	1.4368 (16)
C4—C5	1.414 (2)	C12—C14	1.5185 (19)
C4—H4	0.9300	C12—H12	0.9800
C5—C6	1.4117 (19)	C13—O4	1.2220 (16)
C5—C10	1.4253 (18)	C13—N1	1.3233 (18)
C6—C7	1.357 (2)	C13—O3	1.3565 (16)
C6—H6	0.9300	C14—O2	1.1817 (17)
C7—C8	1.402 (2)	C14—O1	1.3049 (17)
C7—H7	0.9300	N1—H1	0.834 (17)
C8—C9	1.3647 (19)	O1—H1A	0.94 (2)
C2—C1—C10	119.40 (12)	C9—C10—C5	117.90 (12)
C2—C1—C11	120.46 (12)	C9—C10—C1	123.49 (11)
C10—C1—C11	120.12 (11)	C5—C10—C1	118.61 (12)
C1—C2—C3	121.59 (13)	N1—C11—C1	113.21 (11)
C1—C2—H2	119.2	N1—C11—C12	98.44 (10)
C3—C2—H2	119.2	C1—C11—C12	112.92 (11)

C4—C3—C2	120.38 (13)	N1—C11—H11	110.6
C4—C3—H3	119.8	C1—C11—H11	110.6
C2—C3—H3	119.8	C12—C11—H11	110.6
C3—C4—C5	120.58 (13)	O3—C12—C14	108.85 (10)
C3—C4—H4	119.7	O3—C12—C11	104.67 (10)
C5—C4—H4	119.7	C14—C12—C11	114.74 (11)
C6—C5—C4	121.45 (13)	O3—C12—H12	109.5
C6—C5—C10	119.12 (13)	C14—C12—H12	109.5
C4—C5—C10	119.43 (12)	C11—C12—H12	109.5
C7—C6—C5	121.27 (13)	O4—C13—N1	129.31 (13)
C7—C6—H6	119.4	O4—C13—O3	120.76 (12)
C5—C6—H6	119.4	N1—C13—O3	109.92 (12)
C6—C7—C8	119.93 (13)	O2—C14—O1	124.10 (14)
C6—C7—H7	120.0	O2—C14—C12	124.05 (13)
C8—C7—H7	120.0	O1—C14—C12	111.81 (11)
C9—C8—C7	120.66 (14)	C13—N1—C11	113.40 (11)
C9—C8—H8	119.7	C13—N1—H1	120.9 (11)
C7—C8—H8	119.7	C11—N1—H1	123.9 (11)
C8—C9—C10	121.10 (12)	C14—O1—H1A	111.7 (13)
C8—C9—H9	119.5	C13—O3—C12	108.59 (10)
C10—C9—H9	119.5		
C10—C1—C2—C3	-0.4 (2)	C2—C1—C11—N1	-17.73 (18)
C11—C1—C2—C3	-178.76 (13)	C10—C1—C11—N1	163.91 (11)
C1—C2—C3—C4	0.0 (2)	C2—C1—C11—C12	93.05 (15)
C2—C3—C4—C5	0.4 (2)	C10—C1—C11—C12	-85.31 (14)
C3—C4—C5—C6	179.30 (15)	N1—C11—C12—O3	21.29 (12)
C3—C4—C5—C10	-0.4 (2)	C1—C11—C12—O3	-98.41 (12)
C4—C5—C6—C7	-178.87 (14)	N1—C11—C12—C14	-97.95 (12)
C10—C5—C6—C7	0.8 (2)	C1—C11—C12—C14	142.36 (11)
C5—C6—C7—C8	-1.0 (2)	O3—C12—C14—O2	4.2 (2)
C6—C7—C8—C9	0.1 (2)	C11—C12—C14—O2	121.12 (18)
C7—C8—C9—C10	0.9 (2)	O3—C12—C14—O1	-177.87 (12)
C8—C9—C10—C5	-1.09 (19)	C11—C12—C14—O1	-61.00 (16)
C8—C9—C10—C1	178.86 (13)	O4—C13—N1—C11	-171.98 (14)
C6—C5—C10—C9	0.22 (19)	O3—C13—N1—C11	7.85 (16)
C4—C5—C10—C9	179.92 (12)	C1—C11—N1—C13	101.23 (13)
C6—C5—C10—C1	-179.73 (12)	C12—C11—N1—C13	-18.24 (14)
C4—C5—C10—C1	-0.03 (19)	O4—C13—O3—C12	-172.25 (13)
C2—C1—C10—C9	-179.53 (13)	N1—C13—O3—C12	7.90 (15)
C11—C1—C10—C9	-1.16 (19)	C14—C12—O3—C13	104.23 (12)
C2—C1—C10—C5	0.42 (19)	C11—C12—O3—C13	-18.91 (13)
C11—C1—C10—C5	178.79 (11)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots O4 ⁱ	0.94 (2)	1.72 (2)	2.6591 (15)	174 (2)
N1—H1 \cdots O2 ⁱⁱ	0.834 (17)	2.247 (17)	3.0097 (18)	152.0 (15)

supplementary materials

Symmetry codes: (i) $-x, y-1/2, -z+1/2$; (ii) $-x, y+1/2, -z+1/2$.

Fig. 1

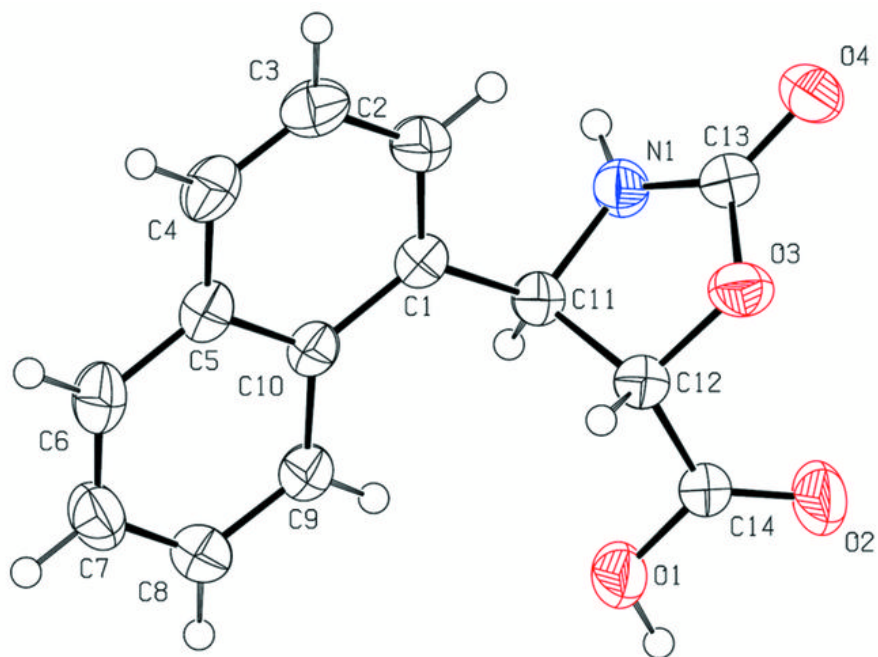


Fig. 2

