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4-(4-Ethoxybenzyl)-1,3-oxazolidin-2-one

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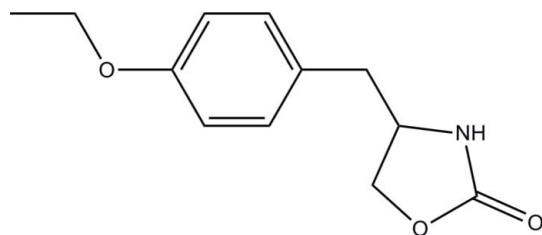
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å;
 R factor = 0.043; wR factor = 0.150; data-to-parameter ratio = 8.5.

 In the title compound, $\text{C}_{12}\text{H}_{15}\text{NO}_3$, the ethoxybenzyl ring plane forms a dihedral angle of $60.3(4)^\circ$ with the mean plane of the oxazolidine ring. The molecules are linked through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into a chain running in the b direction.

Related literature

 For background literature, see: Chrzanowska & Rozwadowska (2004); Rozwadowska (1994); Scott & Williams (2002); Tussetschläger *et al.* (2007).


Experimental

Crystal data

 $\text{C}_{12}\text{H}_{15}\text{NO}_3$
 $M_r = 221.25$
 Orthorhombic, $P2_12_12_1$
 $a = 5.7960(12)$ Å

 $b = 9.924(2)$ Å
 $c = 20.209(4)$ Å
 $V = 1162.4(4)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.30 \times 0.20 \times 0.10$ mm

Data collection

 Enraf–Nonius CAD-4
 diffractometer
 Absorption correction: ψ scan
 (CAD-4 Software; Enraf–Nonius,
 1989)
 $T_{\min} = 0.973$, $T_{\max} = 0.991$
 2427 measured reflections

 1246 independent reflections
 904 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$
 3 standard reflections
 every 200 reflections
 intensity decay: 1%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.150$
 $S = 1.01$
 1246 reflections

 146 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.17$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N}-\text{H}0\text{A}\cdots\text{O}3^i$	0.86	1.99	2.845 (4)	171

 Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, -z + 2$.

Data collection: CAD-4 Software (Enraf–Nonius, 1989); cell refinement: CAD-4 Software; data reduction: XCAD4 (Harms & Wocadlo, 1995); 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: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2144).

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supporting information

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S1. Comment

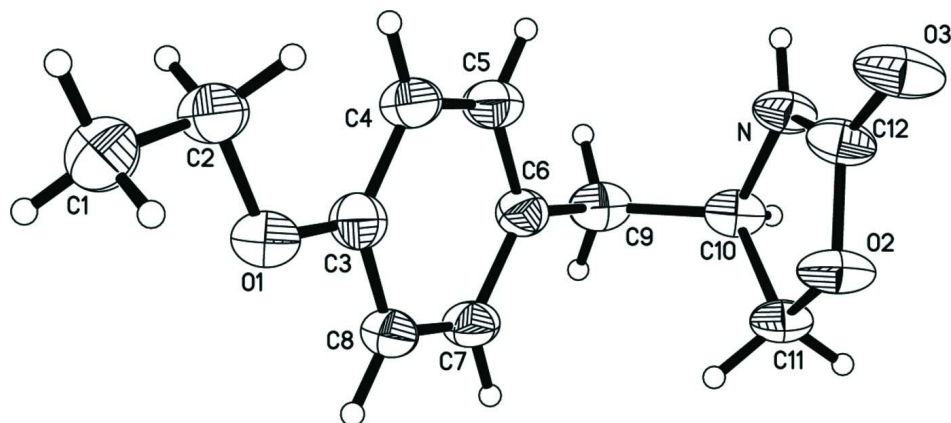
Tetrahydroisoquinoline alkaloids have received much interest because of their tremendous structural diversity and broad spectrum of biological and pharmaceutical activities (Rozwadowska, 1994; Scott & Williams, 2002; Chrzanowska & Rozwadowska, 2004). As part of our own work in this area, we prepared the title compound, (I), as an intermediate in the synthesis of tyrosine-derived *N*-[(phenylsulfonyl)alkyl]oxazolidinones as an extension of Petrini's methodology (Tussetschläger, *et al.*, 2007). The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the mean planes of the C1 - C9/O1 ethoxybenzyl ring and the C10/N/C12/O2/C11/O3 oxazolidine ring is 60.3 (4)°. In the crystal structure, adjacent molecules are linked through N—H⋯O type hydrogen-bonding interactions resulting in chains running in the *b* direction (Table 1). The structure also contains non-classical hydrogen bonds of the type C—H⋯O linking the molecules into chains along the *a*-axis.

S2. Experimental

Sodium borohydride was added to a solution of 2-(*tert*-butoxycarbonylamino)-3-(4-ethoxyphenyl)propanoic acid (3.09 g, 10 mmol) in tetrahydrofuran (50 ml). Then methanol (5 ml) was slowly added to the resulting suspension and the temperature kept below 243 K. After the mixture was stirred for 1 h at room temperature, the excess reagent was destroyed by addition of acetic acid (1 ml). The solvent was evaporated, and the oily residue was diluted with water (50 ml) and extracted three times with ethyl acetate (25 ml) each. The combined organic extracts were washed with brine, dried with sodium sulfate, and concentrated *in vacuo*. The crude *tert*-butyl 1-(4-ethoxyphenyl)-3-hydroxypropan-2-ylcarbamate was obtained 2.7 g. Then *tert*-butyl 1-(4-ethoxyphenyl)-3-hydroxypropan-2-ylcarbamate (2.7 g) in THF (50 ml) was added to a suspension of sodium hydride (0.92 g, 23 mmol) in THF (120 ml) over a period of 20 min, stirred for 12 h, then quenched with a saturated solution of aqueous ammonium chloride (45 ml). The reaction mixture was then extracted three times with ethyl acetate (25 ml) each, the organic layers combined, washed with aqueous hydrochloric acid (60 ml, 5% solution), saturated NaHCO₃ solution (60 ml), and brine (60 ml), and then dried over sodium sulfate. The solvent was then removed *in vacuo* to yield the title compound (1.81 g, 8.2 mmol) as a white solid. The title compound was crystallized by slow evaporation of a solution in methanol.

S3. Refinement

Positional parameters of all the H atoms bonded to C atoms were calculated geometrically and were allowed to ride on the C atoms to which they are bonded, with N—H = 0.86 and C—H = 0.93, 0.96, 0.97 and 0.98 Å for aryl, methyl, methylene and methine H atoms, respectively, and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl})$ and $1.2U_{\text{eq}}(\text{the rest})$ parent atoms. An absolute configuration could not be established by anomalous dispersion effects. Therefore, Friedel pairs (846) were merged.

**Figure 1**

A view of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

4-(4-Ethoxybenzyl)-1,3-oxazolidin-2-one

Crystal data

$C_{12}H_{15}NO_3$

$M_r = 221.25$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 5.7960$ (12) Å

$b = 9.924$ (2) Å

$c = 20.209$ (4) Å

$V = 1162.4$ (4) Å³

$Z = 4$

$F(000) = 472$

$D_x = 1.264$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9\text{--}12^\circ$

$\mu = 0.09$ mm⁻¹

$T = 293$ K

Needle, colourless

$0.30 \times 0.20 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega/2\theta$ scans

Absorption correction: ψ scan

(CAD-4 Software; Enraf–Nonius, 1989)

$T_{\min} = 0.973$, $T_{\max} = 0.991$

2427 measured reflections

1246 independent reflections

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

$R_{\text{int}} = 0.043$

$\theta_{\max} = 25.3^\circ$, $\theta_{\min} = 2.0^\circ$

$h = 0 \rightarrow 6$

$k = 0 \rightarrow 11$

$l = -24 \rightarrow 24$

3 standard reflections every 200 reflections

intensity decay: 1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.150$

$S = 1.01$

1246 reflections

146 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 0.28P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.17$ e Å⁻³

$\Delta\rho_{\min} = -0.19$ e Å⁻³

Extinction correction: *SHELXL97* (Sheldrick, 2008)

Extinction coefficient: 0.031 (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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N	-0.1179 (6)	0.5921 (3)	1.00790 (17)	0.0589 (9)
H0A	-0.1581	0.6728	1.0182	0.071*
O1	0.4026 (6)	0.3182 (3)	1.25901 (14)	0.0754 (9)
O2	0.0990 (5)	0.4271 (3)	0.97230 (16)	0.0696 (9)
O3	0.2494 (5)	0.6348 (3)	0.97241 (19)	0.0845 (11)
C1	0.7072 (9)	0.3538 (5)	1.3348 (2)	0.0801 (14)
H1A	0.7873	0.4207	1.3603	0.120*
H1B	0.8134	0.3107	1.3051	0.120*
H1C	0.6415	0.2878	1.3639	0.120*
C2	0.5179 (9)	0.4202 (4)	1.2955 (2)	0.0725 (12)
H2A	0.4105	0.4650	1.3250	0.087*
H2B	0.5824	0.4869	1.2657	0.087*
C3	0.2251 (8)	0.3587 (4)	1.21793 (19)	0.0596 (11)
C4	0.1675 (9)	0.4911 (4)	1.2056 (2)	0.0651 (12)
H4A	0.2504	0.5608	1.2252	0.078*
C5	-0.0159 (8)	0.5190 (4)	1.1637 (2)	0.0643 (12)
H5A	-0.0542	0.6086	1.1559	0.077*
C6	-0.1449 (8)	0.4190 (4)	1.13291 (19)	0.0595 (11)
C7	-0.0758 (10)	0.2863 (4)	1.1469 (2)	0.0648 (13)
H7A	-0.1548	0.2156	1.1270	0.078*
C8	0.1011 (10)	0.2573 (4)	1.1883 (2)	0.0686 (13)
H8A	0.1391	0.1679	1.1967	0.082*
C9	-0.3390 (7)	0.4521 (4)	1.0869 (2)	0.0660 (12)
H9A	-0.4173	0.5317	1.1033	0.079*
H9B	-0.4490	0.3784	1.0877	0.079*
C10	-0.2686 (7)	0.4773 (4)	1.0154 (2)	0.0558 (10)
H10A	-0.4074	0.4910	0.9885	0.067*
C11	-0.1210 (7)	0.3682 (4)	0.9839 (2)	0.0568 (10)
H11A	-0.1071	0.2914	1.0134	0.068*
H11B	-0.1892	0.3381	0.9427	0.068*
C12	0.0875 (7)	0.5618 (4)	0.9838 (2)	0.0565 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.0555 (19)	0.0374 (15)	0.084 (2)	0.0096 (16)	0.0052 (19)	-0.0044 (15)
O1	0.096 (2)	0.0555 (16)	0.0745 (18)	-0.0051 (18)	0.000 (2)	-0.0037 (14)
O2	0.0475 (15)	0.0404 (13)	0.121 (2)	0.0004 (13)	0.0102 (17)	-0.0137 (15)
O3	0.0402 (15)	0.0525 (16)	0.161 (3)	-0.0080 (15)	0.003 (2)	-0.0047 (18)
C1	0.081 (3)	0.082 (3)	0.077 (3)	0.000 (3)	0.000 (3)	-0.009 (3)
C2	0.079 (3)	0.063 (2)	0.076 (3)	-0.006 (3)	-0.001 (3)	-0.009 (2)
C3	0.067 (3)	0.058 (2)	0.054 (2)	-0.005 (2)	0.010 (2)	0.0022 (18)
C4	0.079 (3)	0.051 (2)	0.065 (2)	-0.007 (2)	0.002 (3)	-0.0078 (19)
C5	0.071 (3)	0.048 (2)	0.074 (3)	-0.001 (2)	0.011 (3)	-0.008 (2)
C6	0.066 (3)	0.053 (2)	0.059 (2)	-0.010 (2)	0.020 (2)	-0.0044 (18)
C7	0.083 (3)	0.048 (2)	0.064 (2)	-0.016 (2)	0.007 (3)	-0.0046 (18)
C8	0.100 (4)	0.045 (2)	0.061 (2)	-0.006 (3)	0.006 (3)	0.0016 (18)
C9	0.054 (2)	0.059 (2)	0.085 (3)	-0.003 (2)	0.015 (2)	-0.012 (2)
C10	0.0358 (18)	0.049 (2)	0.082 (3)	0.0002 (18)	-0.007 (2)	-0.005 (2)
C11	0.054 (2)	0.048 (2)	0.069 (2)	-0.010 (2)	-0.002 (2)	-0.0077 (18)
C12	0.044 (2)	0.0382 (18)	0.088 (3)	0.0028 (18)	-0.011 (2)	-0.0025 (19)

Geometric parameters (Å, °)

N—C12	1.321 (5)	C4—C5	1.387 (6)
N—C10	1.444 (5)	C4—H4A	0.9300
N—H0A	0.8600	C5—C6	1.390 (6)
O1—C3	1.382 (5)	C5—H5A	0.9300
O1—C2	1.420 (5)	C6—C7	1.405 (6)
O2—C12	1.358 (5)	C6—C9	1.497 (6)
O2—C11	1.423 (5)	C7—C8	1.354 (7)
O3—C12	1.208 (5)	C7—H7A	0.9300
C1—C2	1.506 (6)	C8—H8A	0.9300
C1—H1A	0.9600	C9—C10	1.522 (6)
C1—H1B	0.9600	C9—H9A	0.9700
C1—H1C	0.9600	C9—H9B	0.9700
C2—H2A	0.9700	C10—C11	1.519 (5)
C2—H2B	0.9700	C10—H10A	0.9800
C3—C8	1.374 (6)	C11—H11A	0.9700
C3—C4	1.379 (6)	C11—H11B	0.9700
C12—N—C10	113.8 (3)	C7—C6—C9	123.1 (4)
C12—N—H0A	123.1	C8—C7—C6	122.7 (4)
C10—N—H0A	123.1	C8—C7—H7A	118.7
C3—O1—C2	117.1 (3)	C6—C7—H7A	118.7
C12—O2—C11	109.4 (3)	C7—C8—C3	120.6 (4)
C2—C1—H1A	109.5	C7—C8—H8A	119.7
C2—C1—H1B	109.5	C3—C8—H8A	119.7
H1A—C1—H1B	109.5	C6—C9—C10	115.1 (3)
C2—C1—H1C	109.5	C6—C9—H9A	108.5

H1A—C1—H1C	109.5	C10—C9—H9A	108.5
H1B—C1—H1C	109.5	C6—C9—H9B	108.5
O1—C2—C1	107.8 (4)	C10—C9—H9B	108.5
O1—C2—H2A	110.2	H9A—C9—H9B	107.5
C1—C2—H2A	110.2	N—C10—C11	100.2 (3)
O1—C2—H2B	110.2	N—C10—C9	113.0 (3)
C1—C2—H2B	110.2	C11—C10—C9	115.5 (3)
H2A—C2—H2B	108.5	N—C10—H10A	109.2
C8—C3—C4	119.5 (4)	C11—C10—H10A	109.2
C8—C3—O1	116.0 (4)	C9—C10—H10A	109.2
C4—C3—O1	124.4 (4)	O2—C11—C10	106.3 (3)
C3—C4—C5	119.1 (4)	O2—C11—H11A	110.5
C3—C4—H4A	120.5	C10—C11—H11A	110.5
C5—C4—H4A	120.5	O2—C11—H11B	110.5
C4—C5—C6	122.9 (4)	C10—C11—H11B	110.5
C4—C5—H5A	118.6	H11A—C11—H11B	108.7
C6—C5—H5A	118.6	O3—C12—N	129.3 (4)
C5—C6—C7	115.2 (4)	O3—C12—O2	121.3 (4)
C5—C6—C9	121.7 (4)	N—C12—O2	109.4 (4)
C3—O1—C2—C1	-178.3 (3)	C5—C6—C9—C10	85.5 (5)
C2—O1—C3—C8	-173.6 (4)	C7—C6—C9—C10	-92.8 (5)
C2—O1—C3—C4	6.2 (6)	C12—N—C10—C11	-5.5 (4)
C8—C3—C4—C5	0.3 (6)	C12—N—C10—C9	118.0 (4)
O1—C3—C4—C5	-179.5 (4)	C6—C9—C10—N	-62.6 (4)
C3—C4—C5—C6	-0.4 (6)	C6—C9—C10—C11	52.0 (5)
C4—C5—C6—C7	-0.3 (6)	C12—O2—C11—C10	-9.0 (5)
C4—C5—C6—C9	-178.8 (4)	N—C10—C11—O2	8.4 (4)
C5—C6—C7—C8	1.1 (6)	C9—C10—C11—O2	-113.4 (4)
C9—C6—C7—C8	179.5 (4)	C10—N—C12—O3	-179.6 (4)
C6—C7—C8—C3	-1.1 (7)	C10—N—C12—O2	0.3 (5)
C4—C3—C8—C7	0.4 (6)	C11—O2—C12—O3	-174.4 (4)
O1—C3—C8—C7	-179.7 (4)	C11—O2—C12—N	5.7 (5)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N—H0A \cdots O3 ⁱ	0.86	1.99	2.845 (4)	171
C10—H10A \cdots O3 ⁱⁱ	0.98	2.47	3.317 (5)	144

Symmetry codes: (i) $x-1/2, -y+3/2, -z+2$; (ii) $x-1, y, z$.