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

**Hong-Yong Wang, Min-Hao Xie,* Shi-Neng Luo, Yong-Jun He and Pei Zou**

Jiangsu Institute of Nuclear Medicine, Wuxi 214063, People's Republic of China
Correspondence e-mail: wywhy007@yahoo.com.cn

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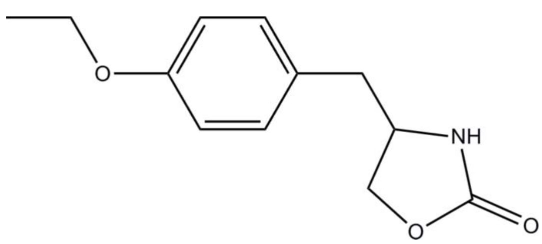
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.043; $wR$ factor = 0.150; data-to-parameter ratio = 8.5.

In the title compound, $C_{12}H_{15}NO_3$, the ethoxybenzyl ring plane forms a dihedral angle of 60.3 (4)° with the mean plane of the oxazolidine ring. The molecules are linked through N—H···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*

$C_{12}H_{15}NO_3$
$M_r$ = 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) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 K
0.30 × 0.20 × 0.10 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (*CAD-4 Software*; Enraf–Nonius, 1989)
$T_{min}$ = 0.973, $T_{max}$ = 0.991
2427 measured reflections
1246 independent reflections
904 reflections with $I > 2\sigma(I)$
$R_{int}$ = 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···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N—H0$A$···O3$^{i}$ | 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).

## References

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## supplementary materials

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

**H.-Y. Wang, M.-H. Xie, S.-N. Luo, Y.-J. He** and **P. Zou**

### 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.

### 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 wash 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 $NaHCO_3$ 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 crystalized by slow evaporation of a solution in methanol.

### 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, mehtylene and methine H atoms, respectively, and $U_{iso}$(H) = 1.5$U_{eq}$(methyl) and 1.2$U_{eq}$(the rest) parent atoms. An absolute configuration could not be established by anomalous dispersion effects. Therefore, Fridel pairs (846) were merged.

### Figures

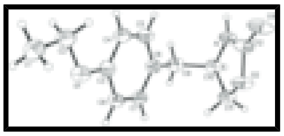

Fig. 1. A view of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

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

*Crystal data*

| | |
|---|---|
| $C_{12}H_{15}NO_3$ | $F_{000}$ = 472 |
| $M_r$ = 221.25 | $D_x$ = 1.264 Mg m$^{-3}$ |
| Orthorhombic, $P2_12_12_1$ | Mo $K\alpha$ radiation<br>$\lambda$ = 0.71073 Å |
| Hall symbol: P 2ac 2ab | Cell parameters from 25 reflections |
| $a$ = 5.7960 (12) Å | $\theta$ = 9–12º |
| $b$ = 9.924 (2) Å | $\mu$ = 0.09 mm$^{-1}$ |
| $c$ = 20.209 (4) Å | $T$ = 293 K |
| $V$ = 1162.4 (4) Å$^3$ | Needle, colourless |
| $Z$ = 4 | 0.30 × 0.20 × 0.10 mm |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4<br>diffractometer | $R_{int}$ = 0.043 |
| Radiation source: fine-focus sealed tube | $\theta_{max}$ = 25.3º |
| Monochromator: graphite | $\theta_{min}$ = 2.0º |
| $T$ = 293 K | $h$ = 0→6 |
| $\omega/2\theta$ scans | $k$ = 0→11 |
| Absorption correction: $\psi$ scan<br>(CAD-4 Software; Enraf–Nonius, 1989) | $l$ = −24→24 |
| $T_{min}$ = 0.973, $T_{max}$ = 0.991 | 3 standard reflections |
| 2427 measured reflections | every 200 reflections |
| 1246 independent reflections | intensity decay: 1% |
| 904 reflections with $I > 2\sigma(I)$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)]$ = 0.043 | $w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 0.28P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2)$ = 0.150 | $(\Delta/\sigma)_{max}$ < 0.001 |
| $S$ = 1.01 | $\Delta\rho_{max}$ = 0.17 e Å$^{-3}$ |
| 1246 reflections | $\Delta\rho_{min}$ = −0.19 e Å$^{-3}$ |

| | |
|---|---|
| 146 parameters | Extinction correction: SHELXL97 (Sheldrick, 2008) |
| Primary atom site location: structure-invariant direct methods | Extinction coefficient: 0.031 (6) |
| Secondary atom site location: difference Fourier map | |

*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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N—H0A···O3$^{i}$ | 0.86 | 1.99 | 2.845 (4) | 171 |
| C10—H10A···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*.

**Fig. 1**