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(4*RS*,5*RS*)-4-Benzyl-5-(4-tolylthiocarbonyl)-oxazolidin-2-one

William Clegg* and Mark R. J. Elsegood†

Department of Chemistry, University of Newcastle upon Tyne, Newcastle upon Tyne NE1 7RU, England

† Current address: Chemistry Department, Loughborough University, Loughborough Leicestershire LE11 3TU, England

Correspondence e-mail: w.clegg@ncl.ac.uk

Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.005 Å
R factor = 0.073
wR factor = 0.145
Data-to-parameter ratio = 15.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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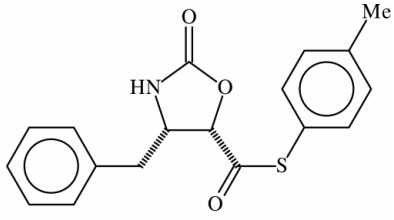
The title compound, $C_{18}H_{17}NO_3S$, is the product of an epoxidation reaction, followed by cyclization and hydrolysis of the resulting epoxide. There are two independent molecules in the asymmetric unit; one has a slightly twisted heterocyclic ring, and the ring in the other is almost planar. Molecules form centrosymmetric pairs through N—H···O=C hydrogen bonds, involving the carbonyl group in the ring.

Comment

The title compound, (I), was synthesized as part of a study of the stereocontrolled synthesis of *anti*-α-hydroxy-β-amino acid derivatives (Ambroise *et al.*, 2002). Epoxidation of 1-arylthio-1-nitroalkenes was expected to give the corresponding epoxides, but instead oxazolidinones, such as (I), were isolated, presumably because of cyclization and hydrolysis during the work-up of the reaction products.

The crystal structure of (I) was determined in order to confirm the relative stereochemistry of the substituents on the heterocyclic ring; this was found to be *cis*. The structure reported here is racemic, as was the starting material. The same reaction can, however, be carried out with a single enantiomer as starting material, in which case it is highly stereoselective (Ambroise *et al.*, 2002), as has also been found also crystallographically for the corresponding compound with methyl instead of benzyl (Clegg & Elsegood, 2002).



(I)

There are two molecules in the asymmetric unit (Fig. 1). For both of them, the bond lengths and angles of the heterocyclic ring are typical of those found in 39 related compounds in the April 2002 release of the Cambridge Structural Database (Allen & Kennard, 1993). In one molecule, the ring has a slight twist, as indicated by the torsion angles in Table 1; C2 lies 0.091 Å on one side and C3 0.198 Å on the other side of the plane defined by the N—C—O segment of the ring. In the other molecule, the ring is more nearly planar, with corresponding deviations of 0.041 and 0.089 Å, both on the same side of the N—C—O plane, for C20 and C21, respectively, and with smaller torsion angles for the ring atoms.

N—H···O=C hydrogen bonds link molecules together in centrosymmetric pairs (Table 2).

Experimental

The synthesis of the title compound is described by Ambroise *et al.* (2002).

Crystal data

$C_{18}H_{17}NO_3S$
M_r = 327.39
Monoclinic, $P2_1/c$
a = 22.696 (3) Å
b = 6.2094 (8) Å
c = 24.555 (3) Å
β = 106.205 (3)°
V = 3323.0 (7) Å^3
Z = 8
D_x = 1.309 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4377 reflections
θ = 1.7–24.6°
μ = 0.21 mm^{-1}
T = 160 (2) K
Plate, colourless
0.56 × 0.14 × 0.07 mm

Data collection

Siemens SMART 1K CCD diffractometer
ω rotation with narrow frames
Absorption correction: multi-scan (*XPREP* in *SHELXTL*; Sheldrick, 1994)
T_{min} = 0.844, T_{max} = 0.936
16452 measured reflections
6588 independent reflections
3940 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 26.3°
$h = -27 \rightarrow 21$
$k = -7 \rightarrow 7$
$l = -29 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.145
S = 1.12
6588 reflections
424 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0292P)^2 + 4.1967P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0014 (2)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.214 (4)	O4—C19	1.223 (4)
C1—O2	1.379 (4)	C19—O5	1.370 (4)
C1—N1	1.327 (5)	C19—N2	1.317 (4)
O2—C2	1.441 (4)	O5—C20	1.441 (4)
C2—C3	1.551 (5)	C20—C21	1.564 (4)
C3—N1	1.452 (5)	C21—N2	1.452 (4)
O1—C1—O2	120.0 (4)	O4—C19—O5	119.3 (3)
O1—C1—N1	130.5 (4)	O4—C19—N2	130.7 (3)
O2—C1—N1	109.4 (3)	O5—C19—N2	109.9 (3)
C1—O2—C2	109.2 (3)	C19—O5—C20	109.5 (3)
O2—C2—C3	105.0 (3)	O5—C20—C21	106.0 (2)
C2—C3—N1	99.8 (3)	C20—C21—N2	99.5 (3)
C1—N1—C3	113.6 (3)	C19—N2—C21	115.0 (3)
N1—C1—O2—C2	3.8 (4)	N2—C19—O5—C20	1.7 (4)
C1—O2—C2—C3	−13.4 (4)	C19—O5—C20—C21	0.8 (3)
O2—C2—C3—N1	16.7 (4)	O5—C20—C21—N2	−2.7 (3)
O2—C1—N1—C3	8.5 (4)	O5—C19—N2—C21	−3.9 (4)
C2—C3—N1—C1	−15.8 (4)	C20—C21—N2—C19	4.0 (4)

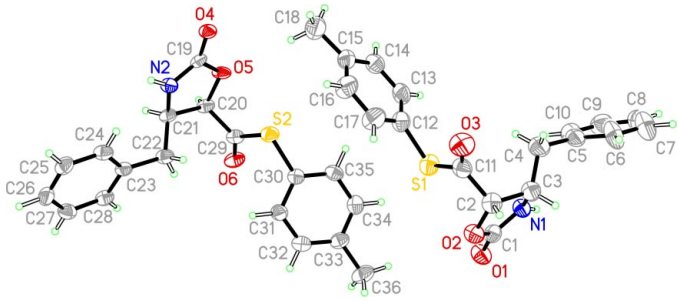


Figure 1
The asymmetric unit of (I), with atom labels and 50% probability ellipsoids for non-H atoms.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.79 (4)	2.12 (4)	2.900 (4)	168 (4)
N2—H2···O4^{ii}	0.78 (3)	2.09 (3)	2.853 (3)	167 (4)

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

H atoms attached to C atoms were placed geometrically and refined with a riding model (including free rotation about C—methyl bonds), and with U_{iso} constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the carrier atom. The positional parameters of the H atom on the oxazolidine N atom were refined freely. The small crystal size leads to weak data and relatively high residual factors.

Data collection: *SMART* (Siemens, 1995); cell refinement: local programs; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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