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(4*S*,5*S*)-4-Methyl-5-(4-tolylthiocarbonyl)-oxazolidin-2-one

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The title compound, $C_{12}H_{13}NO_3S$, is the product of a stereoselective epoxidation reaction, followed by cyclization and hydrolysis of the resulting epoxide. The heterocyclic ring is slightly twisted. Molecules are linked into a chain through intermolecular N—H···O=C hydrogen-bonding interactions.

Key indicators

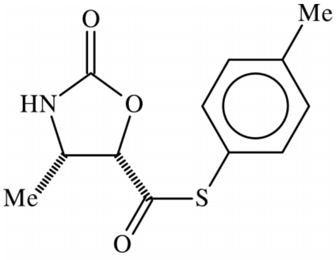
Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.003 Å
R factor = 0.027
wR factor = 0.068
Data-to-parameter ratio = 10.7

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

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 product of the reaction, like the starting material, is enantiomerically pure. This is also indicated by NMR spectroscopy, and demonstrates that the reaction is highly stereoselective.



(I)

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). The ring has a slight twist, as indicated by the torsion angles in Table 1; C2 lies 0.232 Å on one side and C3 0.040 Å on the other side of the plane defined by the N—C—O segment of the ring. This gives a conformation intermediate between twist and envelope (with C2 as the flap angle). The molecular structure is shown in Fig. 1.

An intermolecular hydrogen bond (Table 2) between the N—H and carbonyl groups links molecules into a chain parallel to the *b* axis.

Experimental

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

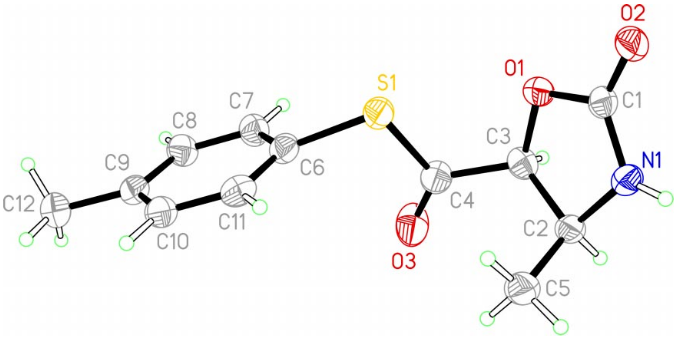


Figure 1
The molecular structure of (I), with atom labels and 50% probability ellipsoids for non-H atoms.

Crystal data

$C_{12}H_{13}NO_3S$
$M_r = 251.29$
Orthorhombic, $P2_12_12_1$
$a = 5.5614$ (7) Å
$b = 7.8100$ (10) Å
$c = 27.520$ (5) Å
$V = 1195.3$ (3) Å^3
$Z = 4$
$D_x = 1.396$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 297 reflections
$\theta = 1.0$–$23.3°$
$\mu = 0.27$ mm^{-1}
$T = 160$ (2) K
Block, colourless
0.80 × 0.64 × 0.32 mm

Data collection

Siemens SMART 1K CCD diffractometer
ω rotation with narrow frames
Absorption correction: multi-scan (*XPREP* in *SHELXTL*; Sheldrick, 1994)
$T_{\min} = 0.816$, $T_{\max} = 0.891$
4745 measured reflections
1718 independent reflections
1714 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 23.2°$
$h = -6 \rightarrow 5$
$k = -8 \rightarrow 8$
$l = -30 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.068$
$S = 1.11$
1718 reflections
160 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0296P)^2 + 0.3596P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.13$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.019 (2)
Absolute structure: Flack (1983), 669 Friedel pairs
Flack parameter = −0.02 (8)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.362 (2)	C1—O2	1.214 (2)
O1—C3	1.445 (2)	N1—C2	1.461 (3)
C1—N1	1.330 (3)	C2—C3	1.549 (3)
C1—O1—C3	109.51 (15)	C1—N1—C2	113.50 (17)
O1—C1—N1	109.61 (17)	N1—C2—C3	99.26 (15)
O1—C1—O2	120.73 (17)	O1—C3—C2	105.30 (15)
N1—C1—O2	129.67 (19)		
C3—O1—C1—N1	−1.7 (2)	C1—O1—C3—C2	11.49 (19)
O1—C1—N1—C2	−10.0 (2)	N1—C2—C3—O1	−15.57 (18)
C1—N1—C2—C3	15.9 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2$^{\rm i}$	0.81 (2)	2.07 (2)	2.859 (2)	163 (2)

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

H atoms attached to C atoms were placed geometrically and refined with a riding model (including free rotation about C—methyl bonds), and with $U_{\rm iso}$ constrained to be 1.2 (1.5 for methyl groups) times $U_{\rm eq}$ of the carrier atom. The positional parameters of the H atom on the oxazolidine N atom were refined freely. The relatively low $\theta_{\max}$ value is due to a short crystal-to-detector distance for this data collection, which was carried out before parameters were optimized for one of the first commercial CCD detector systems.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; 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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References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Ambroise, L., Dumez, E., Szeki, A. & Jackson, R. F. W. (2002). *Synthesis*. In the press.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (1994). *SHELXTL*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1995). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.