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

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Key indicators

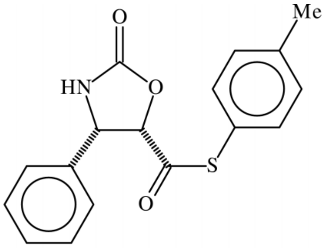
Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.108
Data-to-parameter ratio = 11.6

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

The title compound, $C_{17}H_{15}NO_3S$, 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, both with a slightly twisted heterocyclic ring. N—H···O═C hydrogen bonds link the molecules together into centrosymmetric sets of four.

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.



(I)

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 phenyl (Clegg & Elsegood, 2002).

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). The torsion angles for the ring atoms (Table 1) and the deviations of saturated ring C atoms from the planes of the N—C—O units (C2 −0.140, C3 0.064 Å; C19 0.169, C20 −0.041 Å) indicate a conformation intermediate between twist and envelope, the two rings having very similar conformations.

Hydrogen-bonding interactions for the two independent molecules are different (Table 2). In each molecule, N—H acts as a donor. However, it is the carbonyl atom O1 that acts as

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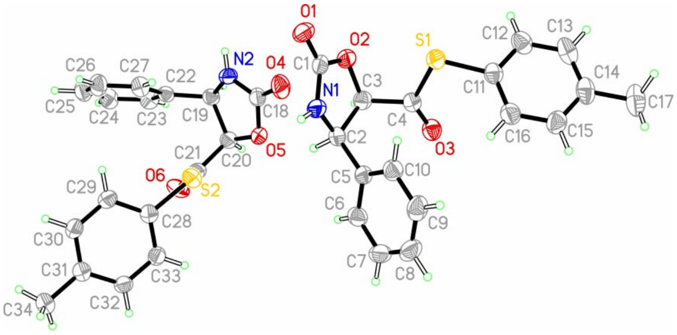

Figure 1
The asymmetric unit of (I), with atom labels and 50% probability ellipsoids for non-H atoms.

acceptor in both cases; the other carbonyl O atoms are not involved in any hydrogen bonding. Thus, the molecules are linked together into centrosymmetric groups of four, with four hydrogen bonds (Fig. 2).

Experimental

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

Crystal data

$C_{17}H_{15}NO_3S$
$M_r = 313.36$
Triclinic, $P\overline{1}$
$a = 8.931$ (3) Å
$b = 12.854$ (5) Å
$c = 14.884$ (4) Å
$\alpha = 75.067$ (18)°
$\beta = 72.757$ (14)°
$\gamma = 75.547$ (15)°
$V = 1548.7$ (9) Å^3
$Z = 4$
$D_x = 1.344$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 68 reflections
$\theta = 1.5$–23.0°
$\mu = 0.22$ mm^{-1}
$T = 160$ (2) K
Block, colourless
0.78 × 0.74 × 0.70 mm

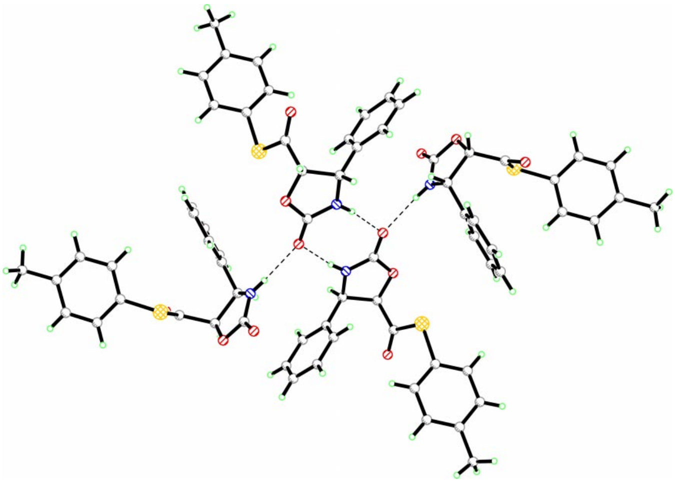

Figure 2
Hydrogen bonds (dashed) for a centrosymmetric group of four molecules (the contents of one unit cell).

Data collection

Siemens SMART 1K CCD diffractometer
ω rotation with narrow frames
Absorption correction: multi-scan (*XPREP* in *SHELXTL*; Sheldrick, 1994)
$T_{\min} = 0.814$, $T_{\max} = 0.950$
6481 measured reflections
4723 independent reflections
4468 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$
$\theta_{\max} = 25.2°$
$h = -7 \rightarrow 10$
$k = -15 \rightarrow 12$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.108$
$S = 1.05$
4723 reflections
406 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 0.8478P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.65$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0111 (15)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.226 (2)	O4—C18	1.207 (2)
C1—N1	1.323 (3)	C18—N2	1.335 (3)
C1—O2	1.353 (2)	C18—O5	1.370 (2)
N1—C2	1.451 (3)	N2—C19	1.449 (3)
C2—C3	1.569 (3)	C19—C20	1.568 (3)
C3—O2	1.446 (2)	C20—O5	1.431 (2)
O1—C1—N1	128.36 (19)	O4—C18—N2	129.9 (2)
O1—C1—O2	121.18 (18)	O4—C18—O5	121.13 (18)
N1—C1—O2	110.45 (17)	N2—C18—O5	108.94 (17)
C1—N1—C2	114.33 (18)	C18—N2—H2	121.5 (17)
N1—C2—C3	99.05 (15)	N2—C19—C20	99.39 (15)
C2—C3—O2	105.35 (15)	C19—C20—O5	105.26 (15)
C1—O2—C3	109.34 (15)	C18—O5—C20	110.33 (14)
O2—C1—N1—C2	−6.1 (2)	O5—C18—N2—C19	−7.3 (2)
C1—N1—C2—C3	11.0 (2)	C18—N2—C19—C20	12.0 (2)
N1—C2—C3—O2	−11.54 (17)	N2—C19—C20—O5	−11.96 (18)
N1—C1—O2—C3	−2.7 (2)	N2—C18—O5—C20	−1.8 (2)
C2—C3—O2—C1	9.32 (19)	C19—C20—O5—C18	9.0 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1$^{\rm i}$	0.80 (3)	2.08 (3)	2.874 (3)	167 (2)
N2—H2···O1$^{\rm ii}$	0.82 (3)	2.06 (3)	2.835 (2)	159 (2)

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

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 largest residual electron-density peak is about three times the size of the next largest. It lies 0.65 Å from an inversion centre, but the pair of symmetry-related positions can not be interpreted sensibly in terms of a partially occupied disordered site for any solvent molecule.

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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