ORGANIC COMPOUNDS

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(2*R*,3*S*)-3-Phenyl-2-[(2*S*,4*S*)-4-phenyl-3-tosyl-1,3-oxazolidin-2-yl]cyclopentanone

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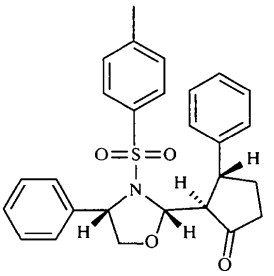
Abstract

The stereochemical parameters of the title compound, $C_{27}H_{27}NO_4S$, are in good agreement with other arylsulfonyloxazolidines. Whereas the oxazolidine ring displays a twist conformation, the cyclopentanone ring has an envelope conformation.

Comment

The acid-catalysed condensation of 2-(*N*-tosyl)-1-alkanoles with aldehydes usually produces the thermodynamically more stable 2,4-*cis*-substituted 1,3-oxazolidines (Hoppe *et al.*, 1991). From the reaction of (*S*)-*N*-tosylphenylglycinol and 2-(hydroxymethylene)-cyclopentanone, we isolated traces of a by-product which turned out to have the *trans* configuration (Bolte & Berger, 1996). However, the reaction of (*S*)-*N*-tosylphenylglycinol and 2-(hydroxymethylene)-4-phenylcyclopentanone afforded the expected *cis* product, (I). The oxazolidine ring adopts a twist conformation [q_2 = 0.275 (3) Å and φ_2 = 121.1 (6)°; Cremer & Pople,



(I)

1975], with C4 and C5 deviating by −0.277 (7) and 0.185 (8) Å, respectively, from the plane of the remaining three atoms. The cyclopentanone ring exhibits an envelope conformation [q_2 = 0.404 (3) Å and φ_2 = 322.3 (4)°], with C25 deviating by 0.621 (4) Å from the plane of the remaining four atoms. The geometric parameters of the sulfonamide moiety agree well with those found by Herbst-Irmer (1990).

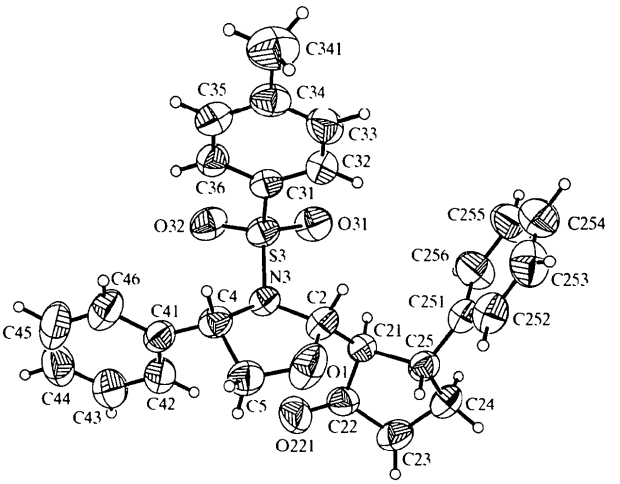


Fig. 1. Perspective view of the title compound with the atom-numbering scheme; displacement ellipsoids are at the 50% probability level.

Experimental

Single crystals of the title compound were obtained from a diethyl ether solution by slow evaporation of the solvent.

Crystal data

$C_{27}H_{27}NO_4S$
M_r = 461.56
Monoclinic
$P2_1$
a = 9.783 (2) Å
b = 11.910 (1) Å
c = 10.395 (2) Å
β = 99.11 (1)°
V = 1195.9 (4) Å^3
Z = 2
D_x = 1.282 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 30–40°
μ = 1.472 mm^{-1}
T = 293 (2) K
Transparent block
0.40 × 0.40 × 0.20 mm
Colourless

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
ω scans
Absorption correction: empirical, measuring nine ψ scans (North *et al.*, 1968)
T_{min} = 0.702, T_{max} = 0.745
3009 measured reflections
2837 independent reflections
2813 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 59.90°
h = −10 → 10
k = −13 → 6
l = −11 → 0
3 standard reflections
frequency: 92 min
intensity decay: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.086
S = 1.071
Extinction correction: *SHELXL*96
Extinction coefficient: 0.0102 (6)

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2837 reflections
300 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2 + 0.2002P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.319$ e Å^{-3}
$\Delta\rho_{min} = -0.224$ e Å^{-3}
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = −0.004 (17)

Table 1. *Selected geometric parameters (Å, °)*

O1—C5	1.394 (4)	C4—C5	1.525 (3)
O1—C2	1.396 (3)	S3—O32	1.4264 (19)
C2—N3	1.497 (3)	S3—O31	1.427 (2)
N3—C4	1.491 (3)	S3—C31	1.762 (3)
N3—S3	1.6441 (19)		
C5—O1—C2	110.7 (2)	O32—S3—O31	120.33 (12)
O1—C2—N3	106.0 (2)	O32—S3—N3	106.30 (10)
C4—N3—C2	107.67 (18)	O31—S3—N3	106.04 (12)
C4—N3—S3	116.96 (16)	O32—S3—C31	107.92 (12)
C2—N3—S3	116.36 (16)	O31—S3—C31	107.91 (12)
N3—C4—C5	100.2 (2)	N3—S3—C31	107.77 (10)
O1—C5—C4	107.1 (2)		
C5—O1—C2—N3	8.2 (3)	C25—C21—C22—C23	−23.5 (3)
O1—C2—N3—C4	11.2 (3)	C21—C22—C23—C24	−1.1 (3)
C2—N3—C4—C5	−23.7 (3)	C2—C21—C25—C24	165.9 (2)
C2—O1—C5—C4	−24.1 (4)	C22—C21—C25—C24	38.3 (2)
N3—C4—C5—O1	28.9 (3)	C23—C24—C25—C21	−39.9 (3)

The structure was solved by extracting the position of the S atom from a sharpened Patterson list and extending the structure with a tangent expansion using *SHELXS*96 (Sheldrick, 1990), and then refined with *SHELXL*96 (Sheldrick, 1996) by full-matrix least-squares methods. All H atoms were located by difference Fourier syntheses and refined with fixed individual displacement parameters [U(H) = 1.5U_{eq}(C_{methyl}) or U(H) = 1.2U_{eq}(C)], using a riding model with C—H(tertiary) = 0.98, C—H(secondary) = 0.97, C—H(methyl) = 0.96 or C—H(aromatic) = 0.93 Å.

Data collection: *SDP* (Enraf–Nonius, 1985). Cell refinement: *SDP*. Data reduction: *SDP*. Molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1271). Services for accessing these data are described at the back of the journal.

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2,3,4,5-Tetrahydrocyclohepta[*b*]pyrido-[2,1-*a*]benzimidazole-7-carbonitrile

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Abstract

The title compound, $C_{17}H_{15}N_3$, is a tetracyclic system in which the atoms of the three unsaturated rings are coplanar to within 0.026 Å. The molecules are stacked in pairs about inversion centres, with the shortest C···C separation being 3.386 (4) Å.

Comment

The imidazole and imidazo[1,2-*a*]pyridine ring systems are components of heterocycles that have shown anti-tumor activity (Elgemeie *et al.*, 1996). Our interest in the development of novel non-classical antimetabolites directed us towards the synthesis of new tetracyclic antimetabolites involving the imidazo[1,2-*a*]pyridine system (Elgemeie & Hussain, 1994; Elgemeie *et al.*, 1994). A single-step synthesis of the title derivative (6) was anticipated *via* the cyclocondensation of 2-cyanobenzimidazole and the sodium salt of 2-(hydroxymethylene)-1-cycloheptanone. However, this reaction could give three other possible regioisomers [structures (3)–(5)]. The direction of ring closure in such cyclocondensations is difficult to predict, and spectroscopic data cannot differentiate between these structures. In order to establish unambiguously the structure of the product, the crystal structure was determined.