C4—C9	1.362 (8)	C4*A*—N5*A*	1.330 (7)
C4—N5	1.425 (7)	C4*A*—C9*A*	1.371 (8)
N5—C6	1.324 (8)	N5*A*—C6*A*	1.411 (8)
C6—O61	1.252 (7)	C6*A*—O61*A*	1.191 (7)
C6—N7	1.409 (8)	C6*A*—N7*A*	1.415 (8)
N7—C8	1.432 (7)	N7*A*—C8*A*	1.403 (8)
C8—O81	1.209 (7)	C8*A*—O81*A*	1.225 (7)
C8—C9	1.415 (8)	C8*A*—C9*A*	1.430 (9)

C25—C21—C22—C23	22.1 (8)
C21—C22—C23—O23	−174.9 (12)
C21—C22—C23—C24	−2.2 (9)
O23—C23—C24—C25	153.4 (11)
C22—C23—C24—C25	−19.8 (9)
C23—C24—C25—C21	32.3 (8)
C22—C21—C25—C24	−32.5 (7)
C25*A*—C21*A*—C22*A*—C23*A*	7.7 (11)
C21*A*—C22*A*—C23*A*—O23*A*	−167.4 (13)
C21*A*—C22*A*—C23*A*—C24*A*	7.8 (13)
O23*A*—C23*A*—C24*A*—C25*A*	153.9 (11)
C22*A*—C23*A*—C24*A*—C25*A*	−21.8 (13)
C23*A*—C24*A*—C25*A*—C21*A*	25.0 (10)
C22*A*—C21*A*—C25*A*—C24*A*	−19.2 (8)

H atoms were refined using a riding model, with isotropic displacement parameters $U(H) = 1.5U_{eq}$(methyl C) and $U(H) = 1.2U_{eq}(C)$, and aromatic C—H = 0.93, tertiary C—H = 0.98, methylene C—H = 0.97 and methyl C—H = 0.96 Å.

Data collection: *SDP* (Enraf–Nonius, 1985). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

The author thanks Dr H. Koeppen (Boehringer Ingelheim) for providing the sample.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: CF1077). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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An Arylsulfonyloxazolidine

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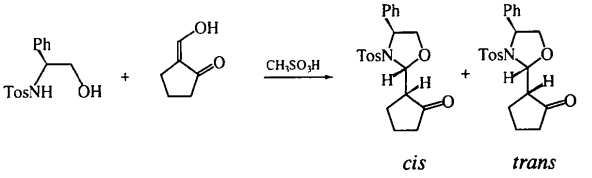
Abstract

The geometric parameters of the title compound, (2*S*)-2-[(2*R*,4*S*)-4-phenyl-3-tosyl-1,3-oxazolidin-2-yl]cyclopentanone, $C_{21}H_{23}NO_4S$, are in good agreement with other arylsulfonyl-oxazolidines. While the oxazolidine ring displays an envelope conformation, the cyclopentanone ring has a twist conformation.

Comment

The acid-catalysed condensation of 2-(*N*-tosyl)-1-alkanols with aldehydes usually produces the thermodynamically more stable 2,4-*cis*-substituted 1,3-oxazolidines (Hoppe, Hoffmann, Gärtner, Krettek & Hoppe, 1991).

From the reaction of (*S*)-*N*-tosyl-phenylglycinol and 2-(hydroxymethylene)cyclopentanone, we isolated traces of a side product which was subjected to X-ray structure analysis and turned out to have the *trans*-configuration (Fig. 1).



cis *trans*

The oxazolidine ring in the *trans* configuration adopts an envelope conformation [q_2 = 0.375 (2)Å, φ_2 = 328.6 (3)° (Cremer & Pople, 1975)] with C5 deviating by 0.570 (4) Å from the plane of the remaining four atoms, the cyclopentanone ring exhibits a twist configuration [q_2 = 0.349 (3)Å, φ_2 = 126.3 (4)°] with C24 and C25 deviating by −0.284 (6) and 0.288 (6) Å, respectively, from the plane of the remaining three atoms. The geometric parameters of the sulfonamide moiety agree well with those found by Herbst-Irmer (1990). The nitrogen N3 is nearly planar (sum of the bond angles: 356.5°). While one of the sulfonyl O atoms, O32, is nearly coplanar with the tolyl substituent [O32—S3—C31—C32 30.0 (2)°], the other, O31, forms a torsion angle of −178.6 (1)° with C2. The two aromatic substituents enclose an angle of 19.8 (1)°.

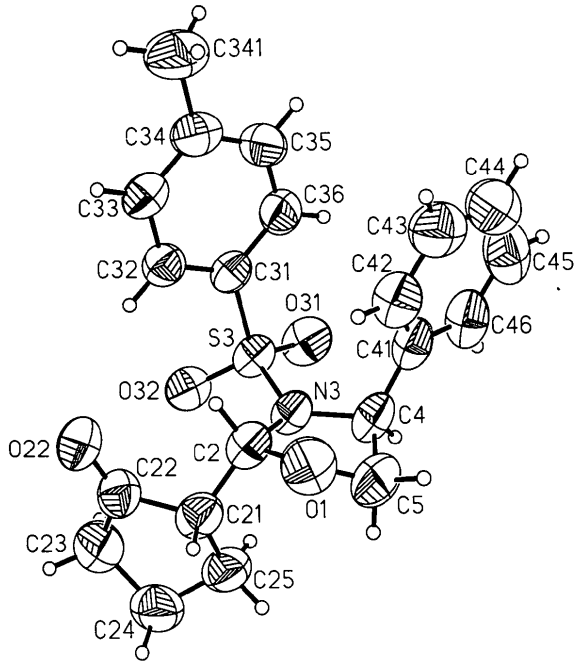


Fig. 1. Perspective view of the title compound with the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

Experimental

Single crystals were obtained from a diethyl ether solution.

Crystal data

$C_{21}H_{23}NO_4S$
M_r = 385.46
Orthorhombic
$P2_12_12_1$
a = 8.704 (1) Å
b = 12.636 (1) Å
c = 17.819 (2) Å
V = 1959.8 (4) Å^3
Z = 4
D_x = 1.306 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 30.00–35.00°
μ = 1.686 mm^{-1}
T = 293 K
Block
0.60 × 0.30 × 0.25 mm
Colourless transparent

Data collection

Enraf–Nonius CAD-4 four-circle-diffractometer
ω scans
Absorption correction: none
9208 measured reflections
2901 independent reflections
2834 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0262
θ_{max} = 59.91°
$h = -9 \rightarrow 9$
$k = -14 \rightarrow 3$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 92 min
intensity decay: 0.80%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0286
$wR(F^2)$ = 0.0815
S = 1.018
2901 reflections
246 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.0925P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.162
$\Delta\rho_{max}$ = 0.164 e Å^{-3}
$\Delta\rho_{min}$ = −0.157 e Å^{-3}
Extinction correction: *SHELXL*96 (Sheldrick, 1996)
Extinction coefficient: 0.0053 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.009 (14)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
O1	0.26660 (19)	0.60047 (14)	0.60343 (9)	0.0918 (5)
C2	0.2760 (2)	0.52268 (14)	0.66019 (10)	0.0650 (5)
N3	0.42698 (18)	0.54534 (11)	0.69568 (8)	0.0603 (4)
C4	0.4989 (3)	0.64052 (14)	0.66180 (12)	0.0735 (5)
C5	0.3552 (3)	0.68911 (18)	0.62743 (18)	0.1014 (8)
C21	0.1386 (2)	0.52650 (14)	0.71284 (12)	0.0696 (5)
C22	0.0984 (2)	0.42071 (16)	0.74916 (12)	0.0742 (5)
O22	0.10474 (19)	0.33658 (11)	0.71803 (10)	0.0910 (5)
C23	0.0428 (3)	0.4416 (2)	0.82746 (14)	0.0944 (7)
C24	0.0198 (3)	0.56023 (19)	0.83150 (15)	0.0944 (7)
C25	0.1365 (3)	0.60535 (16)	0.77656 (15)	0.0860 (6)
S3	0.52127 (5)	0.45522 (3)	0.74082 (2)	0.05931 (16)
O32	0.41634 (17)	0.40684 (10)	0.79184 (6)	0.0694 (3)
O31	0.65706 (17)	0.50515 (11)	0.76839 (8)	0.0800 (4)
C31	0.5735 (2)	0.35723 (13)	0.67498 (9)	0.0583 (4)
C32	0.4709 (2)	0.27824 (13)	0.65695 (10)	0.0621 (4)
C33	0.5093 (2)	0.20458 (14)	0.60247 (11)	0.0708 (5)
C34	0.6498 (3)	0.20900 (16)	0.56601 (11)	0.0751 (5)
C341	0.6891 (4)	0.1293 (2)	0.50597 (15)	0.1135 (9)
C35	0.7508 (3)	0.28712 (16)	0.58626 (12)	0.0775 (5)
C36	0.7167 (2)	0.36176 (15)	0.64006 (11)	0.0706 (5)
C41	0.6257 (2)	0.62108 (14)	0.60591 (12)	0.0694 (5)
C42	0.6043 (3)	0.56354 (18)	0.54043 (12)	0.0827 (6)
C43	0.7237 (3)	0.5492 (2)	0.48962 (14)	0.0969 (7)
C44	0.8639 (4)	0.5936 (2)	0.50333 (16)	0.1036 (8)
C45	0.8871 (3)	0.6513 (2)	0.56696 (18)	0.1077 (9)
C46	0.7685 (3)	0.66572 (18)	0.61910 (15)	0.0865 (7)

Table 2. *Selected geometric parameters (Å, °)*

O1—C2	1.413 (2)	C4—C5	1.522 (3)
O1—C5	1.426 (3)	S3—O31	1.4270 (14)
C2—N3	1.486 (2)	S3—O32	1.4263 (14)
N3—C4	1.484 (2)	S3—C31	1.7653 (17)
N3—S3	1.6179 (14)		
C2—O1—C5	107.49 (16)	O31—S3—O32	120.02 (9)
O1—C2—N3	102.81 (15)	O31—S3—N3	106.27 (8)
C4—N3—C2	110.86 (14)	O32—S3—N3	107.08 (8)
C4—N3—S3	123.96 (13)	O31—S3—C31	108.99 (9)
C2—N3—S3	121.63 (12)	O32—S3—C31	106.74 (8)
N3—C4—C5	98.28 (17)	N3—S3—C31	107.10 (8)
O1—C5—C4	104.38 (16)		
C5—O1—C2—N3	−27.6 (2)	C25—C21—C22—C23	11.4 (2)
O1—C2—N3—C4	3.47 (19)	C21—C22—C23—C24	11.2 (2)
C2—N3—C4—C5	19.6 (2)	C22—C23—C24—C25	−29.4 (3)
C2—O1—C5—C4	41.6 (3)	C22—C21—C25—C24	−29.3 (2)
N3—C4—C5—O1	−35.7 (2)	C23—C24—C25—C21	36.7 (2)

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*86 (Sheldrick, 1985) and 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(aromatic)

= 0.93, C—H(methyl) = 0.96, C—H(secondary) = 0.97 or C—H(tertiary) = 0.98 Å, respectively. The methyl group was allowed to rotate abouts its local threefold axis.

Data collection: *SDP* (Enraf–Nonius, 1985). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991).

We thank Professor Dr D. Hoppe (University of Münster, Germany) for providing the sample.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SK1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Product of Catalysed Diboration of Bis(4-methoxyphenyl)ethyne by Bis-(pinacolato-*O*,*O*′)diboron

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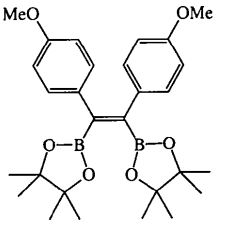
Abstract

The title compound, (*Z*)-1,2-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1,2-bis(4-methoxyphenyl)-ethene, $C_{28}H_{38}B_2O_6$, has a *cis* arrangement of two boronate ester substituents and two 4-methoxyphenyl substituents on its central C=C double bond, which shows a slight twist of *ca* 8.5°. All four substituents are rotated considerably out of the alkene plane to reduce steric hindrance. The BO_2C_2 five-membered rings have a twist conformation.

Comment

We and others have recently reported the catalysed addition of B—B bonds to alkenes (Baker, Nguyen, Marder & Westcott, 1995) and alkynes (Ishiyama, Matsuda, Miyaura & Suzuki, 1993; Iverson & Smith, 1995; Lesley *et al.*, 1996), and have prepared and structurally characterized several platinum(II)–bis(phosphine)–bis(boryl) complexes formed by oxidative addition of the B—B bonds to bisphosphine Pt(0) centres. Initial studies (Ishiyama *et al.*, 1993) of the addition of bis-(pinacolato-*O*,*O*′)diboron, B_2pin_2, to alkynes suggested that this results in *cis*-alkene-1,2-bis(boronate esters) on the basis of NMR measurements, and we have confirmed this stereochemistry by X-ray crystallography for the addition of bis(benzene-1,2-diolato)diboron [or bis-(catecholato-*O*,*O*′)diboron, B_2cat_2] to internal and terminal alkynes as well as to 1,3-diynes (Lesley *et al.*, 1996). We also found that reactions using B_2pin_2 were slower than those using B_2cat_2.



(I)

We report here the structure of the product (I) of the addition of B_2pin_2 to the symmetrical internal alkyne bis(4-methoxyphenyl)ethyne, which confirms that this addition also gives *cis*-boronate ester substituents (Fig. 1.) The geometry of the central C_4B_2 part of the molecule is essentially the same as for the B_2cat_2 adducts. There is a small twist about the C=C double bond, as measured by the C—C—C—C and B—C—C—B torsion angles of 8.0 (2) and 9.0 (2)°, respectively. The central C_4B_2 mean plane [r.m.s. deviation 0.067 (2) Å] has dihedral angles with the two aromatic rings [r.m.s. deviations 0.006 (2) and 0.005 (2) Å] of 44.6 (1) and 51.4 (1)°, and with the two CBO_2 boron coordination mean planes [r.m.s. deviations 0.027 (2) and 0.006 (2) Å] of 66.0 (1) and 22.1 (1)°, to avoid steric congestion.

The two BO_2C_2 five-membered boronate ester rings have very similar twist conformations, each with one C atom on one side [by 0.235 (2) and 0.269 (2) Å] and one on the other side [by 0.257 (2) and 0.213 (2) Å] of