

C4—C9	1.362 (8)	C4A—N5A	1.330 (7)
C4—N5	1.425 (7)	C4A—C9A	1.371 (8)
N5—C6	1.324 (8)	N5A—C6A	1.411 (8)
C6—O61	1.252 (7)	C6A—O61A	1.191 (7)
C6—N7	1.409 (8)	C6A—N7A	1.415 (8)
N7—C8	1.432 (7)	N7A—C8A	1.403 (8)
C8—O81	1.209 (7)	C8A—O81A	1.225 (7)
C8—C9	1.415 (8)	C8A—C9A	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)
C25A—C21A—C22A—C23A	7.7 (11)
C21A—C22A—C23A—O23A	-167.4 (13)
C21A—C22A—C23A—C24A	7.8 (13)
O23A—C23A—C24A—C25A	153.9 (11)
C22A—C23A—C24A—C25A	-21.8 (13)
C23A—C24A—C25A—C21A	25.0 (10)
C22A—C21A—C25A—C24A	-19.2 (8)

H atoms were refined using a riding model, with isotropic displacement parameters $U(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$ and $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, 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: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (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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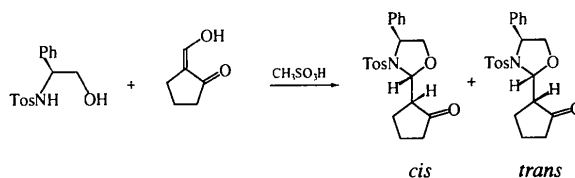
Abstract

The geometric parameters of the title compound, (2*S*)-2-[(2*R*,4*S*)-4-phenyl-3-tosyl-1,3-oxazolidin-2-yl]cyclopentanone, $\text{C}_{21}\text{H}_{23}\text{NO}_4\text{S}$, 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).



The oxazolidine ring in the *trans* configuration adopts an envelope conformation [$q_2 = 0.375(2)\text{Å}$, $\varphi_2 = 328.6(3)^\circ$ (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 [$q_2 = 0.349(3)\text{Å}$, $\varphi_2 = 126.3(4)^\circ$] 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 $30.0(2)^\circ$], the other, O31, forms a torsion angle of $-178.6(1)^\circ$ with C2. The two aromatic substituents enclose an angle of $19.8(1)^\circ$.

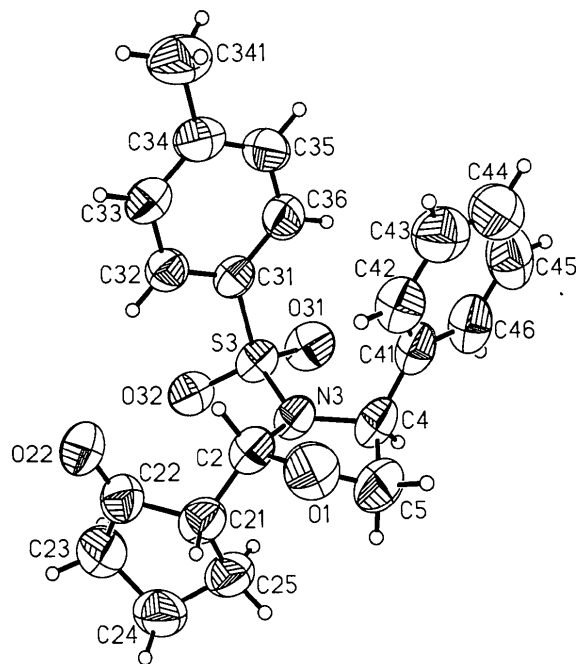


Fig. 1. Perspective view of the title compound with the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

$$S = 1.018$$

2901 reflections

246 parameters

$$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.0925P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.162$$

$$\Delta\rho_{\max} = 0.164 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.157 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	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 (\AA , $^\circ$)

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 *SHELXS86* (Sheldrick, 1985) and refined with *SHELXL96* (Sheldrick, 1996) by full-matrix least-squares methods. All H atoms were located by difference Fourier syntheses and refined with fixed individual displacement parameters [$U(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$ or $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$] using a riding model with C—H(aromatic)

Experimental

Single crystals were obtained from a diethyl ether solution.

Crystal data

C₂₁H₂₃NO₄S

$M_r = 385.46$

Orthorhombic

$P2_12_12_1$

$a = 8.704(1) \text{ \AA}$

$b = 12.636(1) \text{ \AA}$

$c = 17.819(2) \text{ \AA}$

$V = 1959.8(4) \text{ \AA}^3$

$Z = 4$

$D_x = 1.306 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation

$\lambda = 1.5418 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 30.00\text{--}35.00^\circ$

$\mu = 1.686 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block

$0.60 \times 0.30 \times 0.25 \text{ mm}$

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

$[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0262$

$\theta_{\text{max}} = 59.91^\circ$

$h = -9 \rightarrow 9$

$k = -14 \rightarrow 3$

$l = -20 \rightarrow 20$

3 standard reflections

frequency: 92 min

intensity decay: 0.80%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.0286$

$wR(F^2) = 0.0815$

Extinction correction:

SHELXL96 (Sheldrick, 1996)

= 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 about its local threefold axis.

Data collection: *SDP* (Enraf–Nonius, 1985). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL96* (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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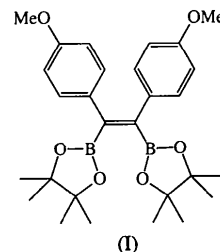
Abstract

The title compound, (*Z*)-1,2-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1,2-bis(4-methoxyphenyl)ethene, C₂₈H₃₈B₂O₆, 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 BO₂C₂ 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, B₂pin₂, 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, B₂cat₂] to internal and terminal alkynes as well as to 1,3-diyne (Lesley *et al.*, 1996). We also found that reactions using B₂pin₂ were slower than those using B₂cat₂.



We report here the structure of the product (I) of the addition of B₂pin₂ 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 C₄B₂ part of the molecule is essentially the same as for the B₂cat₂ adducts. There is a small twist about the C=C double bond, as measured by the C—C—C and B—C—C—B torsion angles of 8.0 (2) and 9.0 (2)°, respectively. The central C₄B₂ 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 CBO₂ 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 BO₂C₂ 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