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### *cis*-5-Methyl-4-phenyl-3-(4-toluenesulfonyl)-1,3-oxazin-2-one

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#### Abstract

The structure of the title compound,  $C_{18}H_{19}NO_4S$ , has been determined. This confirms the *anti* relative stereochemistry of the product of the lithium aluminium hydride reduction of methyl 2-methylene-3-phenyl-3-(4-toluenesulfonylamino)propanoate.

#### Comment

In order to further exploit a method for preparing substituted  $\alpha$ -methylene- $\beta$ -aminoesters (Perlmutter & Teo, 1984) some simple synthetic transformations of these compounds have been studied (Campi, Holmes, Perlmutter & Teo, 1994). In one case it was found that the product of a reduction was obtained as a single diastereoisomer. In order to determine the relative stereochemistry of this product it was converted to the title compound, (I). As there can sometimes be ambiguities in the  $^1H$  NMR analysis of the stereochemistry of oxazin-2-ones, the crystal structure of the title compound was determined. The results of this study confirmed the relative stereochemistry as 4,5-*cis*. Thus, the oxazinone adopts a conformation in which the methyl and 4-toluenesulfonyl substituents occupy pseudo-equatorial positions and the phenyl group has a pseudo-axial orientation. The factors which govern this striking feature are currently under investigation.

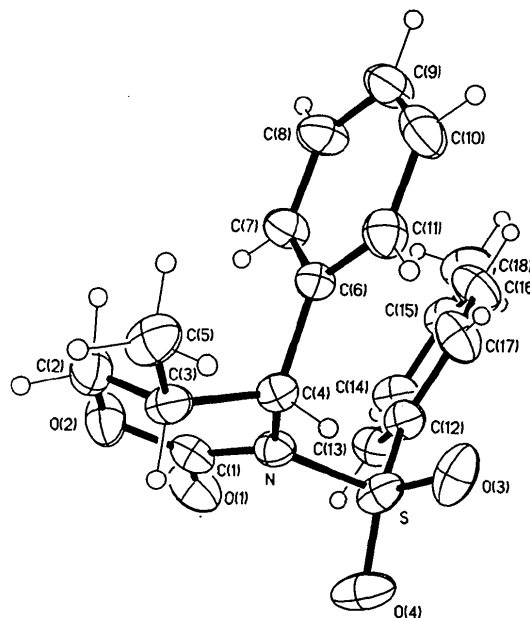
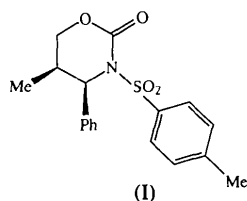


Fig. 1. A view of the title compound showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 40% probability level for non-H atoms; H atoms are drawn as circles of arbitrary radii.

#### Experimental

##### Crystal data

$C_{18}H_{19}NO_4S$

$M_r = 345.4$

Orthorhombic

*Pbca*

$a = 18.688$  (9) Å

$b = 11.428$  (6) Å

$c = 15.797$  (8) Å

$V = 3374$  (3) Å<sup>3</sup>

$Z = 8$

$D_x = 1.36$  Mg m<sup>-3</sup>

$D_m = 1.35$  (1) Mg m<sup>-3</sup>

$D_m$  measured by flotation in  $CCl_4$ /hexane

Cu  $K\alpha$  radiation

$\lambda = 1.5418$  Å

Cell parameters from 24 reflections

$\theta = 14$ – $19^\circ$

$\mu = 1.85$  mm<sup>-1</sup>

$T = 293$  K

Prism

$0.17 \times 0.10 \times 0.07$  mm

Colourless

##### Data collection

Philips PW1100 diffractometer

$\theta$  scans

Absorption correction:

Gaussian (SHELX76;

Sheldrick, 1976)

$T_{min} = 0.767$ ,  $T_{max} =$

0.874

3259 measured reflections

2875 independent reflections

2006 observed reflections  
[ $I > 3\sigma(I)$ ]

$\theta_{max} = 65.0^\circ$

$h = 0 \rightarrow 20$

$k = 0 \rightarrow 13$

$l = 0 \rightarrow 18$

3 standard reflections

frequency: 240 min

intensity decay: none

##### Refinement

Refinement on  $F$

$R = 0.048$

$wR = 0.058$

$\Delta\rho_{max} = 0.243$  e Å<sup>-3</sup>

$\Delta\rho_{min} = -0.476$  e Å<sup>-3</sup>

Extinction correction: none

$S = 0.772$   
 2006 reflections  
 217 parameters  
 H-atom parameters not  
 refined  
 $w = 1/[\sigma^2(F) + 0.0095F^2]$   
 $(\Delta/\sigma)_{\max} = 0.005$

Atomic scattering factors  
 from *International Tables*  
 for *X-ray Crystallography*  
 (1974, Vol. IV, Table  
 2.3.1) for C, N, O and  
 S, and Stewart, Davidson  
 & Simpson (1965) for H

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: TA1011). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
S	0.37386 (4)	0.00710 (7)	0.19207 (6)	0.0443 (3)
N	0.2950 (1)	-0.0512 (2)	0.2238 (2)	0.035 (1)
O(1)	0.3518 (1)	-0.2064 (3)	0.2838 (2)	0.059 (1)
O(2)	0.2355 (1)	-0.2043 (2)	0.2931 (2)	0.055 (1)
O(3)	0.3528 (1)	0.1101 (2)	0.1481 (2)	0.061 (1)
O(4)	0.4195 (1)	0.0163 (3)	0.2636 (2)	0.066 (1)
C(1)	0.2971 (2)	-0.1558 (3)	0.2675 (2)	0.041 (1)
C(2)	0.1668 (2)	-0.1510 (3)	0.2750 (2)	0.050 (1)
C(3)	0.1701 (2)	-0.0225 (3)	0.2612 (2)	0.040 (1)
C(4)	0.2270 (2)	0.0050 (3)	0.1954 (2)	0.034 (1)
C(5)	0.0965 (2)	0.0246 (3)	0.2379 (3)	0.052 (1)
C(6)	0.2093 (2)	-0.0301 (3)	0.1047 (2)	0.034 (1)
C(7)	0.2204 (2)	-0.1420 (3)	0.0742 (2)	0.044 (1)
C(8)	0.2058 (2)	-0.1705 (3)	-0.0090 (2)	0.053 (1)
C(9)	0.1793 (2)	-0.0877 (4)	-0.0626 (2)	0.056 (1)
C(10)	0.1667 (2)	0.0247 (4)	-0.0343 (2)	0.058 (1)
C(11)	0.1821 (2)	0.0539 (3)	0.0498 (2)	0.046 (1)
C(12)	0.4114 (2)	-0.0896 (3)	0.1183 (2)	0.041 (1)
C(13)	0.4603 (2)	-0.1740 (3)	0.1440 (2)	0.046 (1)
C(14)	0.4911 (2)	-0.2460 (3)	0.0842 (3)	0.051 (1)
C(15)	0.4747 (2)	-0.2360 (3)	-0.0018 (2)	0.052 (1)
C(16)	0.4246 (2)	-0.1519 (4)	-0.0252 (2)	0.063 (1)
C(17)	0.3932 (2)	-0.0782 (4)	0.0328 (2)	0.058 (1)
C(18)	0.5092 (2)	-0.3132 (4)	-0.0673 (3)	0.073 (2)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

S—N	1.694 (3)	O(2)—C(1)	1.339 (4)
S—O(3)	1.422 (3)	O(2)—C(2)	1.451 (4)
S—O(4)	1.420 (3)	C(2)—C(3)	1.485 (5)
S—C(12)	1.753 (3)	C(3)—C(4)	1.521 (4)
N—C(1)	1.381 (4)	C(3)—C(5)	1.521 (5)
N—C(4)	1.493 (4)	C(4)—C(6)	1.523 (4)
O(1)—C(1)	1.204 (4)		
N—S—O(3)	103.3 (1)	O(1)—C(1)—O(2)	117.8 (3)
N—S—O(4)	108.5 (1)	O(2)—C(2)—C(3)	114.1 (3)
O(3)—S—O(4)	119.6 (2)	C(2)—C(3)—C(4)	109.5 (3)
N—S—C(12)	107.3 (1)	C(2)—C(3)—C(5)	110.4 (3)
O(3)—S—C(12)	108.0 (2)	C(4)—C(3)—C(5)	113.2 (3)
O(4)—S—C(12)	109.6 (2)	N—C(4)—C(3)	107.5 (2)
S—N—C(1)	117.7 (2)	N—C(4)—C(6)	110.8 (2)
S—N—C(4)	118.8 (2)	C(3)—C(4)—C(6)	115.9 (3)
C(1)—N—C(4)	123.2 (2)	C(4)—C(6)—C(7)	122.6 (3)
C(1)—O(2)—C(2)	121.8 (3)	C(4)—C(6)—C(11)	119.0 (3)
N—C(1)—O(1)	123.1 (3)	S—C(12)—C(13)	120.5 (2)
N—C(1)—O(2)	119.0 (3)	S—C(12)—C(17)	119.0 (3)

The scan width was  $\pm(0.75 + 0.2\text{tan}\theta)^\circ$  in  $\theta$  from the calculated Bragg angle. Measurements were made using a scan speed of  $0.04^\circ \text{ s}^{-1}$  and background counts were collected for 50% of the scan time on each side of every scan. Data collection, cell refinement and data reduction: Philips PW1100 software. Structure solution and refinement: *SHELX76* (Sheldrick, 1976). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990).

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## (±)-[6 $\alpha$ (H)]-7,7,9 $\beta$ -Trimethyl-cis-tricyclo[6.3.1.0<sup>1,6</sup>]dodecan-2-one

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## Abstract

The title compound, C<sub>15</sub>H<sub>24</sub>O, possesses a bridged tricyclic structure with the cyclohexanone ring (A) and the cyclopentane ring (B) *cis* fused. The conformations of rings A and B can be best described as a distorted boat and an envelope, respectively. The cyclohexane ring (C) adopts a distorted chair conformation and the secondary methyl group at C9 is *cis* to the H atom at C8 [C13—C9—C8—H8 -52.6(3) $^\circ$ ].