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Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$

R factor = 0.035

wR factor = 0.107

Data-to-parameter ratio = 10.3

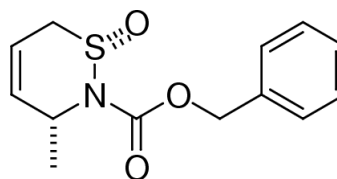
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Benzyl (1*R**,3*S**)-3,6-dihydro-3-methyl-1 λ^4 ,2-thiazine-2-carboxylate 1-oxideThe title compound, C₁₃H₁₅NO₃S, is shown to be the (1*R**,3*S**) isomer with a *cis* arrangement of the S=O group and the methyl group on the thiazine ring.

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Comment

The title compound, (I), was obtained by a [2+4] cycloaddition of *trans*-1,3-pentadiene and *N*-sulfinyl benzylcarbamate (Garigipati *et al.*, 1984) at room temperature in CH₂Cl₂. The reaction product contained two isomers in the ratio 9:1, with different orientations of the S=O bond relative to the methyl group (Hansen *et al.*, 2001). The minor isomer is *cis* (1*R**,3*S**), while the (1*R**,3*R**) isomer has a *trans* orientation. The structure of the minor isomer is reported here.

(I)

The title compound crystallizes in the orthorhombic non-centrosymmetric space group $P2_12_12_1$. A molecule with the atomic numbering scheme is shown in Fig. 1. The total puckering amplitude parameter Q_T is 0.523 (2) Å (Cremer & Pople, 1975; Iulek & Zukerman-Schpector, 1997). This ring puckering is described as 55% half-boat and 35% half-chair. It should be noted that there is a local pseudo-mirror through S1 and C3, and a local pseudo-twofold axis through the midpoints of the S1–N1 and C2–C3 bonds. The S=O bond is in a quasi-axial position, in accordance with several 1,2-thiazine 1-oxides (Boger & Weinreb, 1987). Least-squares planes through the phenyl ring (atoms C8–C13) and the thiazine ring (atoms C1–C4), show an angle of 9.2 (2)° between the two planes. Atoms S1 and N1 are displaced 0.641 (6) and 0.172 (6) Å, respectively, on opposite sides of the plane through atoms C1–C4. A plane through atoms N1/C6/O2/O3/C7/C8 shows a planar zigzag conformation for this part of the molecule (r.m.s. deviation 0.03 Å). A selection of bond lengths shows that these are all within the normal range for such bonds (Allen *et al.*, 1987). The S1=O1 bond length is 1.465 (2) Å. This value is in complete agreement with the values found in the crystal structures of (1*R**,3*S**)-3,6-dihydro-3-methyl-2-(toluene-4-sulfonyl)-1 λ^4 ,2-thiazine 1-oxide (Hansen *et al.*, 2001) and (1*R**,3*R**,6*S**)-3,6-dihydro-3,6-di-

methyl-2-(toluene-4-sulfonyl)-1 λ^4 ,2-thiazine 1-oxide (Hansen *et al.*, 2002). The molecules are packed in the crystal through a series of intra- and intermolecular short contacts (Taylor & Kennard, 1982) (see Table 1).

Experimental

The 1,4-thiazine 1-oxide was dissolved in warm Et₂O, and cold heptane was added until saturation was reached. The resulting solution was warmed carefully before crystals were grown by vapour diffusion of the solvent at room temperature.

Crystal data

C ₁₃ H ₁₅ NO ₃ S	Mo K α radiation
$M_r = 265.32$	Cell parameters from 25 reflections
Orthorhombic, $P2_12_12_1$	$\theta = 9\text{--}17^\circ$
$a = 6.1961$ (14) Å	$\mu = 0.24$ mm ⁻¹
$b = 13.613$ (4) Å	$T = 298$ (2) K
$c = 15.842$ (2) Å	Block, colourless
$V = 1336.2$ (6) Å ³	$0.60 \times 0.50 \times 0.40$ mm
$Z = 4$	
$D_x = 1.319$ Mg m ⁻³	

Data collection

Enraf–Nonius CAD-4 diffractometer	1245 reflections with $I > 2\sigma(I)$
ω - 2θ scans	$R_{\text{int}} = 0.005$
Absorption correction: ψ scan [McArdle & Daly (1999) (ABSCALC in OSCALEIL) and North <i>et al.</i> (1986)]	$\theta_{\text{max}} = 27.0^\circ$
$T_{\text{min}} = 0.869$, $T_{\text{max}} = 0.910$	$h = 0 \rightarrow 7$
1734 measured reflections	$k = 0 \rightarrow 17$
1695 independent reflections	$l = 0 \rightarrow 20$
	3 standard reflections
	frequency: 120 min
	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\text{max}} = 0.006$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$\Delta\rho_{\text{max}} = 0.22$ e Å ⁻³
$wR(F^2) = 0.107$	$\Delta\rho_{\text{min}} = -0.18$ e Å ⁻³
$S = 1.04$	Extinction correction: SHELXL97
1695 reflections	Extinction coefficient: 0.004 (2)
165 parameters	Absolute structure: (Flack, 1983)
H-atom parameters not refined	Flack parameter = 0.22 (14)
$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.0882P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1

Hydrogen-bonding geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
C1—H1B \cdots O2 ⁱ	0.97	2.39	3.345 (4)	169
C9—H9 \cdots O3	0.93	2.40	2.722 (4)	100
C5—H5A \cdots O1	0.96	2.62	3.269 (4)	125
C2—H2 \cdots O1 ⁱⁱ	0.93	2.59	3.485 (5)	161
C4—H4 \cdots O2	0.98	2.42	2.736 (4)	98
C7—H7A \cdots O2	0.97	2.58	2.677 (4)	85
C13—H13 \cdots O1 ⁱⁱⁱ	0.93	2.53	3.354 (4)	148

Symmetry codes: (i) $-x, y - \frac{1}{2}, \frac{3}{2} - z$; (ii) $x - 1, y, z$; (iii) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$.

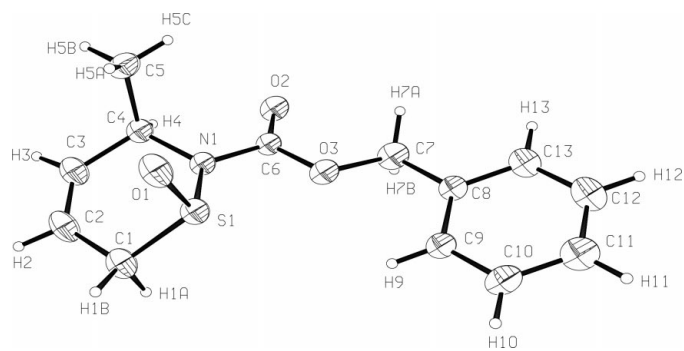


Figure 1

A view of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 20% probability level.

Data collection: CAD-4-PC Software (Enraf–Nonius, 1992); cell refinement: CELDIM in CAD-4-PC Software; data reduction: XCAD4 (McArdle & Higgins, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEX (McArdle, 1995); software used to prepare material for publication: OSCALEIL (McArdle, 1993).

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References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Boger, D. L. & Weinreb, S. W. (1987). *Hetero Diels–Alder Methodology in Organic Synthesis*, pp. 1–33. San Diego: Academic Press.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1359.
- Enraf–Nonius. (1992). *CAD-4-PC Software*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Garigipati, R. S., Freyer, A. J., Whittle, R. R. & Weinreb, S. M. (1984). *J. Am. Chem. Soc.* **106**, 7861–7867.
- Hansen, L. Kr., Bayer, A. & Gautun, O. R. (2001). *Acta Cryst.* **E57**, o1109–o1110.
- Hansen, L. Kr., Bayer, A. & Gautun, O. R. (2002). *Acta Cryst.* **E58**. Submitted.
- Iulek, J. & Zukerman-Schpector, J. (1997). *Quim. Nova*, **20**, 433–434.
- McArdle, P. (1993). *J. Appl. Cryst.* **26**, 752.
- McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
- McArdle, P. & Higgins, T. (1995). *XCAD4*. National University of Ireland, Galway, Ireland.
- McArdle, P. & Daly, P. (1999). *ABSCALC*. PC version. National University of Ireland, Galway, Ireland.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.