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(1*R**,3*R**,6*S**)-3,6-Dihydro-3,6-dimethyl-2-(toluene-4-sulfonyl)-1λ^4,2-thiazine 1-oxide

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The title compound, $C_{13}H_{17}NO_3S_2$, is shown to have a *trans* arrangement of the S═O group and the methyl groups in the thiazine ring.

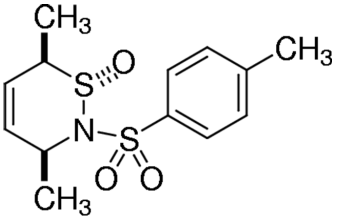
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.117
Data-to-parameter ratio = 13.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Hetero-[4+2]-cycloadditions of *N*-sulfinyl compounds (*R*—N═S═O) and dienes provide 3,6-dihydro-1,2-thiazine 1-oxides (Boger & Weinreb, 1987; Kresze & Wucherpfennig, 1967). 3,6-Dihydro-3,6-dimethyl-2-(toluene-4-sulfonyl)-1λ^4,2-thiazine 1-oxide ($C_{13}H_{17}NO_3S_2$) was obtained by a [2+4]-cycloaddition of (*E,E*)-2,4-hexadiene and *N*-sulfinyl-4-toluenesulfonamide in CH_2Cl_2 at room temperature (Mock & Nugent, 1975). The reaction product contained two isomers in the ratio 6:1, with different orientations of the S═O bond relative to the methyl groups. The (1*R**,3*S**,6*R**) isomer has the S═O bond *cis* to the methyl groups, while the (1*R**,3*R**,6*S**) isomer has a *trans* orientation. The configuration at sulfur has earlier been assigned by lanthanide-induced shielding effects in ^{1}H NMR (Mock & Nugent, 1975). The structure of the minor product is reported here.



(I)

The title compound, (I), crystallizes in the monoclinic centrosymmetric space group $P2_1/n$. A molecule with the atomic numbering scheme is shown in Fig. 1. The crystal structure was found to be that of the (1*R**,3*R**,6*S**)-isomer and the assignment of Mock & Nugent (1975) was confirmed. However, those authors assumed a half-chair conformation with the S═O bond in an equatorial position. The total puckering amplitude parameter Q_T is 0.604 (3) Å (Cremer & Pople, 1975; Iulek & Zukerman-Schpector, 1997). This ring puckering is described as 33% half-chair and 32% half-boat. One also sees that 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 part (atoms C6–C11) and the thiazine part (atoms C1–C4) have a dihedral angle of 51.00 (9)°. Atoms S1 and N1 are displaced 1.075 (4) and 0.303 (4) Å, respectively, out of the plane through atoms C1–C4. Atom C12 is almost coplanar with the phenyl group [deviation 0.094 (4) Å], while atom S2 lies

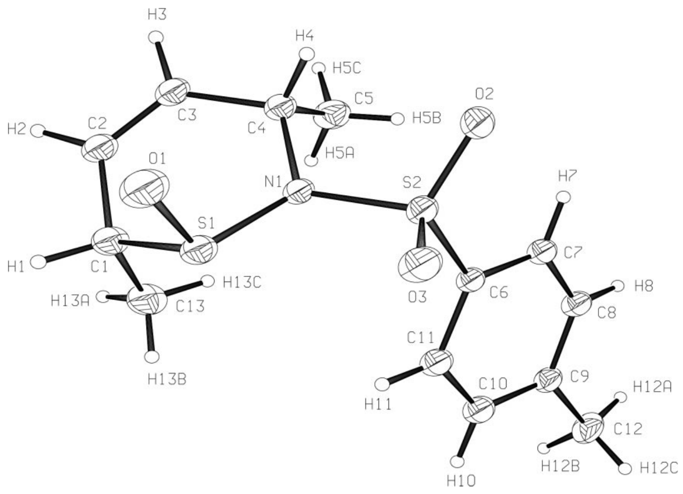


Figure 1
A view of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 20% probability level.

0.229 (3) Å out of the plane. A selection of bond lengths shows that these are all within normal ranges (Allen *et al.*, 1987). S2=O2 and S2=O3 are 1.423 (2) and 1.422 (2) Å, respectively, while S1=O1 is 1.466 (2) Å. These values are in complete agreement with the values found in the crystal structure of (1*R**,3*S**)-3,6-dihydro-3-methyl-2-(toluene-4-sulfonyl)-1λ^4,2-thiazine 1-oxide (Hansen *et al.*, 2001). 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 CH_2Cl_2 and heptane was added until saturation. Crystals were grown by slow evaporation of the solvent in the refrigerator.

Crystal data

$C_{13}H_{17}NO_3S_2$
$M_r = 299.40$
Monoclinic, $P2_1/n$
$a = 9.8136$ (16) Å
$b = 12.485$ (2) Å
$c = 11.9359$ (19) Å
$\beta = 103.610$ (14)°
$V = 1421.3$ (4) Å^3
$Z = 4$

$D_x = 1.399$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 12$–18°
$\mu = 0.38$ mm^{-1}
$T = 298$ (2) K
Block, colourless
0.5 × 0.4 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan [McArdle & Daly (1999) (*ABSCALC* in *OSCAIL*) and North *et al.* (1968)]
$T_{\min} = 0.828$, $T_{\max} = 0.893$
2478 measured reflections
2275 independent reflections

1998 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.011$
$\theta_{\max} = 25.0$°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 14$
$l = -14 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.117$
$S = 1.02$
2275 reflections
175 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0842P)^2 + 0.3859P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.018$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2$^{\rm i}$	0.98	2.62	3.463 (6)	144
C12—H12B···O2$^{\rm ii}$	0.96	2.66	3.564 (6)	158
C2—H2···O3$^{\rm i}$	0.93	2.73	3.612 (6)	158
C13—H13A···O1$^{\rm i}$	0.96	2.67	3.625 (3)	173
C8—H8···O1$^{\rm iii}$	0.93	2.70	3.365 (6)	130
C12—H12C···O1$^{\rm iv}$	0.96	2.74	3.358 (6)	123
C7—H7···O2	0.93	2.55	2.916 (6)	104
C5—H5B···O2	0.96	2.60	3.160 (6)	117
C4—H4···O2	0.98	2.48	2.806 (6)	99

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

Owing to an error in the data-collection procedure some reflections with $h > 9$ are missing.

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1992); cell refinement: *CELDIM* in *CAD*-4-*PC Software*; data reduction: *XCAD*4 (McArdle & Higgins, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *OSCAIL* (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 Inc.
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.
Hansen, L. Kr., Bayer, A. & Gautun, O. R. (2001). *Acta Cryst.* E**57**, o1109–o1110.
Iulek, J. & Zukerman-Schpector, J. (1997). *Quim. Nova*, **20**, 433–434.
Kresze, G. & Wucherpfennig, W. (1967). *Angew. Chem. Int. Ed.* **6**, 149–167.
McArdle, P. (1993). *J. Appl. Cryst.* **26**, 752.
McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
McArdle, P. & Higgins, T. (1995). *XCAD*4. National University of Ireland, Galway, Ireland.
McArdle, P. & Daly, P. (1999). *ABSCALC*. PC version. National University of Ireland, Galway, Ireland.
Mock, W. L. & Nugent, R. M. (1975). *J. Am. Chem. Soc.* **97**, 6521–6526.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

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