exo-3,6-Dihydro-3-methyl-2-(toluene-4-sulfonyl)-1λ^4,2-thiazine 1-oxide

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The title compound, $C_{12}H_{15}NO_3S_2$, is shown to be the *exo* isomer with a *cis* arrangement of the S=O group and the methyl group in the thiazine ring.

## Comment

Hetero-[4+2]-cycloadditions of *N*-sulfinyl compounds (R—N=S=O) and dienes provide 1,2-thiazine 1-oxides, which can be further transformed into synthetically useful derivatives, *e.g.* homoallylic amines or vicinal aminoalcohols (Weinreb, 1988). The title compound, (I), was obtained by a [2+4]-cycloaddition of *trans*-1,3-pentadiene and *N*-sulfinyl-4-toluenesulfonamide at room temperature in CH_2Cl_2 (Kresze & Wagner, 1972). The reaction product contained two isomers in the ratio 4:1 with different orientations of the S=O bond relative to the methyl group. The *exo* isomer has the S=O bond *cis* to the methyl group, while the *endo* isomer has a *trans* orientation. Here, the structure of the major compound is reported. The major compound was found to be the *exo* isomer.
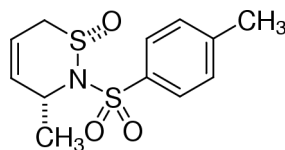
(I)

The title compound crystallizes in the orthorhombic non-centrosymmetric space group $P2_12_12_1$ and is therefore a conglomerate under the applied conditions of crystallization. A molecule with the atomic numbering scheme is shown in Fig. 1. The thiazine part of the molecule is found to be in a half-chair conformation, and one also sees that the S=O and the C4—C5 bonds are on the same side of the thiazine ring, giving the *exo* isomer. Least-squares planes through the phenyl part (atoms C6–C11) and the thiazine part (atoms C1–C4) show an angle of 71.2 (2)° between the two planes. Atoms S1 and N1 are displaced by 0.525 (9) and 0.261 (8) Å, respectively, on opposite sides with respect to the plane through atoms C1–C4. The total puckering amplitude parameter (Cremer & Pople, 1982) $Q_T = 0.499$ (3) Å and one notices the presence of a local twofold axis running through the midpoints of the S1—N1 and C2—C3 bonds. Atoms C12 and S2 nearly lie in the phenyl group plane, being displaced by 0.016 (9) and 0.099 (6) Å on the same side with respect to that plane. A selection of bond lengths shows that these are all within the normal range of such bonds (Allen *et al.*, 1987).
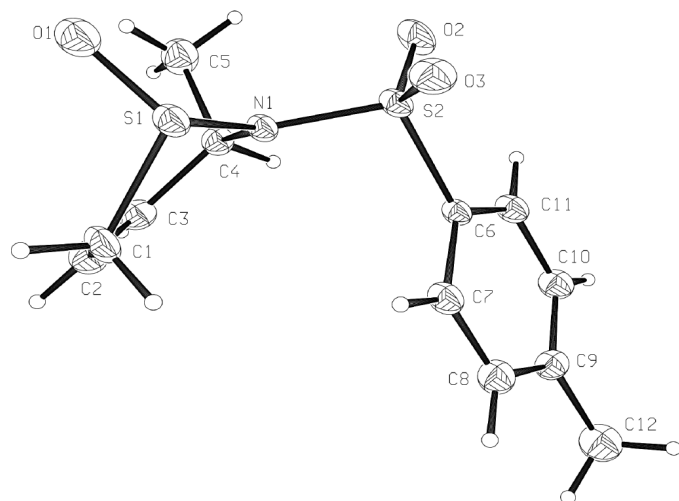
Figure 1
A view of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 20% probability level.

S2=O2 and S2=O3 are both 1.428 (3) Å, while the S1=O1 distance is 1.473 (4) Å. 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 vapour diffusion of the solvent in the refrigerator.

Crystal data

$C_{12}H_{15}NO_3S_2$	Mo $K\alpha$ radiation
$M_r = 285.37$	Cell parameters from 25
Orthorhombic, $P2_12_12_1$	reflections
$a = 8.277 (2)$ Å	$\theta = 8–14°$
$b = 9.723 (2)$ Å	$\mu = 0.40$ mm^{-1}
$c = 16.500 (2)$ Å	$T = 298 (2)$ K
$V = 1327.9 (5)$ Å^3	Block, colourless
$Z = 4$	$0.5 \times 0.3 \times 0.2$ mm
$D_x = 1.427$ Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 26.9°$
ω–2θ scans	$h = 0 \to 10$
1710 measured reflections	$k = -3 \to 12$
1669 independent reflections	$l = -6 \to 21$
1386 reflections with $I > 2\sigma(I)$	3 standard reflections
$R_{int} = 0.020$	frequency: 120 min
	intensity decay: 1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1332P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.057$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.162$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.91$ e Å^{-3}
1669 reflections	$\Delta\rho_{min} = -0.41$ e Å^{-3}
165 parameters	Absolute structure: Flack (1983)
H-atom parameters constrained	Flack parameter = 0.12 (19)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1B···O2^i	0.97	2.39	3.225 (6)	144
C11—H11···O2	0.93	2.51	2.888 (6)	104
C3—H3···O2^{ii}	0.93	2.58	3.487 (6)	165
C1—H1A···O3^{iii}	0.97	2.66	3.601 (6)	164
C5—H5A···O1	0.96	2.56	3.249 (8)	128
C2—H2···O1^{iv}	0.93	2.63	3.555 (6)	178

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

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992); cell refinement: *CELDIM* in *CAD-4-PC Software*; data reduction: *XCAD* (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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