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N-(4,4-Dimethoxy-1-oxo-1λ^6-thianylidene)-4-methyl-1-benzenesulfonamide

Steffen P. Creaser, Simon M. Pyke and Edward R. T. Tiekink*

Department of Chemistry, The University of Adelaide, Australia 5005

Correspondence e-mail: edward.tiekink@adelaide.edu.au

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Key indicators

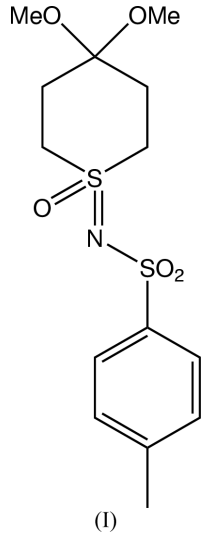
Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.136
Data-to-parameter ratio = 18.8

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

In the title compound, $C_{14}H_{21}NO_5S_2$, the imine group belonging to the tosylamide substituent occupies an axial position at the S atom of the thiane ring; the latter adopts a chair conformation. The torsion angle C(Ph)—S(O_2)—N═S in the bridge between the two rings is −93.6 (2)° and the S—N═S bond angle is 122.15 (14)°.

Comment

A view of the molecular structure of the title compound, (I), is shown in Fig. 1, from which it can be seen that the thiopyran ring adopts a chair conformation and the imine group at the ring S atom occupies an axial position. The torsion angles C7—S2—N1—S1 and S2—N1—S1—C6 are −93.6 (2) and −173.39 (18)°, respectively.



(I)

The S1—N1 distance of 1.561 (2) Å is significantly shorter than the S2—N1 distance of 1.620 (2) Å, a result that is consistent with the predominance of the canonical structure shown in the Scheme. This difference in the S—N distances shows an opposite sense, compared to the difference between the S—N bonds observed in the unoxidized (at S1) form of the molecule [1.639 (3) and 1.609 (3) Å for the distances corresponding to S1—N1 and S2—N1, respectively, as reported in Creaser *et al.* (2001)]. Oxidation at S1 also results in an expansion in the S1—N1—S2 angle to 122.15 (14)°, from 111.5 (1)°.

Conformational preferences for molecules closely related to (I) have been examined by Kálmán *et al.* (Jalsovszky, Kucsman, Ruff, Argay *et al.*, 1987; Jalsovszky, Kucsman, Ruff, Koritsánszky *et al.*, 1987).

Experimental

The title compound was prepared in 70% yield by the sodium hypochlorite oxidation (Campbell & Johnson, 1978) of *N*-(4,4-dimethoxy-1λ^4-thianylidene)-4-methyl-1-benzenesulfonamide (Creaser *et al.*, 2001) and was recrystallized from an ethanol solution of the compound to give colourless crystals, m.p. 424–426 K. Found: C 48.6, H 6.4, N 4.0%; calculated for $C_{14}H_{21}NO_5S_2$: C 48.4, H 6.9, N 4.0%.

Crystal data

$C_{14}H_{21}NO_5S_2$	D_x = 1.415 Mg m^{-3}
M_r = 347.44	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 21 reflections
a = 19.516 (8) Å	θ = 8.0–26.0°
b = 6.039 (2) Å	μ = 0.35 mm^{-1}
c = 14.940 (7) Å	T = 223 K
β = 112.17 (3)°	Plate, colourless
V = 1631 (1) Å^3	0.44 × 0.37 × 0.15 mm
Z = 4	

Data collection

Rigaku AFC-6*R* diffractometer	$h = -25 \rightarrow 23$
ω–2θ scans	$k = -7 \rightarrow 0$
4273 measured reflections	$l = 0 \rightarrow 19$
3762 independent reflections	3 standard reflections
2668 reflections with $I > 2\sigma(I)$	every 400 reflections
R_{int} = 0.020	intensity decay: 1.2%
θ_{max} = 27.6°	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 1.3963P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.047	
$wR(F^2)$ = 0.136	
S = 1.02	$(\Delta/\sigma)_{max} < 0.001$
3762 reflections	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
200 parameters	$\Delta\rho_{min}$ = −0.39 e Å^{-3}
H-atom parameters constrained	

The H atoms were placed in geometrically calculated positions and included in the final refinement in the riding-model approximation with a refined overall displacement parameter.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1992); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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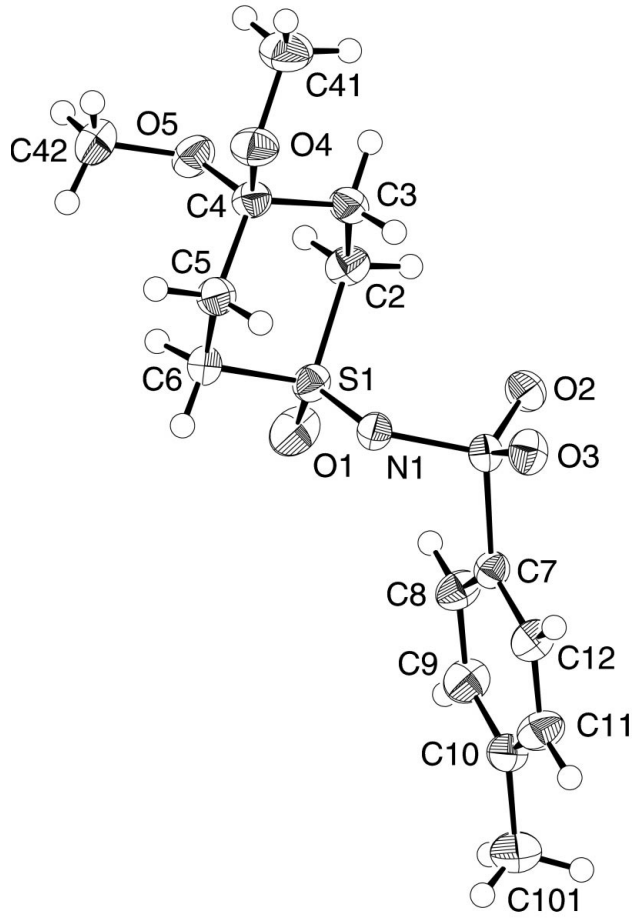

Figure 1
The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).

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