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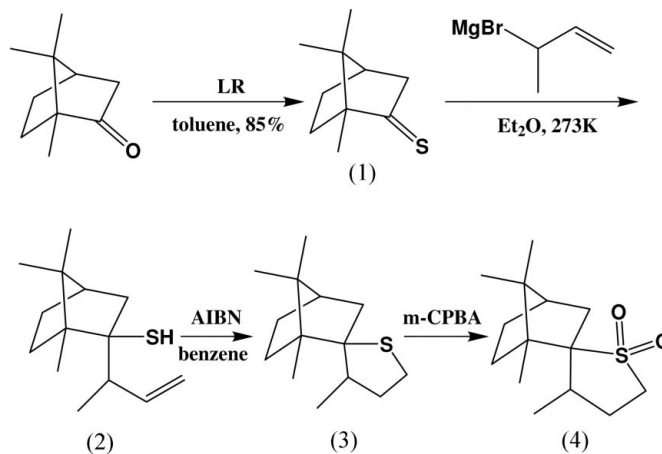
Key indicators

Single-crystal X-ray study
 $T = 273$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.052
 wR factor = 0.110
Data-to-parameter ratio = 16.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

1,3',7,7-Tetramethylspiro[bicyclo[2.2.1]-heptane-2,2'-thiolane] 1',1'-dioxide

In the title compound, $\text{C}_{14}\text{H}_{24}\text{O}_2\text{S}$, the tetrahydrothiophene ring adopts a half-chair conformation. $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into a ribbon-like structure along the a axis.Received 27 June 2006
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Comment

Chiral sulfides are important synthetic intermediates for convenient transformation into chiral sulfonium ylides, which can be used in the asymmetric synthesis of epoxides, cyclopropanes and aziridines (Li *et al.*, 1997). The reaction of thione (1) with allyl Grignard reagent under N_2 gives thiol (2) (Dagonneau *et al.*, 1974), which can be treated with 2,2'-azobis(2-methylbutyronitrile) (Aggarwal *et al.*, 2001) in benzene under reflux to obtain the spiro sulfide (3). Now we have oxidized the compound (3) to give the corresponding sulfone, (4), which is a colourless solid. We undertook the X-ray crystallographic analysis of (4) in order to elucidate the conformation and configuration.The bond lengths and angles are in good agreement with expected values (Allen *et al.*, 1987; Doye *et al.*, 1998). The tetrahydrothiophene ring adopts a half-chair conformation while the two cyclopentane rings adopt envelope conformations (the flap atom is C7) (Fig. 1). The crystal packing is stabilized by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1). These interactions link the molecules into a ribbon-like structure along the a axis (Fig. 2).

Experimental

Compound (3) (1.12 g, 5.0 mmol) in dichloromethane (8 ml) was added at 273 K to a solution of *m*-chloroperbenzoic acid (*m*-CPBA, 1.90 g, 11.0 mmol) in dichloromethane (25 ml). After stirring for 48 h,

the reaction mixture was filtered. Compound (4) crystallized on evaporation of the solvent.

Crystal data

$C_{14}H_{24}O_2S$ $Z = 8$
 $M_r = 256.39$ $D_x = 1.248 \text{ Mg m}^{-3}$
 Orthorhombic, $C222_1$ Mo $K\alpha$ radiation
 $a = 6.8918(8) \text{ \AA}$ $\mu = 0.23 \text{ mm}^{-1}$
 $b = 12.9137(14) \text{ \AA}$ $T = 273(2) \text{ K}$
 $c = 30.668(3) \text{ \AA}$ Block, colourless
 $V = 2729.4(5) \text{ \AA}^3$ $0.32 \times 0.26 \times 0.24 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer 7435 measured reflections
 2671 independent reflections
 φ and ω scans 2111 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan $R_{int} = 0.052$
 (SADABS; Bruker, 2000) $\theta_{max} = 26.0^\circ$
 $T_{min} = 0.93, T_{max} = 0.95$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 1.22P]$
 $R[F^2 > 2\sigma(F^2)] = 0.052$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.110$ $(\Delta/\sigma)_{max} = 0.001$
 $S = 0.95$ $\Delta\rho_{max} = 0.22 \text{ e \AA}^{-3}$
 2671 reflections $\Delta\rho_{min} = -0.19 \text{ e \AA}^{-3}$
 158 parameters Absolute structure: Flack (1983),
 H-atom parameters constrained 1139 Friedel pairs
 Flack parameter: 0.21 (11)

Table 1

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C2-H2A \cdots O1$	0.97	2.36	2.958 (4)	120
$C9-H9C \cdots O1$	0.96	2.25	3.186 (4)	165
$C10-H10A \cdots O2$	0.96	2.22	2.892 (4)	126
$C11-H11B \cdots O1^i$	0.97	2.58	3.521 (4)	165
$C11-H11A \cdots O2^{ii}$	0.97	2.46	3.392 (4)	160

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

H atoms were placed in calculated positions, with $C-H = 0.96-0.98 \text{ \AA}$, and included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(\text{methyl } C)$. The structure contains four chiral atoms, C1, C3, C6 and C13, but the configuration was not established unambiguously as the Flack (1983) parameter is 0.21 (11). For the inverted structure, the Flack parameter is 0.76 (12).

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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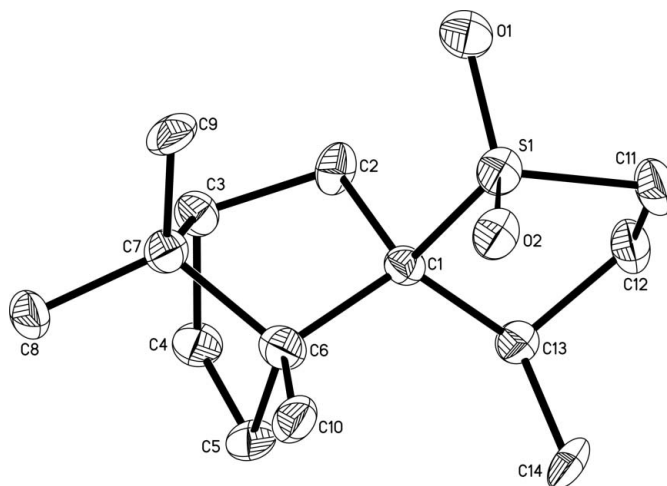


Figure 1

A view of the molecular structure of (4), showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

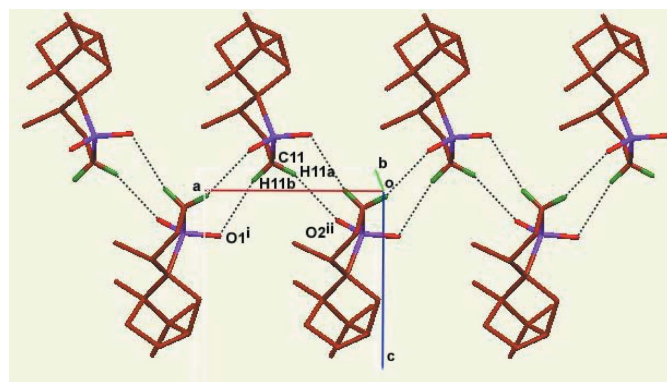


Figure 2

A view of the $C-H \cdots O$ hydrogen-bonded (dashed lines) chains in (4). Symmetry codes (i) and (ii) are as given in Table 2.

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