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(1*R*,2*R*,4*S*,5*S*)-1,2,4,8,8-Pentamethyl-3-thiabicyclo[3.2.1]octane 3,3-dioxide

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The title compound, $C_{12}H_{22}O_2S$, contains four chiral C atoms, which have been assigned. There are intermolecular C—H···O interactions in the crystal structure.

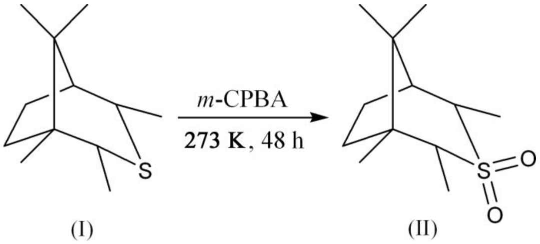
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.087
Data-to-parameter ratio = 21.0

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

Comment

Chiral sulfides are important synthetic intermediates for convenient transformation into chiral sulfur ylides, which can be used in the asymmetric synthesis of epoxides, cyclopropanes and aziridines (Li *et al.*, 1997). Our group has synthesized a new chiral sulfide 1,2,4,8,8-pentamethyl-3-thiabicyclo[3.2.1]octane, (I). We then oxidized this compound into the corresponding sulfone (II), which is a solid. As part of this study, we undertook the X-ray crystallographic analysis of (II) in order to elucidate the conformation and configuration of this oxidation product.



The molecular structure of (II) (Fig. 1) contains four chiral C atoms in the *R*,*R*,*S*,*S* diastereomeric form. The bond lengths and angles are in good agreement with expected values (Allen *et al.*, 1987). The five-membered ring (C1/C7/C4–C6) adopts an envelope conformation, with a dihedral angle of 45.5 (2)° between the C1/C4/C7 and C1/C4–C6 planes.

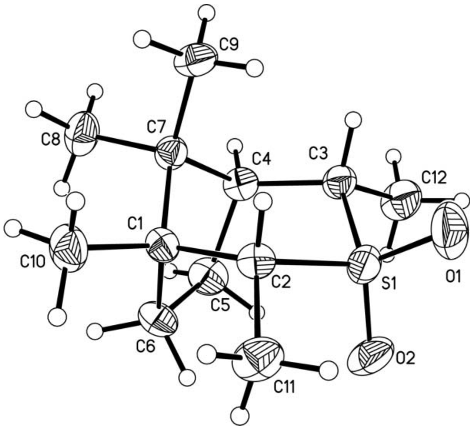


Figure 1
The structure of (II), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

The packing is stabilized by intermolecular C—H$\cdots$O interactions in the crystal structure (Fig. 2 and Table 1).

Experimental

Compound (I) (0.79 g, 4.0 mmol) in dichloromethane (5 ml) was added at 273 K to a solution of *m*-chloroperbenzoic acid (1.56 g, 9.0 mmol) in dichloromethane (20 ml). After stirring for 48 h, the reaction mixture was filtered. Compound (II) crystallized in 95% yield after evaporation of the solvent. Single crystals of (II) were obtained by slow evaporation of a petroleum ether–chloroform (1:2 *v*/*v*) solution of (II).

Crystal data

$C_{12}H_{22}O_2S$
$M_r = 230.36$
Orthorhombic, $P2_12_12_1$
$a = 7.9967$ (6) Å
$b = 10.6591$ (8) Å
$c = 14.5696$ (10) Å
$V = 1241.88$ (16) Å^3
$Z = 4$
$D_x = 1.232$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.24$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.32 × 0.26 × 0.26 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.93$, $T_{\max} = 0.94$
7787 measured reflections
2981 independent reflections
2530 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.056$
$\theta_{\max} = 28.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.087$
$S = 1.07$
2981 reflections
142 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.132 (6)
Absolute structure: Flack (1983), 1203 Friedel pairs
Flack parameter: −0.03 (7)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C4—H4$\cdots$O1$^{\rm i}$	0.98	2.53	3.454 (2)	156
C8—H8$A\cdots$O2$^{\rm ii}$	0.96	2.62	3.578 (2)	177
C11—H11$A\cdots$O1$^{\rm iii}$	0.96	2.58	3.424 (3)	147

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

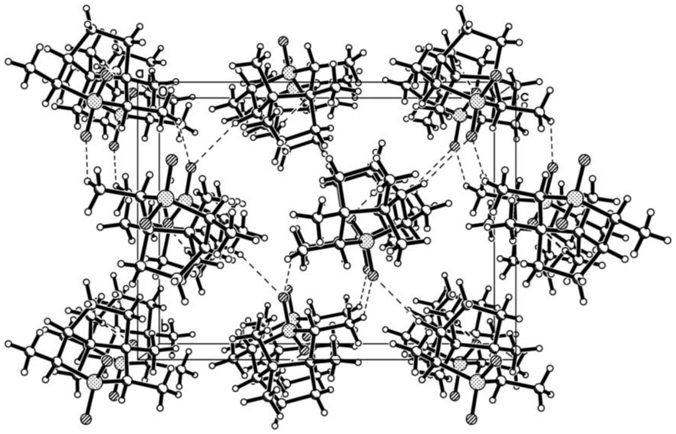


Figure 2
The crystal packing of (II), viewed down the *a* axis. Dashed lines indicate intermolecular C—H$\cdots$O hydrogen bonds.

H atoms were included in the riding-model approximation, with C—H = 0.96, 0.97 and 0.98 Å for methyl, methylene and methine H atoms, respectively, and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(methylene and methine C) or $1.5U_{\rm eq}$(methyl C).

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* and *PLATON* (Spek, 2003).

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