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(4a*SR*,9a*SR*)-9a-Methyl-5,5-dioxo-1,2,4a,6,7,9a-hexahydro-8-oxa-5λ^6-thiabenzo-1,4-cyclohepten-9-one

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Key indicators

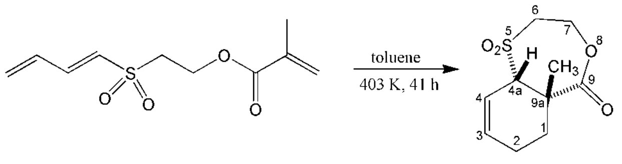
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.102
Data-to-parameter ratio = 14.5

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

The title compound, $C_{10}H_{14}O_4S$, crystallizes as a racemic mixture in space group $P2_1/c$, with two crystallographically independent molecules in the asymmetric unit. The seven-membered rings exhibit a chair conformation.

Comment

As detailed in the first article in this series (Zeller *et al.*, 2004), two of us (NC and PS) have been exploring the potential utility of sulfone-based tethers in intramolecular Diels–Alder cycloaddition reactions. This has resulted in the preparation of a series of bicyclic β-acyloxy sulfone cycloadducts, several of which have been subjected to analysis using single-crystal X-ray diffraction. In the present paper, the solid-state structure of the title cycloadduct, (I), as established by single-crystal X-ray diffraction methods, is described.



(I)

Compound (I) crystallizes in space group $P2_1/c$, with Z = 8 and two independent molecules in the asymmetric unit, as a

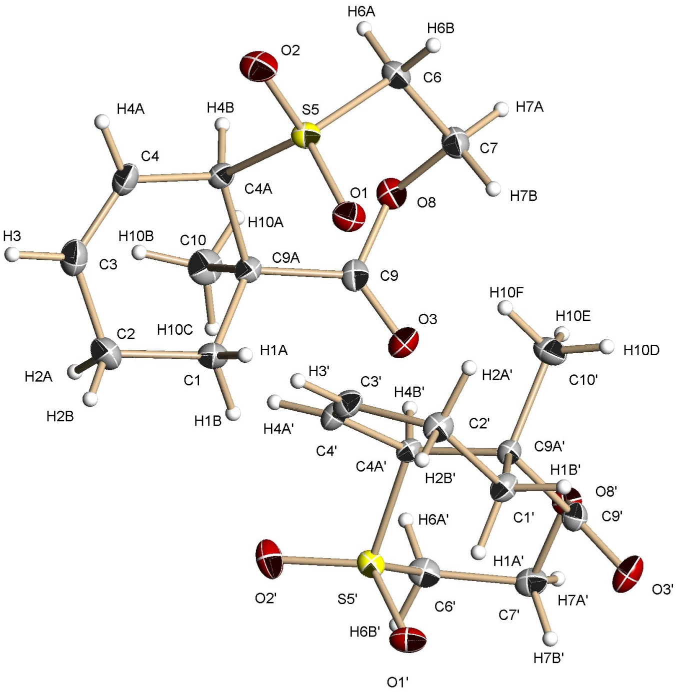


Figure 1
The structure of the asymmetric unit, showing 50% probability displacement ellipsoids.

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racemic mixture of both enantiomers. The conformations of the two independent molecules are almost indentical. Bond lengths and angles differ only marginally, and the weighted r.m.s. deviation for the molecule fit is 0.0477 Å. The six-membered rings both exhibit the half-chair conformation expected for cyclohexenes, and the angles at the unsaturated C atoms are 123.87 (14)° (for C4—C3—C2), 124.09 (13)° (for C3—C4—C4*A*), 123.93 (12)° (for C4′—C3′—C2′) and 123.94 (12)° (for C3′—C4′—C4*A*′; Fig. 1). The seven-membered rings both display a chair-like conformation.

Experimental

Compound (I) was isolated in 50% yield as a single diastereomer from an intramolecular Diels–Alder cycloaddition reaction of 2-[(*E*)-buta-1,3-dienylsulfonyl]ethyl methacrylate in toluene at 403 K. Compound (I) was purified by silica column chromatography (ethyl acetate/petroleum ether, 1:1). Crystals suitable for analysis using X-ray diffraction were grown *via* crystallization from ethanol with slow cooling.

Crystal data

$C_{10}H_{14}O_4S$
$M_r = 230.27$
Monoclinic, $P2_1/c$
$a = 10.9599$ (8) Å
$b = 22.4386$ (16) Å
$c = 9.1966$ (7) Å
$\beta = 113.690$ (1)°
$V = 2071.1$ (3) Å^3
$Z = 8$

$D_x = 1.477$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8706 reflections
$\theta = 2.6$–28.3°
$\mu = 0.30$ mm^{-1}
$T = 100$ (2) K
Block, colorless
0.40 × 0.40 × 0.29 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 1997–1999)
$T_{\min} = 0.585$, $T_{\max} = 0.916$
21071 measured reflections

5162 independent reflections
4902 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 28.3$°
$h = -14 \rightarrow 14$
$k = -29 \rightarrow 29$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.102$
$S = 1.07$
5162 reflections
355 parameters
Only coordinates of H atoms refined

$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 1.337P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.78$ e Å^{-3}
$\Delta\rho_{\min} = -0.45$ e Å^{-3}

All H atoms were positioned geometrically. Their coordinates were then refined freely, and their $U_{\rm iso}$ values were defined as 1.2 or $1.5U_{\rm eq}$ of the parent C atom. The s.u. values of the cell parameters are taken from the software, recognizing that the values are unreasonably small (Herbstein, 2000).

Data collection: *SMART* (Bruker, 1997–2000); cell refinement: *SAINT-Plus* (Bruker, 1997–1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXL*97.

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