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# (4a*SR*,7*RS*,9a*SR*)-7,9a-Dimethyl-5,5-dioxo-1,2,4a,6,7,9a-hexahydro-8-oxa-5$\lambda^6$-thia-1,4-benzocyclohepten-9-one

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**Key indicators**

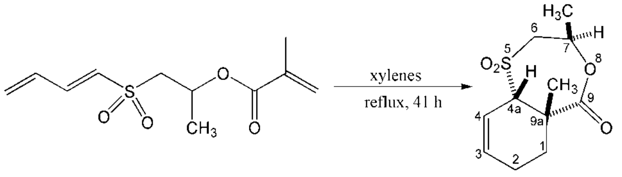
Single-crystal X-ray study
*T* = 100 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.035
*wR* factor = 0.097
Data-to-parameter ratio = 14.7

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

The title compound, $C_{11}H_{16}O_4S$, crystallizes as a racemic mixture in space group $P2_1/n$. The seven-membered ring exhibits a chair conformation and the methyl substituents point away from each other.

## 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 $P2_1/n$, with $Z = 4$, as a racemic mixture of both enantiomers. The six-membered ring exhibits the half-chair conformation expected for cyclohexenes, and the angles at the unsaturated C atoms are 123.93 (12)° (for C4—C3—C2) and 124.05 (12)° (for C3—C4—C4*A*; Fig. 1). The seven-membered ring displays a chair-like conformation. The methyl group on atom C7 is located in the equatorial position, pointing away from the methyl substituent on the bridgehead atom C9*A*.

## Experimental

Compound (I) was isolated in 48% yield as the major diastereomer from an intramolecular Diels–Alder cycloaddition reaction of 1-[(*E*)-buta-1,3-dienylsulfonyl]propan-2-yl methacrylate in refluxing xylenes. The crude product yielded crystals of (I) suitable for X-ray diffraction analysis *via* crystallization from ethyl acetate/hexane (1:2) with slow cooling.

### *Crystal data*

$C_{11}H_{16}O_4S$
$M_r = 244.30$
Monoclinic, $P2_1/n$
$a = 10.0232$ (10) Å
$b = 10.6255$ (10) Å
$c = 11.6726$ (11) Å
$\beta = 114.062$ (2)°
$V = 1135.13$ (19) Å$^3$
$Z = 4$
$D_x = 1.430$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 9433 reflections
$\theta = 2.2$–28.3°
$\mu = 0.28$ mm$^{-1}$
$T = 100$ (2) K
Block, colorless
0.4 × 0.2 × 0.2 mm

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*Data collection*

Bruker SMART APEX CCD diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 1997–1999)
$T_{\min} = 0.735$, $T_{\max} = 0.945$
11460 measured reflections
2829 independent reflections
2729 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 28.3°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 15$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.097$
$S = 1.10$
2829 reflections
193 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.5113P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.44$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.32$ 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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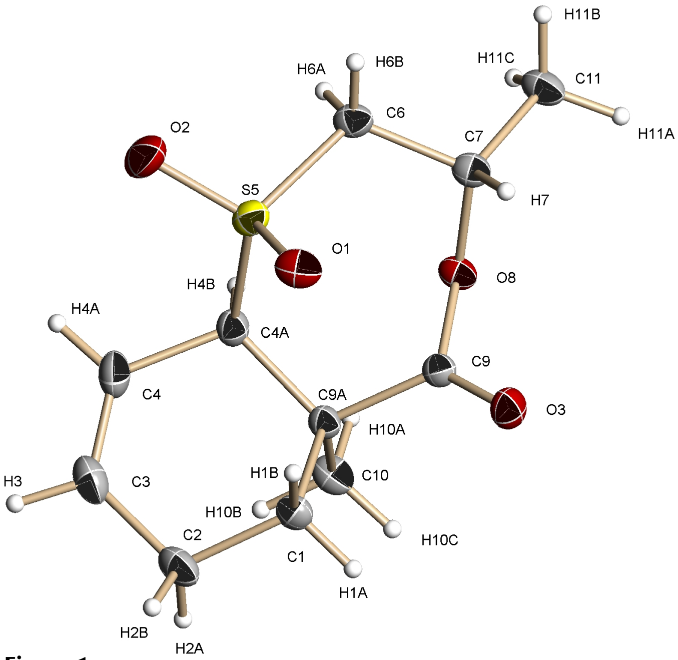

**Figure 1**
The molecular structure, showing 50% probability displacement ellipsoids.

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