

## **(4a*SR*,7*RS*,9a*RS*)-7-*tert*-Butyl-9a-methyl-5,5-dioxo-1,2,4a,9a-tetrahydro-8-oxa-5 $\lambda^6$ -thiabenzocycloheptan-9-one. Corrigendum**

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In the paper by Zeller, Hunter, Sampson & Chumachenko [*Acta Cryst.* (2006), **E62**, o366–o367], the stereochemical descriptors are given incorrectly in the title. The correct title is '(4a*RS*,7*RS*,9a*SR*)-7-*tert*-Butyl-9a-methyl-5,5-dioxo-1,2,4a,9a-tetrahydro-8-oxa-5 $\lambda^6$ -thiabenzocycloheptan-9-one'.

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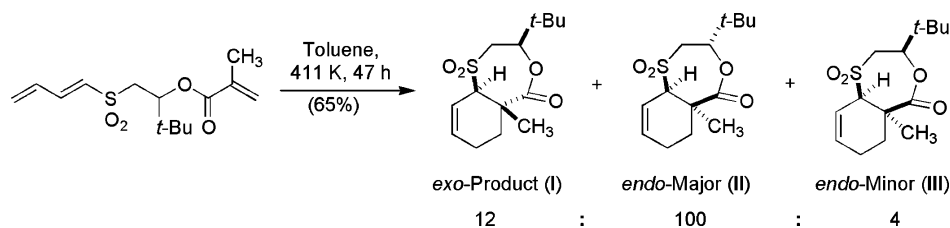
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## Key indicators

Single-crystal X-ray study  
T = 100 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$   
R factor = 0.038  
wR factor = 0.100  
Data-to-parameter ratio = 20.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.**(4a*SR*,7*RS*,9a*RS*)-7-*tert*-Butyl-9a-methyl-5,5-dioxo-  
1,2,4a,9a-tetrahydro-8-oxa-5 $\lambda^6$ -thiabenzocyclo-  
heptan-9-one**The title compound, C<sub>14</sub>H<sub>22</sub>O<sub>4</sub>S, crystallizes with two crystallographically independent molecules in the asymmetric unit. In both molecules, the six-membered ring exhibits a sofa conformation while the seven-membered ring is in a boat-like conformation with the *tert*-butyl group in a pseudo-equatorial position.

## Comment

As detailed in an earlier series of articles (Zeller *et al.*, 2004*a,b,c,d*; Chumachenko *et al.*, 2005), two of the current authors (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  $\beta$ -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.The title compound was isolated as one of three diastereomers from an intramolecular Diels–Alder cycloaddition reaction of 1-[(*E*)-butadienesulfonyl]-3,3-dimethyl-2-butyl methacrylate in toluene at 441 K (Chumachenko *et al.*, 2005) (see scheme).Compound (I) crystallizes in the monoclinic space group  $P2_1/c$  with  $Z = 8$  and two crystallographically independent molecules in the asymmetric unit, as a racemic mixture of both enantiomers (Fig. 1). The conformations of the two independent molecules are almost identical. Bond lengths and angles differ only marginally, and the weighted r.m.s. deviation for the molecular fit is 0.0445 Å.The six-membered ring may be described as in a sofa conformation with only C9a being non-coplanar with the other atoms of the ring. The deviation for C9a (or C9a') from the least-squares plane defined by C1 to C4a (or C1' to C4a') is 0.699 (2) Å, the largest deviation of any atom defining the plane being 0.1069 (9) Å for C2'. This differentiates the *exo* compound (I) from four similar *endo* cycloadducts described previously (Zeller *et al.*, 2004*a,b,c,d*). Each of these compounds exhibited a half-chair conformation that is not easily accessible for the *exo*-adduct (I) described here. The

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angles at the unsaturated C atoms in (I) are 123.2 (1), 123.1 (1), 122.6 (1), and 122.8 (1)° for C4a–C4–C3, C4a'–C4'–C3', C4–C3–C2, and C4'–C3'–C2', respectively.

The seven-membered ring displays a boat-like conformation with the bulky *tert*-butyl group in a pseudo-equatorial position. In the crystal structure, some weak intermolecular C–H···O interactions with H···O distances in the range 2.3–2.5 Å are found [O3···H4A<sup>i</sup> = 2.39 Å, H6B'<sup>iii</sup>···O1<sup>iii</sup> = 2.30 Å, O1···H6B<sup>iii</sup> = 2.43 Å; symmetry codes: (i)  $x, y - 1, z$ ; (ii)  $2 - x, y - \frac{1}{2}, \frac{1}{2} - z$ ; (iii)  $1 - x, y + \frac{1}{2}, \frac{1}{2} - z$ ]. These interactions seem to be somehow symmetrical, *i.e.* the interaction between S=O atom O1 and H6B<sup>iii</sup> is similarly present for O1<sup>iii</sup> and H6B' of the other symmetry-independent molecule, and for the interaction of C=O atom O3 with H4A<sup>i</sup> a similar but less pronounced counterpart is also found (O3'···H4A = 2.68 Å). This symmetric arrangement of the short contacts can be seen as an indication that these weak interactions may play a significant role as a driving force towards the kind of packing present in the crystal structure of (I). All bond lengths and angles of (I) are within their expected ranges.

## Experimental

The title compound was synthesized as described by Chumachenko *et al.* (2005) as a mixture of the *exo*- and two *endo*-cycloadducts, (I), (II) and (III), in a ratio of 12/100/4 as established by <sup>1</sup>H NMR of the crude reaction product (see scheme). The major *endo* compound (II) was removed from the mixture by selective crystallization in 1:3 ethyl acetate–hexanes. [The single-crystal structure of (II) is described in the next article of this series.] The mother liquor was then evaporated to dryness and the *exo* compound (I) was isolated by column chromatography (dichloromethane) and purified by recrystallization from a minimal amount of ethyl acetate to yield crystals suitable for X-ray structural analysis.

### Crystal data

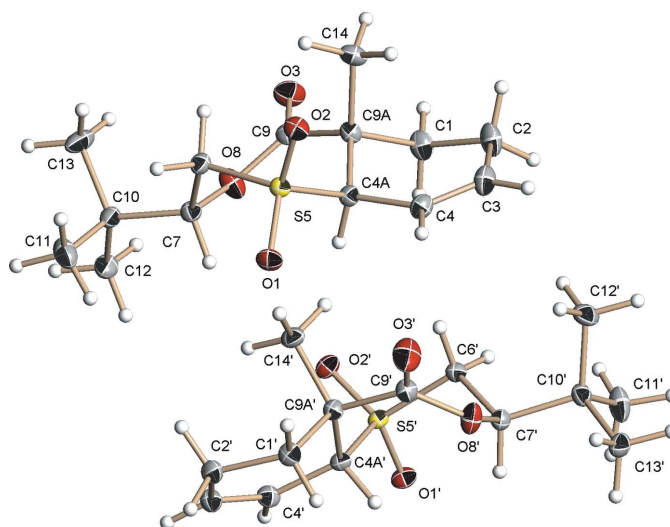
C <sub>14</sub> H <sub>22</sub> O <sub>4</sub> S	$D_x = 1.314 \text{ Mg m}^{-3}$
$M_r = 286.38$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 8187 reflections
$a = 15.5204$ (7) Å	$\theta = 2.3\text{--}30.5^\circ$
$b = 9.8367$ (4) Å	$\mu = 0.23 \text{ mm}^{-1}$
$c = 23.0445$ (10) Å	$T = 100$ (2) K
$\beta = 124.6260$ (10)°	Block, colourless
$V = 2895.0$ (2) Å <sup>3</sup>	$0.43 \times 0.42 \times 0.29 \text{ mm}$
$Z = 8$	

### Data collection

Bruker SMART APEX CCD diffractometer	7195 independent reflections
$\omega$ scans	6801 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS in SAINT-Plus; Bruker, 2003)	$R_{\text{int}} = 0.025$
$T_{\text{min}} = 0.891, T_{\text{max}} = 0.935$	$\theta_{\text{max}} = 28.3^\circ$
29131 measured reflections	$h = -20 \rightarrow 20$
	$k = -13 \rightarrow 13$
	$l = -30 \rightarrow 30$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 1.2868P]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.100$	$(\Delta/\sigma)_{\text{max}} = 0.002$
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.49 \text{ e } \text{Å}^{-3}$
7195 reflections	$\Delta\rho_{\text{min}} = -0.52 \text{ e } \text{Å}^{-3}$
351 parameters	
H-atom parameters constrained	



**Figure 1**  
The asymmetric unit of (I), showing 50% probability displacement ellipsoids.

All H atoms were positioned geometrically, with a C–H bond distance of 1.00 or 0.99 Å. They were refined with isotropic displacement parameters of 1.5 (methyl) or 1.2 (all others) times  $U_{\text{eq}}(\text{C})$ . Methyl H atoms were allowed to rotate to best fit the experimental data. 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, 2002); cell refinement: *SAINTE-Plus* (Bruker, 2003); data reduction: *SAINTE-Plus*; 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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