

## **(4aSR,7SR,9aSR)-7-tert-Butyl-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**, o370–o371], the stereochemical descriptors are given incorrectly in the title. The correct title is '(4aRS,7SR,9aRS)-7-tert-Butyl-5,5-dioxo-1,2,4a,9a-tetrahydro-8-oxa-5 $\lambda^6$ -thiabenzocycloheptan-9-one'.

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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 = 0.102  
Data-to-parameter ratio = 20.3

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

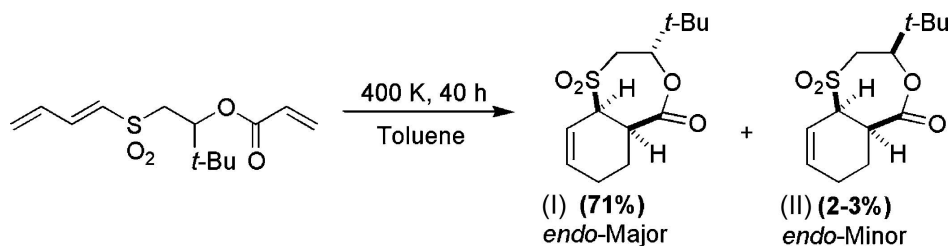
(4a*SR*,7*SR*,9a*SR*)-7-*tert*-Butyl-5,5-dioxo-1,2,4a,9a-tetrahydro-8-oxa-5 $\lambda^6$ -thiabenzocycloheptan-9-one

The six-membered cyclohexene ring of the title compound, C<sub>13</sub>H<sub>20</sub>O<sub>4</sub>S, exhibits a sofa conformation, and the seven-membered ring displays an irregular chair-like conformation. Only weak intermolecular C—H···O interactions are observed.

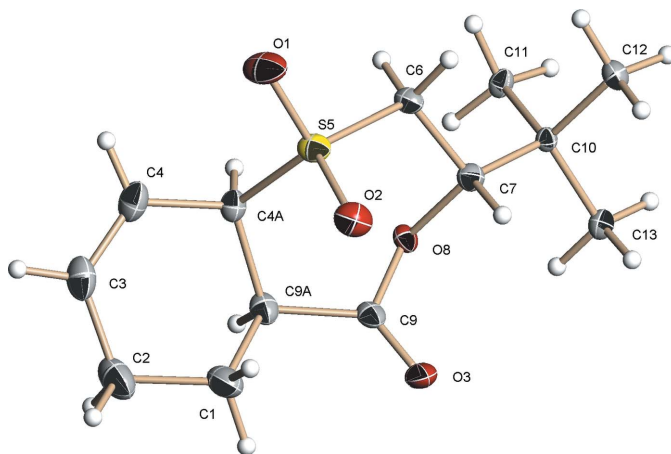
Comment

The background to this study is described in the first paper of this series (Zeller *et al.*, 2006a).

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The title compound, (I), crystallizes in the monoclinic space group *P*2<sub>1</sub>/*n* with *Z* = 4 and thus as a racemic mixture of both enantiomers. In line with the six other cycloadducts described previously (Zeller *et al.*, 2004*a,b,c,d*; Zeller *et al.*, 2006*a,b*) the six-membered ring of (I) exhibits a sofa conformation expected for cyclohexenes. In the present case, only atom C1 is non-coplanar with the other atoms of the ring. The r.m.s. fit for the plane defined by atoms C2, C3, C4 and C4A is 0.0488 Å, and the deviation for C1 from the least-squares plane is 0.646 (2) Å. For the atoms defining the plane, the largest deviation is 0.0585 (9) Å for C4A. The angles at the unsatu-



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

rated C atoms are  $124.15(13)^\circ$  for C4–C3–C2 and  $123.39(13)^\circ$  for C3–C4–C4A (Fig. 1). The seven-membered ring displays an irregular chair-like conformation. The sterically demanding *tert*-butyl group at C7 is located in a pseudo-equatorial position.

All intermolecular contacts in (I) are weak, the most pronounced being two C–H $\cdots$ O contacts: C6 $\cdots$ O2<sup>i</sup> with a C $\cdots$ O distance of 3.400 (2) Å and a C–H $\cdots$ O angle of  $165.4^\circ$ , and C9 $\cdots$ O3<sup>ii</sup> with a slightly shorter C $\cdots$ O distance of 3.304 (1) Å but with a C–H $\cdots$ O angle unfavorably small for a strong interaction ( $96.11^\circ$ ) [symmetry codes: (i)  $-x, 1 - y, -z$ ; (ii)  $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$ ].

## Experimental

The title compound was synthesized following an analogous procedure to that described by Chumachenko *et al.* (2005) for related compounds. 1-[(*E*)-Butadienesulfonyl]-3,3-dimethyl-2-butyl acrylate (1.36 g, 5.00 mmol) was heated in toluene (450 ml, 0.011 M) at 400 K for 40 h. Crystallization of the crude reaction mixture from a minimum amount of ethyl acetate gave the title compound as a white crystalline solid (0.83 g, 3.05 mmol, 61%; m.p. 462 K). Column chromatography (dichloromethane) of the mother liquor gave, as the first fraction, an additional 0.13 g (0.48 mmol, 10%) of (I) resulting in a combined yield for (I) of 0.96 g (3.53 mmol, 71%). A small second chromatographic fraction contained the minor *endo*-product (II) in about 2% yield, contaminated with traces of (I).

### Crystal data

|  |   |
|--|---|
| C <sub>13</sub> H <sub>20</sub> O <sub>4</sub> S | $D_x = 1.333 \text{ Mg m}^{-3}$           |
| $M_r = 272.35$                                   | Mo $K\alpha$ radiation                    |
| Monoclinic, $P2_1/n$                             | Cell parameters from 6071 reflections     |
| $a = 11.8554(8) \text{ \AA}$                     | $\theta = 3.0\text{--}30.5^\circ$         |
| $b = 5.6653(4) \text{ \AA}$                      | $\mu = 0.24 \text{ mm}^{-1}$              |
| $c = 20.2326(14) \text{ \AA}$                    | $T = 100(2) \text{ K}$                    |
| $\beta = 92.681(1)^\circ$                        | Block, colorless                          |
| $V = 1357.42(16) \text{ \AA}^3$                  | $0.45 \times 0.38 \times 0.36 \text{ mm}$ |
| $Z = 4$  |   |

### Data collection

|  |  |
|--|--|
| Bruker SMART APEX CCD diffractometer                                   | 3368 independent reflections           |
| $\omega$ scans   | 3308 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (SADABS in SAINT-Plus; Bruker, 2003) | $R_{\text{int}} = 0.021$               |
| $T_{\text{min}} = 0.850, T_{\text{max}} = 0.916$                       | $\theta_{\text{max}} = 28.3^\circ$     |
| 13221 measured reflections   | $h = -15 \rightarrow 15$               |
|  | $k = -7 \rightarrow 7$                 |
|  | $l = -26 \rightarrow 26$               |

### Refinement

|                                 |  |
|---------------------------------|--|
| Refinement on $F^2$             | $w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.6798P]$    |
| $R[F^2 > 2\sigma(F^2)] = 0.038$ | where $P = (F_o^2 + 2F_c^2)/3$                       |
| $wR(F^2) = 0.102$               | $(\Delta/\sigma)_{\text{max}} < 0.001$               |
| $S = 1.15$                      | $\Delta\rho_{\text{max}} = 0.52 \text{ e \AA}^{-3}$  |
| 3368 reflections                | $\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$ |
| 166 parameters                  |  |
| H-atom parameters constrained   |  |

All H atoms were positioned geometrically (C–H = 0.95–1.00 Å). They were refined with isotropic displacement parameters of 1.5 (methyl) or 1.2 (all others) times that of the equivalent isotropic displacement parameter of the adjacent C atom. 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: SAINT-Plus (Bruker, 2003); data reduction: SAINT-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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