

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

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In the paper by Zeller, Hunter, Sampson & Chumachenko [*Acta Cryst.* (2006), **E62**, o368–o369], the stereochemical descriptors are given incorrectly in the title. The correct title is '(4a*SR*,7*RS*,9a*SR*)-7-*tert*-Butyl-9a-methyl-5,5-dioxo-1,2,4a,9a-tetrahydro-8-oxa-5 λ^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 = 0.049
wR factor = 0.119
Data-to-parameter ratio = 67.3

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-9a-methyl-5,5-dioxo-1,2,4a,9a-tetrahydro-8-oxa-5 λ^6 -thiabenzocycloheptan-9-one

The title compound, C₁₄H₂₂O₄S, crystallizes as a non-merohedral twin with a twinning ratio close to 1:1. The six-membered ring exhibits a half-chair conformation, and the seven-membered ring displays an irregular chair-like conformation.

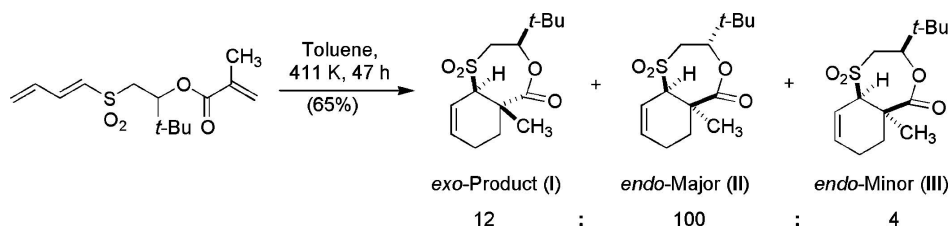
Comment

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

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Compound (II) crystallizes in the triclinic space group $P\bar{1}$ with $Z = 2$ as a racemic mixture of both enantiomers (Fig. 1). The crystal under investigation was found to be non-merohedrally twinned with two components of similar contribution. The final twin ratio was found to be 0.4675 (6). Similar to four related *endo*-cycloadducts described previously (Zeller *et al.*, 2004*a,b,c,d*), the six-membered ring of (II) exhibits the half-chair conformation expected for cyclohexenes. The angles at the unsaturated C atoms are 124.3 (1)° for C4–C3–C2 and 123.5 (1)° for C3–C4–C4A (Fig. 1).

The seven-membered ring displays an irregular chair-like conformation. The sterically demanding *tert*-butyl group on

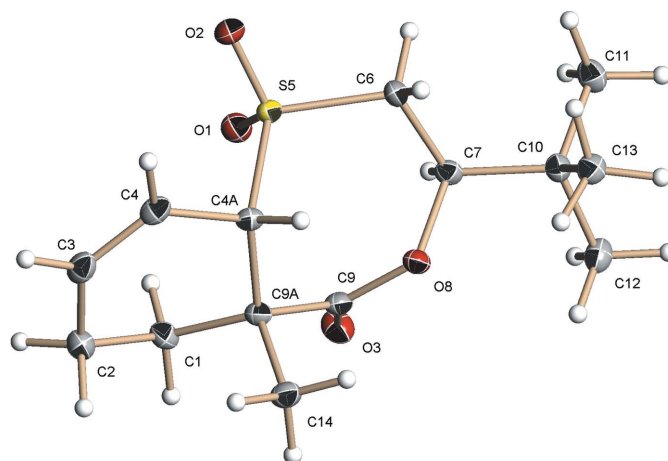


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

C7 is located in a pseudo-equatorial position. All intermolecular contacts in (II) are weak, the most pronounced being 3.465 (1) Å for O2...C13(-x, 1 - y, 1 - z). All bond lengths and angles 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, *viz.* (I), (II) and (III), in a ratio of 12/100/4, as established by ¹H NMR of the crude reaction product. The title compound, (II), was isolated from the mixture by selective crystallization from ethyl acetate/hexanes (1:3 *v/v*) to yield crystals suitable for X-ray structural analysis. [The single-crystal structure of (I) is described in the previous article of this series (Zeller *et al.*, 2006).]

Crystal data

C ₁₄ H ₂₂ O ₄ S	Z = 2
M _r = 286.38	D _x = 1.345 Mg m ⁻³
Triclinic, P $\bar{1}$	Mo K α radiation
a = 5.6949 (5) Å	Cell parameters from 6500 reflections
b = 11.1309 (10) Å	θ = 3.2–30.6°
c = 12.3085 (11) Å	μ = 0.24 mm ⁻¹
α = 112.539 (2)°	T = 100 (2) K
β = 90.278 (2)°	Block, colourless
γ = 100.160 (2)°	0.29 × 0.20 × 0.18 mm
V = 707.07 (11) Å ³	

Data collection

Bruker SMART APEX CCD diffractometer	11949 measured reflections
ω scans	11949 independent reflections
Absorption correction: multi-scan (TWINABS in SAINTE-Plus; Bruker, 2003)	11372 reflections with I > 2 σ (I)
T _{min} = 0.870, T _{max} = 0.96	θ_{\max} = 28.3°
	h = -7 → 7
	k = -14 → 14
	l = -16 → 16

Refinement

Refinement on F ²	w = 1/[$\sigma^2(F_o^2) + (0.0415P)^2 + 0.3942P$]
R[F ² > 2 σ (F ²)] = 0.049	where P = (F _o ² + 2F _c ²)/3
wR(F ²) = 0.119	(Δ/σ) _{max} < 0.001
S = 1.16	$\Delta\rho_{\max}$ = 0.56 e Å ⁻³
11919 reflections	$\Delta\rho_{\min}$ = -0.39 e Å ⁻³
177 parameters	
H-atom parameters constrained	

The dataset under investigation had 8323 identified reflections associated with component 1 only, 5345 reflections with component 2 only and 2963 are belonging to both components. The crystal under investigation was found to be non-merohedrally twinned. Two components were identified using GEMINI (Bruker, 1999) and both components were simultaneously integrated using SAINTE-Plus (Bruker, 2003). The twin relationship was found to be described by the matrix (1.00039, 0.00139, -0.00035; -0.68441, -1.00009, 0.00096;

-0.02404, -0.00039, -1.00027). The data set under investigation had 5380 identified reflections associated with component 1 only, 5367 reflections with component 2 only and 296 belonging to both components. I/ σ for overlapping reflections was 18.7. The data set was corrected for absorption using TWINABS (Bruker, 2003), which was also used to create both an HKLF 4 as well as an HKLF 5 format file. The HKLF 4 format file (with the non-overlapping reflections of component 1 only) was used to establish the space group and solve the structure. The HKLF 5 format file was used for the refinement of the structure; the twin ratio refined to 0.4675 (6). The overlapping of reflections by non-merohedral twinning prevents the merging of equivalent reflections before refinement. 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: 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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