

endo*-4-Phenyl-7-*n*-propyl-2-oxa-6-thia-3-aza-bicyclo[3.2.0^{1,4}]hept-3-ene 6,6-dioxide*Graeme J. Gainsford*** and
Anthony D. WoolhouseIndustrial Research Limited, PO Box 31-310,
Lower Hutt, New Zealand

Correspondence e-mail: g.gainsford@irl.cri.nz

Key indicators

Single-crystal X-ray study

 $T = 130\text{ K}$ Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$ R factor = 0.057 wR factor = 0.121

Data-to-parameter ratio = 10.0

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

Two independent molecules of the title compound, $\text{C}_{13}\text{H}_{15}\text{NO}_3\text{S}$, containing the novel *endo*-thiabicyclo[3.2.0^{1,4}]-hept-3-ene ring, crystallize in a triclinic cell. Both fused rings in the bicyclic system are non-planar, with the five-membered C_3NO rings adopting envelope conformations.

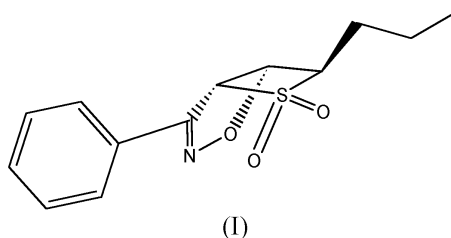
Received 26 July 2002

Accepted 9 August 2002

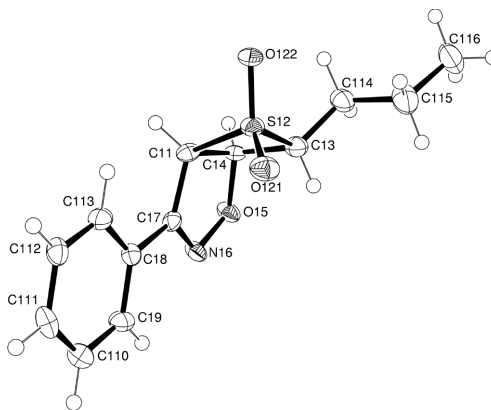
Online 6 September 2002

Comment

The title compound, (I), was prepared as part of a study of the use of carnivore odours in mammal pest control. It is closely related to two previously reported structures: *exo*-7-aza-6-oxa-4-*n*-propyl-3-thiabicyclo[5.4.1^{1,7,1,2,5}]undecane 3,3-dioxide (Woolhouse *et al.*, 1993) and the *exo*-stereoisomer *exo*-7-phenyl-3-*n*-propyl-5-oxa-2-thia-6-azabicyclo[3.2.0^{1,4}]hept-6-ene 2,2-dioxide (Gainsford & Woolhouse, 2002), being formed from a thiete sulfone (Gainsford & Woolhouse, 1994).



The crystal structure of (I) consists of two nearly identical independent molecules (one of these is shown in Fig. 1) and their centrosymmetrically related molecules. Most intermolecular interactions are between each independent molecule and their centrosymmetrically related molecules, *e.g.*

**Figure 1**

The molecular structure of one of the two independent molecules of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms have arbitrary radii.

C113—H113···O122ⁱ, with C113···O122ⁱ = 3.303 (5) Å [symmetry code: (i) 1 - x, 1 - y, 1 - z], making two infinite chains along the *b* crystal axis. The Cj1—Cj4—Oj5—Nj6—Cj7 five-membered rings, where *j* = 1 or 2, have envelope conformations, with flap atoms Cj4 at distances of 0.222 (7) and 0.185 (8) Å from the four-atom plane for *j* = 1 and 2, respectively. The four-membered C₃S fused rings are not planar, with mean deviations from the mean plane of 0.0787 (3) and 0.0487 (3) Å for *j* = 1 and 2, respectively. The angles between the mean planes through the two rings are identical at 66.0 (3)°. This is unlike the *exo*-molecule where the fused four- and five-membered rings are planar (Gainsford & Woolhouse, 2002). The pendant planar phenyl rings Cj8—Cj13 are twisted by 5.2 (2) and 6.9 (3)° for *j* = 1 and 2, respectively, from their bound five-membered ring. The same comments about the oxygen Oj5 binding in fused-ring systems constructed by cycloaddition apply (see Gainsford & Woolhouse, 2002).

Experimental

Compound (I) was prepared as described previously by Gainsford & Woolhouse (2002). Crystals were grown from an ethyl acetate–hexane mixture.

Crystal data

C ₁₃ H ₁₅ NO ₃ S	Z = 4
<i>M_r</i> = 265.32	<i>D_x</i> = 1.374 Mg m ⁻³
Triclinic, P1	Mo <i>K</i> α radiation
<i>a</i> = 5.282 (4) Å	Cell parameters from 24 reflections
<i>b</i> = 11.241 (3) Å	<i>θ</i> = 5.8–14.6°
<i>c</i> = 22.588 (6) Å	<i>μ</i> = 0.25 mm ⁻¹
<i>α</i> = 79.65 (2)°	<i>T</i> = 130 (2) K
<i>β</i> = 89.67 (4)°	Needle, colourless
<i>γ</i> = 76.58 (3)°	0.84 × 0.32 × 0.18 mm
<i>V</i> = 1282.4 (11) Å ³	

Data collection

Siemens/Nicolet R3m four-circle diffractometer	<i>θ</i> _{max} = 22.5°
<i>ω</i> scans	<i>h</i> = 0 → 5
3583 measured reflections	<i>k</i> = -11 → 11
3157 independent reflections	<i>l</i> = -24 → 24
2378 reflections with <i>I</i> > 2σ(<i>I</i>)	3 standard reflections every 97 reflections
<i>R</i> _{int} = 0.035	intensity decay: none

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0097P)^2 + 3.7137P]$
$R[F^2 > 2\sigma(F^2)] = 0.057$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.121$	(Δ/σ) _{max} < 0.001
<i>S</i> = 1.16	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
3157 reflections	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$
316 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.0068 (8)

Table 1

Selected geometric parameters (Å, °).

S12—O121	1.425 (4)	S22—O221	1.425 (4)
S12—O122	1.434 (3)	O25—N26	1.409 (5)
S12—C11	1.822 (5)	N16—C17	1.287 (6)
O121—S12—O122	118.9 (2)	C17—N16—O15	110.8 (4)
C11—S12—C13	80.5 (2)	C14—C11—S12	88.9 (3)
C14—O15—N16—C17	9.8 (5)	C13—S12—C11—C17	91.5 (4)
C23—S22—C21—C24	-6.3 (3)	C23—S22—C21—C27	95.6 (4)

All H atoms were constrained to ride on their parent atom, with a *U*_{iso} value 1.2 times the *U*_{eq} value of the parent atom.

Data collection: *SHELXTL* (Siemens, 1983); cell refinement: *SHELXTL*; data reduction: *SHELXTL*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* in *WinGX* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997) and *PLATON* (Spek, 1990).

The authors thank Dr J. Wikaira and Professor Ward T Robinson of the University of Canterbury for their assistance.

References

- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Gainsford, G. J. & Woolhouse, A. D. (1994). *Acta Cryst.* **C50**, 606–607.
 Gainsford, G. J. & Woolhouse, A. D. (2002). *Acta Cryst.* **E58**, o715–o716.
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
 Siemens (1983). *R3M Software*. Version 4.11. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.
 Woolhouse, A. D., Gainsford, G. J. & Crump, D. R. (1993). *J. Heterocycl. Chem.* **30**, 873–880.