Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

exo-7-Phenyl-3-*n*-propyl-5-oxa-2-thia-6-azabicyclo[3.2.0^{1,4}]hept-6-ene 2,2-dioxide

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Key indicators

Single-crystal X-ray study
T = 130 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.092
Data-to-parameter ratio = 12.5

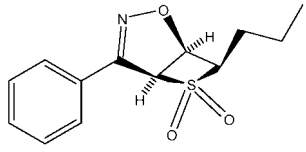
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 14 May 2002
Accepted 30 May 2002
Online 8 June 2002

The title compound, $C_{13}H_{15}NO_3$, contains the novel *exo*-thiabicyclo[3.2.0^{1,4}]hept-6-ene ring system, with pendant phenyl and *n*-propyl substituents. Both fused rings in the bicyclic system are planar, their planes forming a dihedral angle of 65.6 (1)°.

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 a previously reported structure of *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), which had been obtained from a thiete sulfone (Gainsford & Woolhouse, 1994).



(I)

The crystal structure of (I) is built of isolated molecules (Fig. 1) associated into infinite chains along the *b* axis of the crystal *via* weak intermolecular contacts C14—H14*A*···O5^{i} [symmetry code: (i) 1/2−*x*, 1/2+*y*, 1/2−*z*], with H14*A*···O5 and C14···O5 distances of 2.56 (2) and 3.343 (3) Å, respectively.

The fused four- and five-membered rings are each planar, with average deviations of 0.015 (2) and 0.011 (2) Å, respectively; their least-squares planes form a dihedral angle of 65.6 (1)°. The pendant planar phenyl ring (C8–C13) is twisted by 7.3 (1)° from the five-membered C_3NO ring.

Only two other compounds have been reported [Allen & Kennard (1993) and *ConQuest* (Cambridge Crystallographic Data Centre, 2002)] with an oxygen bound to the C_3SO_2 ring. Each of these (Beagley *et al.*, 1992; Adiwidjala *et al.*, 2000) have the oxygen bound to the carbon remote from the sulfur heteroatom, as is found when the fused ring system is constructed by cycloaddition.

Experimental

To an ethereal solution of 2-*n*-propyl-Δ^3-thiete sulfone (0.68 g, 4.7 mmol) and benzhydroxamoyl chloride (0.8 g, 5.1 mmol) at 273 K was added, dropwise, a solution of triethylamine (0.52 g, 5.1 mmol) in ether. The solution was stirred for 4 h at ambient temperature, then filtered and concentrated. The compound was obtained with the *endo*-fused stereoisomer (0.62 g, 50%) by flash chromatography over silica (0.42 g, 34% yield); crystals were grown from an ethylacetate/hexane mixture.

Crystal data

$C_{13}H_{15}NO_3S$
$M_r = 265.32$
Monoclinic, $P2_1/n$
$a = 12.593\ (5)$ Å
$b = 5.081\ (2)$ Å
$c = 20.169\ (8)$ Å
$\beta = 104.028\ (15)^\circ$
$V = 1252.0\ (9)$ Å^3
$Z = 4$
$D_x = 1.408$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
$\theta = 5.6$–14.2°
$\mu = 0.26$ mm^{-1}
$T = 130\ (2)$ K
Needle, colourless
$0.44 \times 0.19 \times 0.04$ mm

Data collection

Siemens/Nicolet *R*3*m* four-circle diffractometer
ω scans
Absorption correction: none
2933 measured reflections
2794 independent reflections
1801 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 27.9^\circ$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 6$
$l = -26 \rightarrow 25$
3 standard reflections
every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.092$
$S = 0.85$
2794 reflections
223 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.69$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S2—O21	1.4451 (17)	O5—C4	1.453 (2)
S2—O22	1.4413 (15)	N6—C7	1.298 (2)
S2—C1	1.834 (2)	C1—C7	1.502 (3)
S2—C3	1.835 (2)	C1—C4	1.545 (3)
O5—N6	1.411 (2)	C3—C4	1.557 (3)
O22—S2—O21	118.61 (9)	C4—C1—S2	89.86 (13)
C1—S2—C3	80.68 (10)	C4—C3—S2	89.46 (13)
N6—O5—C4	109.94 (14)	O5—C4—C1	104.82 (17)
C7—N6—O5	110.41 (16)	O5—C4—C3	113.08 (17)
C7—C1—C4	101.85 (16)	C1—C4—C3	99.92 (16)
C7—C1—S2	112.64 (15)	N6—C7—C1	112.90 (18)
C4—O5—N6—C7	−2.9 (2)	O5—N6—C7—C8	177.53 (17)
S2—C1—C4—O5	−114.97 (14)	S2—C1—C7—N6	95.10 (19)
C7—C1—C4—C3	115.42 (17)	S2—C1—C7—C8	−80.6 (2)

All H atoms were refined with isotropic displacement parameters. The C—H bonds are in the range 0.92 (3)–1.01 (3) Å.

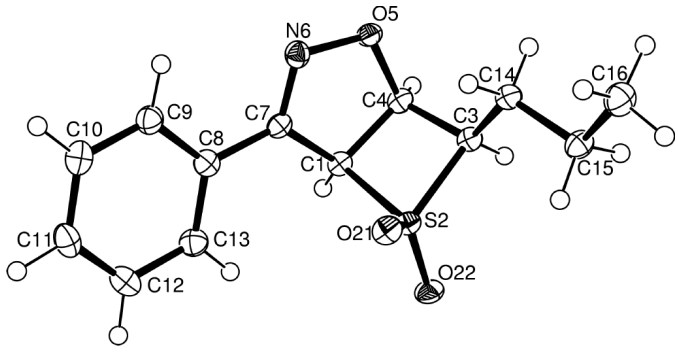


Figure 1
The molecular structure of (I) (Farrugia, 1997). Displacement ellipsoids are drawn at the 50% probability level. H atoms have arbitrary radii.

Data collection: *R*3*M Software* (Siemens, 1983); cell refinement: *R*3*M Software*; data reduction: *R*3*M Software*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 in *WinGX* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 1990).

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

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