

Andris J. Liepa,^{a*} Saba Jahangiri,^a Gary D. Fallon,^b Craig M. Forsyth^b and Andrew C. Warden^a

^aCSIRO Molecular and Health Technologies, Bag 10 Clayton South, Victoria 3169, Australia, and ^bSchool of Chemistry, Monash University, Wellington Rd., Clayton, Victoria 3800, Australia

Correspondence e-mail: andy.liepa@csiro.au

Key indicators

Single-crystal X-ray study
T = 123 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.042
wR factor = 0.102
Data-to-parameter ratio = 18.4

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

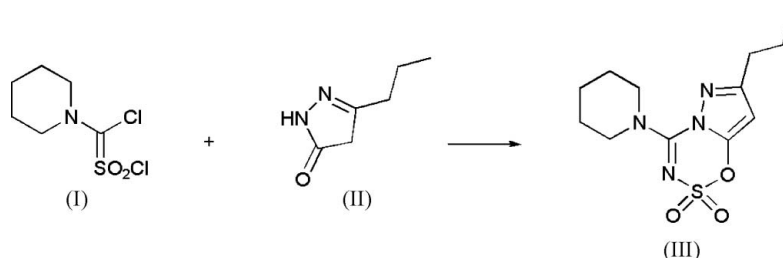
7-(Piperidin-1-yl)-2-propyl-4-oxa-5-thia-1,6,7a-triazaindene 5,5-dioxide, a derivative of a new ring system

The title compound, $\text{C}_{12}\text{H}_{18}\text{N}_4\text{O}_3\text{S}$, is the first example of a fused dicyclic hexahydro-4-oxa-5-thia-1,6,7a-triazaindene ring system. The structure comprises two molecules in the asymmetric unit, which differ in terms of the orientation of their pendant *n*-propyl groups.

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Comment

The dichloro compound, (I), has been demonstrated to undergo attack by 1,2- and 1,3-dinucleophilic species to afford new or otherwise rare heterocycles (Fallon, Francis *et al.*, 2005; Fallon, Jahangiri *et al.*, 2005; Markovskii *et al.*, 1974; Liepa *et al.*, 2006). The title new ring system, (III), was prepared from dichloride (I) with pyrazolone (II) (see scheme). In order to assign accurately the regiochemistry of nucleophilic attack, crystallographic analysis of the product was necessary.



The structure of (III) (Fig. 1) has two independent but closely similar molecules in the asymmetric unit (denoted *A* and *B*), and confirms the presence of the oxathiazaindene ring system. The two structures are differentiated by their *n*-propyl chains (from the five-membered rings), which form opposite handed screws. The cyclic S—O bond is longer than those of the SO₂ moiety (Table 1). There is a C12—H12B···N3 interaction of 2.914 (2) Å for molecule *A* and a similar one for molecule *B* [C20—H20B···N7 = 2.851 (2) Å; Table 2]. The oxothiadiazine ring adopts an envelope conformation, with atom S1 deviating by 0.559 (3) Å from the plane defined by C1/N2/C7/N1 in molecule *A*, and atom S2 deviating by 0.499 (3) Å from the C13/N6/C19/N5 plane in molecule *B*. Also noteworthy is the fact that atoms O3 and O6 also deviate from the aforementioned planes, by 0.224 (3) and 0.245 (3) Å, respectively. Interestingly, the two fused rings in both *A* and *B* molecules (excluding S and O atoms) are nearly coplanar: the dihedral angle in molecule *A* is 0.79 (12)°, and that in molecule *B* is 0.88 (14)°. This is in contrast with the octahydro-1-thia-2,3a,8a-triazacyclopenta[*a*]indene ring system recently reported by our group, which is formed by a similar method (Liepa *et al.*, 2006), where there is a fold of 41.0 (6)° around the shared N—N bond.

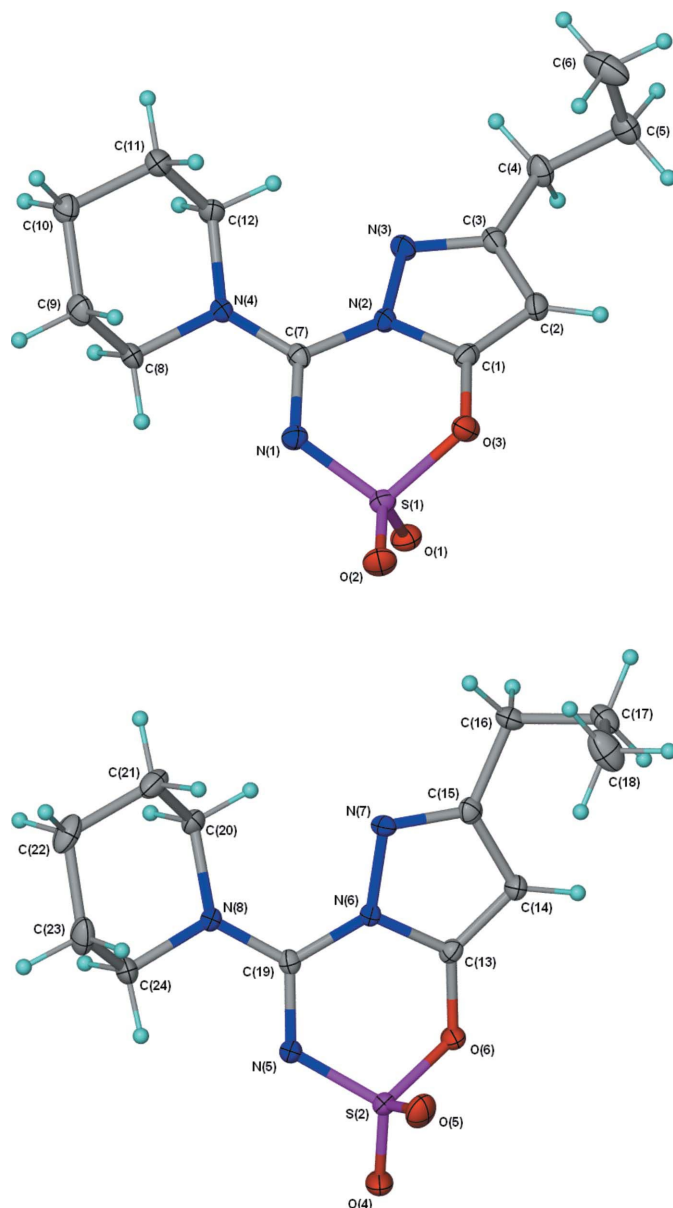


Figure 1

A view of the independent molecules of (III), showing the atom-labelling scheme. Molecule *A* is at the top and molecule *B* is at the bottom. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

A solution of 5-propyl-2,4-dihydropyrazol-3-one (0.26 g, 2 mmol) and the dichloro compound, (I) (0.72 g, 3 mmol), in 1,3-dimethyl-3,4,5,6-tetrahydro-2(1*H*)-pyrimidinone (2 ml) was heated at 373 K for 4 h. The cooled solution was stirred into water (30 ml) and extracted with ethyl acetate (20 ml). The organic phase was separated and washed with water, the solvent removed under reduced pressure, and the residue dissolved and purified by chromatography over silica gel. Elution with dichloromethane gave a fraction containing the product, (III), which was obtained as a colourless solid (0.23 g, 38%) following removal of the solvent. Recrystallization from propan-2-ol gave colourless prisms of (III) (m.p. 353–355 K). Analysis, found: C 48.4, H 6.1, N 19.0, S 10.8%; $C_{12}H_{18}N_4O_3S$ requires: C 48.3, H 6.1, N 18.8,

S 10.8%. 1H NMR ($CDCl_3$, δ , p.p.m.): 5.84 (1H, s, C=CH), 4.31 (2H, br s, NCH₂), 3.76 (2H, br s, NCH₂), 2.57 (2H, t, $J = 7.5$ Hz, CH₂C=N), 1.74 (6H, br s, 3CH₂), 1.67 (2H, sextet, CH₃), 0.97 (3H, t, $J = 7.4$ Hz, CH₂CH₃); ^{13}C NMR ($CDCl_3$, δ , p.p.m.): 13.7, 21.3, 23.9, 25.4, 26.2, 31.0, 48.5, 51.0, 92.5, 146.8, 151.0, 157.3.

Crystal data

$C_{12}H_{18}N_4O_3S$
 $M_r = 298.36$
 Triclinic, $P\bar{1}$
 $a = 9.6834$ (1) Å
 $b = 11.9792$ (2) Å
 $c = 13.5579$ (2) Å
 $\alpha = 82.820$ (1)°
 $\beta = 69.571$ (1)°
 $\gamma = 68.184$ (1)°
 $V = 1368.22$ (4) Å³
 $Z = 4$
 $D_x = 1.448$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.25$ mm⁻¹
 $T = 123$ (2) K
 Prism, colourless
 $0.25 \times 0.20 \times 0.18$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
 Thick-slice ϕ and ω scans
 Absorption correction: none
 18184 measured reflections
 6665 independent reflections
 4307 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.045$
 $\theta_{max} = 28.3^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.102$
 $S = 1.00$
 6665 reflections
 363 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0483P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.29$ e Å⁻³
 $\Delta\rho_{min} = -0.44$ e Å⁻³

Table 1

Selected bond lengths (Å).

S1—O1	1.4248 (13)	S2—O4	1.4168 (13)
S1—O2	1.4161 (12)	S2—O5	1.4253 (13)
S1—O3	1.6511 (12)	S2—O6	1.6410 (13)
S1—N1	1.5845 (13)	S2—N5	1.5825 (13)
N1—C7	1.334 (2)	C13—C14	1.344 (2)
N2—C7	1.401 (2)	O6—C13	1.363 (2)
N3—C3	1.325 (2)	N6—C13	1.383 (2)
C1—C2	1.342 (2)	C14—C15	1.420 (2)
O3—C1	1.364 (2)	N5—C19	1.329 (2)
N2—N3	1.3908 (18)	N6—N7	1.3901 (18)
N2—C1	1.381 (2)	N6—C19	1.383 (2)
C2—C3	1.420 (2)	N7—C15	1.320 (2)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C12—H12B \cdots N3	0.99	2.19	2.914 (2)	129
C20—H20B \cdots N7	0.99	2.14	2.851 (2)	127

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å and with $U_{iso}(H) = 1.2\text{--}1.5U_{eq}(C)$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997);

molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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7-(Piperidin-1-yl)-2-propyl-4-oxa-5-thia-1,6,7a-triazindene 5,5-dioxide, a derivative of a new ring system. Corrigendum

Andris J. Liepa,^{a*} Saba Jahangiri,^a Gary D. Fallon,^b Craig M. Forsyth^b and Andrew C. Warden^a

^aCSIRO Molecular and Health Technologies, Bag 10 Clayton South, Victoria 3169, Australia, and ^bSchool of Chemistry, Monash University, Wellington Rd., Clayton, Victoria 3800, Australia

Correspondence e-mail: andy.liepa@csiro.au

In the paper by Liepa, Jahangiri, Fallon, Forsyth & Warden [*Acta Cryst.* (2006), **E62**, o4470–o4472], there is an error in the reaction scheme. The correct scheme is given below.

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