Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Dimethylamino-1,1-dioxo-4,5,6,7-tetrahydro-1*H*-λ^6-thia-2,3a,8a-triazacyclopenta[*a*]inden-8-one, a derivative of a new ring system

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.091
Data-to-parameter ratio = 17.5

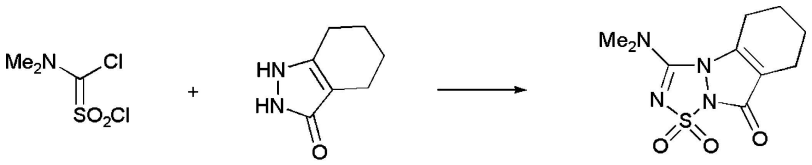
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 7 June 2006
Accepted 23 June 2006

The title compound, $C_{10}H_{14}N_4O_3S$, is the first example of the fused tricyclic octahydro-1-thia-2,3a,8a-triazacyclopenta[*a*]-indene ring system. The S-containing heterocycle adopts a typical envelope conformation, fused at the 2,3-positions to a 2,3a,4,5,6,7-hexahydroindazol-3-one unit.

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). The new ring system (III) was prepared from the reaction of dichloride (I) with tetrahydroindazolone (II). The structure of (III) (Fig. 1) confirms the regiochemistry of nucleophilic attack and comprises a tricyclic system with two fused 5-membered heterocyclic rings, folded by 41.0 (6)° along the N1—N2 bond [defined by the torsion angle C2—N1—N2—C1 141.13 (12)°]. Bond distances and angles (Table 1) are consistent with the assigned bonding (see scheme), with shorter distances for the two double bonds C3═C8 and C2═N3, and the sum of angles around N1 and N2 (341.7 and 336.0°) indicative of pyramidal N atoms. The diazacyclopentenone group is planar [maximum deviation 0.040 (1) Å for atom C8], whilst the thiatriaza ring has a typical envelope conformation, with atom S1 lying 0.174 (2) Å out of the N1/N2/C2/N3 least-squares plane. Notably, the N2—S1 bond distance is significantly longer [by 0.115 (3) Å] than the corresponding N3—S1 bond length. However, these values compare well with data for related heterocycles (Friedrichsen *et al.*, 1983).



(I) (II) (III)

Experimental

N,*N*-Disopropylethylamine (3.12 g, 24 mmol) was added dropwise to a mixture of 2,3a,4,5,6,7-hexahydroindazol-3-one (1.23 g, 9 mmol) and the dichloride (I) (2.4 g, 12 mmol) in 1,3-dimethyl-3,4,5,6-tetrahydro-2(1*H*)-pyrimidinone (6 ml). The solution was stirred overnight, diluted with water (6 ml), filtered and washed with water to give the product (1.9 g, 59%). Recrystallization from dichloromethane/acetonitrile (1:2) gave colourless needles (m.p. 757–759 K). Analysis found: C 44.5, H 5.3, N 20.9, S 11.9; $C_{10}H_{14}N_4O_3S$ requires: C 44.4, H

5.2, N 20.7, S 11.9%. ^{1}H NMR ($CDCl_3$) δ_H 3.19 (3H, *s*, NCH_3), 3.16 (3H, *s*, NCH_3), 2.67–3.64 (2H, *m*, $CH_2C{=}C$), 2.45–2.42 (2H, *m*, $CH_2C{=}C$), 1.80–1.66 (4H, *m*, 2 × CH_2).

Crystal data

$C_{10}H_{14}N_4O_3S$	$Z = 4$
$M_r = 270.31$	$D_x = 1.513$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.6327$ (1) Å	$\mu = 0.28$ mm^{-1}
$b = 16.3610$ (3) Å	$T = 123$ (2) K
$c = 8.4883$ (1) Å	Needle, colourless
$\beta = 98.117$ (1)°	0.20 × 0.08 × 0.08 mm
$V = 1186.87$ (3) Å^3	

Data collection

Nonius KappaCCD diffractometer	2893 independent reflections
Thick-slice φ or ω scans	2182 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.046$
17785 measured reflections	$\theta_{max} = 28.3°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0435P)^2 + 0.299P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.039$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.091$	$\Delta\rho_{max} = 0.33$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.45$ e Å^{-3}
2893 reflections	
165 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—N2	1.4295 (17)	C2—N1	1.4079 (19)
N2—S1	1.7194 (13)	C2—N3	1.316 (2)
N3—S1	1.6038 (14)	C3—N1	1.421 (2)
C1—N2	1.439 (2)	C3—C8	1.344 (2)
C1—C8	1.439 (2)		
S1—N3—C2	111.01 (11)	N2—S1—N3	95.62 (6)
N1—N2—S1	107.03 (9)	N2—N1—C2	108.48 (12)
N1—N2—C1	107.92 (12)	N2—N1—C3	106.40 (12)
N1—C2—N3	116.80 (14)	N2—C1—C8	105.65 (13)
N1—C3—C8	110.16 (14)	C1—C8—C3	109.49 (14)

H atoms were placed in calculated positions, with C—H distances of 0.98 and 0.99 Å, and were included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$, or $1.5U_{eq}(C)$ for methyl H atoms.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; 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).

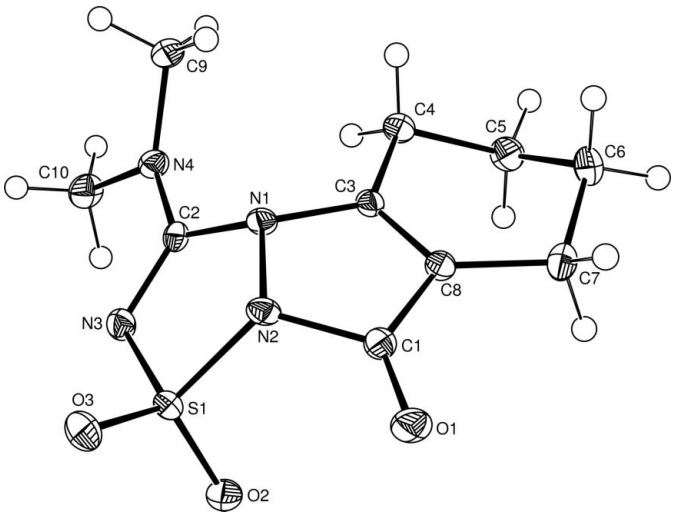


Figure 1
View of (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

We acknowledge the Australian Research Council for financial support.

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Andris J. Liepa,[a]* Saba Jahangiri,[a] Gary D. Fallon[b] and Craig M. Forsyth[b]

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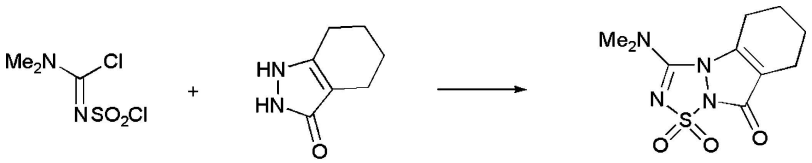
In the paper by Liepa, Jahangiri, Fallon & Forsyth [*Acta Cryst.* (2006), E**62**, o3170–o3171], there is an error in the reaction scheme. The correct scheme is given below.



(I) (II) (III)