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(2,3-Dimethyl-7,7-dioxo-7H-4-oxa-7λ^6-thia-1,6,7a-triazainden-5-yl)diethyamine

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The title compound, $C_{10}H_{16}N_4O_3S$, is the first example of a fused bicyclic oxathiatriazaindene ring system The six-membered heterocycle adopts a boat conformation. Molecules are linked by weak C—H···O hydrogen bonds.

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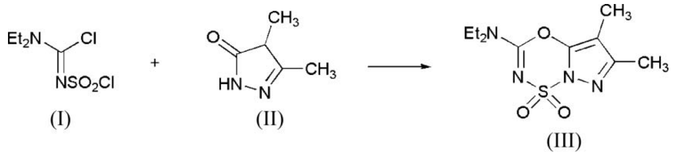
Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.090
Data-to-parameter ratio = 18.7

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

Comment

The dichloro compound, (I), is a versatile intermediate for the synthesis of new or otherwise rare heterocycles by addition of 1,2 and 1,3-dinucleophilic species (Fallon, Francis *et al.*, 2005; Fallon, Jahangiri *et al.*, 2005; Markovskii *et al.*, 1974; Liepa, Jahangiri, Fallon, & Forsyth, 2006; Liepa, Jahangiri, Fallon, Forsyth & Warden, 2006). We now report the synthesis of a new ring system, (III), prepared from dichloride (I) with pyrazolone (II). As with previous examples, the regiochemistry of the addition could not be established from NMR data and elucidation by X-ray crystallography was necessary.



The structure of (III) (Fig. 1) represents a 4-oxa-7-thia-1,6,7a-triazaindene ring system. The six-membered heterocycle adopts a boat conformation, with atoms S1 and O1 0.32 and 0.16 Å, respectively, above the N2/C3/C4/N3 plane. The O1/C4/N3/S1 and O1/C3/N2/S1 planes subtend an angle of 22.06 (6)°. The S—N bonds are of unequal length, with S1—N2 *ca* 0.095 Å longer than S1—N3. Similar bond-length differences (<0.17 Å) have been observed in related bicyclic dioxothiaazaindene compounds (Fallon, Francis *et al.*, 2005). The fused pyrazole ring is planar (maximum deviation 0.03Å). Molecules of (III) are connected by weak C—H···O hydrogen bonds, forming isolated chains with a $C(7)$ motif (Etter *et al.*, 1990).

Experimental

N,*N*-Diisopropylethylamine (1.04 g, 8 mmol) was added dropwise to a mixture of 4,5-dimethyl-2,4-dihydropyrazol-3-one (0.33 g, 3 mmol) and (I) (0.92 g, 4 mmol) in 1,3-dimethyl-3,4,5,6-tetrahydropyrimidin-2(1*H*)-one (4 ml). The solution was stirred overnight, poured into water (50 ml) and extracted with ethyl acetate (20 ml). The organic phase was separated, washed with water, the solvent removed under reduced pressure and the residue was dissolved in dichloromethane and purified by chromatography over silica gel. Elution with dichloromethane gave fractions containing two successive products. The more polar material was obtained as a colourless solid (0.12 g, 22%) following removal of the solvent. Recrystallization from acetonitrile gave colourless prisms [m.p. 379 K (dec)]. Analysis found: C 44.1, H 5.9, N 20.6, S 11.6%; $C_{10}H_{16}N_4O_3S$ requires: C 44.1, H 5.9, N 20.6, S 11.8%. NMR: δ_H ($CDCl_3$) 3.55 (2H, *q*, *J* = 7.3 Hz, NCH_2), 3.54 (2H, *q*, *J* = 7.3 Hz, NCH_2), 2.23 (3H, *s*, CH_3), 1.93 (3H, *s*, CH_3), 1.30 (3H, *t*, *J* = 7.3 Hz, NCH_2CH_3), 1.27 (3*H*, *t*, *J* = 7.3 Hz, NCH_2CH_3); δ_C ($CDCl_3$) 152.6, 149.2, 144.6, 97.4, 44.7, 43.0, 13.6, 12.7, 12.2, 6.0.

Crystal data

$C_{10}H_{16}N_4O_3S$
M_r = 272.33
Monoclinic, $C2/c$
a = 23.2639 (2) Å
b = 8.6792 (1) Å
c = 16.0670 (2) Å
β = 128.710 (1)°
V = 2531.45 (6) Å^3
Z = 8
D_x = 1.429 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 123 (2) K
Prism, colourless
0.31 × 0.27 × 0.23 mm

Data collection

Nonius KappaCCD diffractometer
Thick-slice φ and ω scans
Absorption correction: none
13225 measured reflections
3127 independent reflections
2598 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.036
$\theta_{\rm max}$ = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.090
S = 1.06
3127 reflections
167 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 2.3884P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.34 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.41 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C9—H9*A*⋯O3$^{\rm i}$	0.99	2.50	3.4782 (19)	168

Symmetry code: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$.

All H atoms were initially located in a difference Fourier map but were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.95–1.00 Å and $U_{\rm iso}$(H) = 1.2 or 1.5 times $U_{\rm eq}$(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).

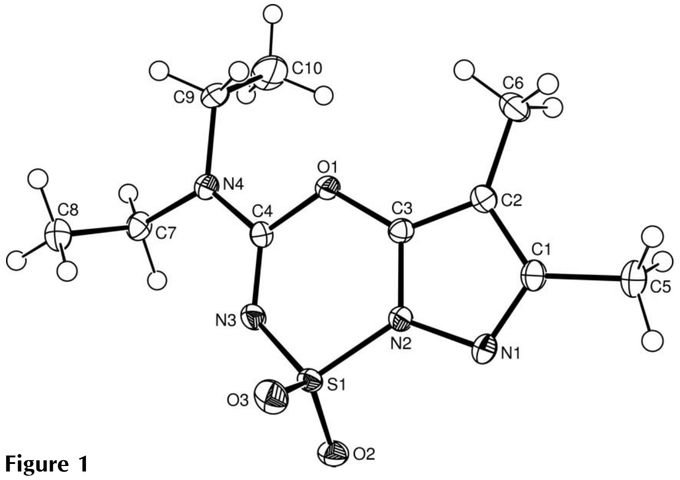


Figure 1
A view of the molecule of (III), with ellipsoids drawn at the 50% probability level.

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