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5-Azido-3,3-dimethyl-2-oxo-1-thia-4,6-diazaindan 1,1-dioxide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.087
Data-to-parameter ratio = 12.1

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

In the title molecule, $C_7H_7N_5O_3S$, all of the atoms in the fused ring system, except for the O atom, are essentially in the same plane. The O atom forms the flap of the envelope conformation of the sultone ring. In the absence of any hydrogen-bond or π–π stacking interactions, the structure appears to be stabilized only by normal van der Waals interactions.

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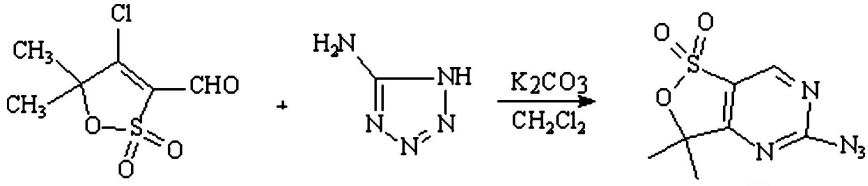
Comment

The Vilsmeier reagent, HCONR_1R_2/$POCl_3$ (where R_1 and R_2 can be CH_3—, CH_3CH_2— or Ph—), is extensively used in the synthesis of aldehyde derivatives and formamidines (Meth-Cohn, 1991). Recently, we found that the Vilsmeier reaction applied to acetylphosphonate leads stereospecifically to (*Z*)-β-phosphonyl-β-chlorovinylaldehyde (Qian *et al.*, 2000). We then applied this syhthesis to β-carbonyl sultone (Ingate *et al.*, 1997). The action of DMF/$POCl_3$ on β-carbonyl sultone at room temperature afforded a single product, *viz*. 4-chloro-5,5-dimethyl-3-formyl-1,2-oxathiolene 2,2-dioxide, (1), in 64.5% yield. The reaction of (1) with aminotetrazole (2) (see scheme) gave the title compound, (3). This compound was characterized by ^{1}H NMR and elemental analyses and its crystal structure is reported here.



(1) (2) (3)

The title molecule is shown in Fig. 1. The molecule comprises a pyrimidine ring fused to a sultone ring. This is in contrast to the product formed from a similar reaction when 5-phenyl-4-amino-3-mercapto-(4*H*)-1,2,4-triazole is used instead of aminotetrazole (Tian & Liu, 2004). The bond lengths and angles in (3) are in the normal ranges (Table 1). All of the atoms in the fused ring system (N1/N2/C4/C5/C6/C7/S1/C3), except for atom O3, are essentially in the same plane (r.m.s. deviation = 0.006 Å). Atom O3 forms the flap of the envelope conformation of the five-membered sultone ring, deviating by 0.304 (2) Å from the plane formed by atoms S1/C3/C4/C7 (r.m.s. deviation = 0.001 Å). The azide group is bent slightly out of the plane formed by atoms N1/N2/C4/C5/C6/C7/S1/C3 by 0.037 (2), 0.184 (2) and 0.325 (3) Å for atoms N3, N4 and N5, respectively.

The are no significant hydrogen-bond or π–π stacking interactions and the crystal structure (Fig. 2) appears to be stabilized by normal van der Waals forces.

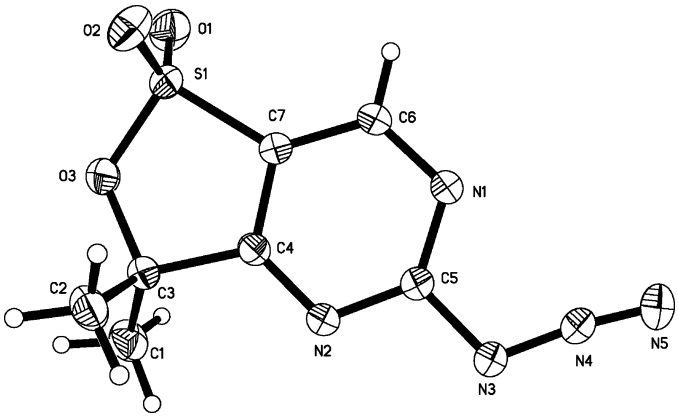


Figure 1
View of the title compound, shown with 40% probability displacement ellipsoids.

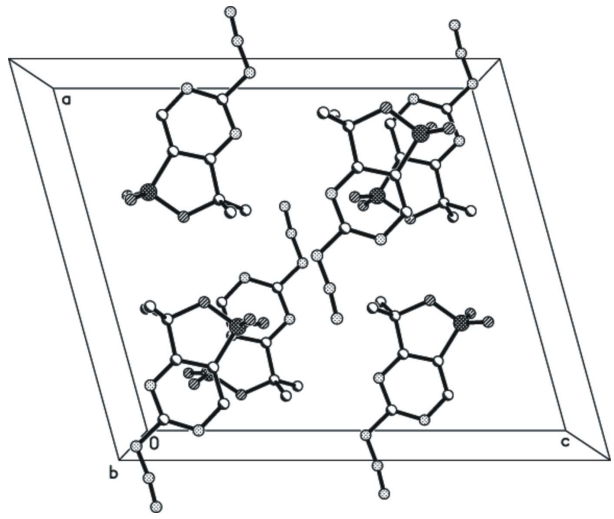


Figure 2
The molecular packing of (3), viewed along the *b* axis. H atoms have been omitted.

Experimental

A solution of compound (1) (1 mmol, 0.21 g) in dichloromethane (5 ml) was added dropwise a solution of aminotetrazole (2) (1 mmol, 0.16 g) in water (2 ml) at 273–283 K, then K_2CO_3 (1 mmol, 0.14 g) in water (2 ml) was added dropwise at this temperature. The reaction mixture was kept at room temperature for 2–3 h. The aqueous layer was extracted with dichloromethane. The combined organic layers were washed with saturated brine, dried, filtered and concentrated. The residue was seperated by silica gel to afford the title compound, which was purified by recrystallization from AcOEt–cyclohexane. Colourless single crystals were grown from a solution of AcOEt–cyclohexane (1:3). ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.91 (*s*, 1H, C6—H), 1.82 [*s*, 6H, $(CH_3)_2$]. Analysis calculated for $C_7H_7N_5O_3S$: C 34.85, H 2.93, N,29.03%; found: C 34.77, H 2.88, N 28.93%.

Crystal data

$C_7H_7N_5O_3S$
M_r = 241.24
Monoclinic, $C2/c$
a = 14.4162 (16) Å
b = 8.5691 (10) Å
c = 17.0016 (19) Å
β = 105.365 (1)°
V = 2025.2 (4) Å^3
Z = 8
D_x = 1.582 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3013 reflections
θ = 2.5–27.8°
μ = 0.32 mm^{-1}
T = 293 (2) K
Block, colourless
0.22 × 0.16 × 0.10 mm

Data collection

Bruker APEX-II CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.933, T_{max} = 0.969
5286 measured reflections
1784 independent reflections
1587 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.0°
$h = -17 \rightarrow 17$
$k = -7 \rightarrow 10$
$l = -17 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.087
S = 1.06
1784 reflections
148 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 1.3512P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0045 (5)

Table 1
Selected geometric parameters (Å, °).

N1—C6	1.328 (2)	N4—N5	1.107 (2)
N1—C5	1.340 (2)	C3—C4	1.514 (2)
N2—C4	1.316 (2)	C4—C7	1.380 (2)
N2—C5	1.338 (2)	C6—C7	1.379 (2)
N3—N4	1.256 (2)	C6—H6	0.9300
N3—C5	1.394 (2)		
C6—N1—C5	116.35 (14)	N2—C5—N1	128.13 (15)
C4—N2—C5	113.70 (13)	N2—C5—N3	113.13 (14)
N4—N3—C5	113.33 (14)	N1—C5—N3	118.74 (14)
N5—N4—N3	172.71 (19)	N1—C6—C7	120.17 (15)
C2—C3—C4	111.28 (14)	N1—C6—H6	119.9
C1—C3—C4	112.70 (14)	C7—C6—H6	119.9
N2—C4—C7	123.46 (15)	C6—C7—C4	118.17 (15)
N2—C4—C3	122.62 (14)	C6—C7—S1	132.21 (13)
C7—C4—C3	113.92 (14)	C4—C7—S1	109.61 (12)

All H atoms were placed in calculated positions, with C—H = 0.93 or 0.96 Å, and refined using the riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for methyl H atoms.

Data collection: *APEXII* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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