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6-Cyano-3,3-dimethyl-5-methylsulfanyl-1,3-dihydro-2-oxo-1-thia-3b,4,8-triaza-indacene 1,1-dioxide

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Key indicators

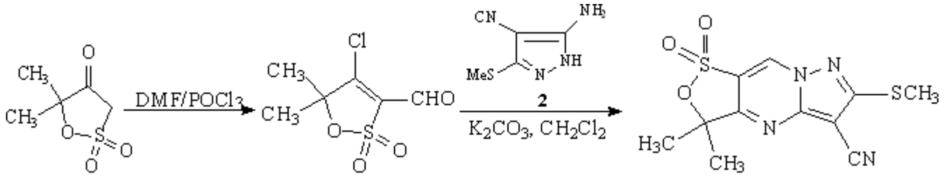
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.039
wR factor = 0.121
Data-to-parameter ratio = 12.4

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

The title compound, $C_{11}H_{10}N_4O_3S_2$, has normal bond lengths and angles. The molecule includes a pyrazole ring, a pyrimidine ring and a sulfone ring. The crystal packing is mainly stabilized by van der Waals interactions.

Comment

Vilsmeier reagents, HCONR_1R_2/$POCl_3$, are extensively used in the synthesis of aldehyde derivatives and formamidines (Meth-Cohn, 1991). Recently, it has been found that application of the Vilsmeier reaction with acetylphosphonates leads stereospecifically to (*Z*)-β-phosphonyl-β-chlorovinylaldehydes (Qian *et al.*, 2000). Chloroformylation of β-carbonyl sulfone (Ingate *et al.*, 1997) with the Vilsmeier reagent DMF/$POCl_3$ afforded the cyclic β-chlorovinylaldehyde 4-chloro-5,5-dimethyl-3-formyl-1,2-oxathiolene 2,2-dioxide, (1). This compound is a very useful intermediate for the synthesis of heterocyclic compounds, and reaction with 5-amino-4-cyano-3-methylsulfanyl pyrazole, (2), gives the title heterocyclic compound, (3). We report here the crystal structure of (3).



(1)

(3)

The molecular structure of (3) is shown in Fig. 1. All bond lengths and angles are normal (Table 1). The crystal packing

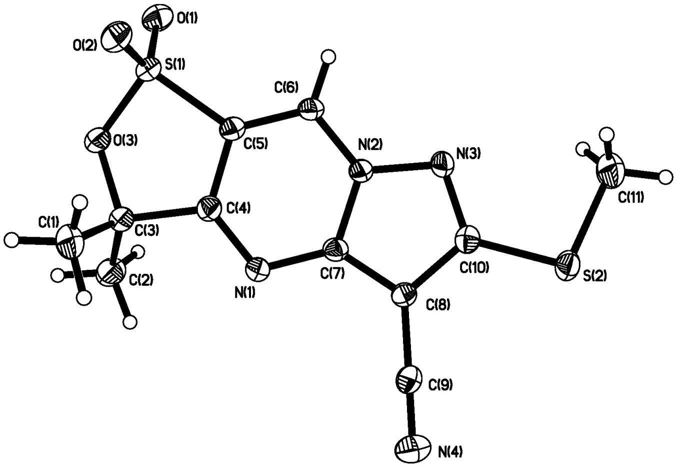


Figure 1
A view of the title compound, showing displacement ellipsoids at the 40% probability level.

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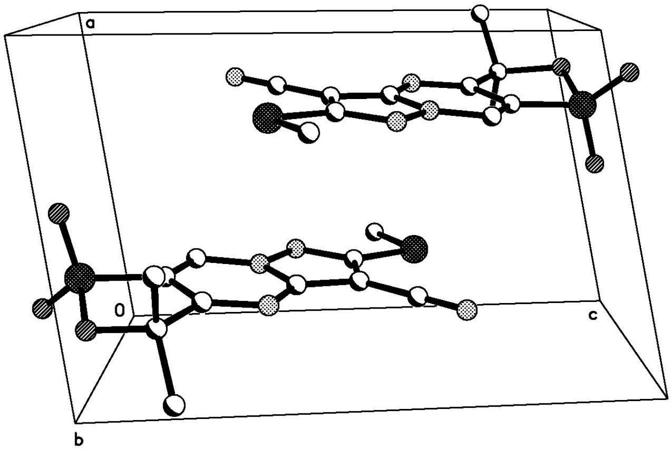

Figure 2
The molecular packing of (3), viewed along the *b* axis. H atoms have been omitted for clarity.

(Fig. 2) is stabilized by van der Waals interactions. As depicted in Fig. 1, there is a six-membered pyrimidine ring, N1/C4/C5/C6/N2/C7, which is formed by the amino group of (2) bonded with atom C4 of (1) and the imino group of (2) bonded with the formyl group of (1), rather than a combination of the amino group of (2) combined with the formyl group of (1), as has been previously observed by Tian & Liu (2004). The short bond lengths of C5—C6, C7—C8 and C4—N1 (Table 1) are indicative of significant double-bond character.

Experimental

A solution of 5-amino-4-cyano-3-methylsulfanyl pyrazole, (2) (1 mmol, 0.16 g), in water (2 ml) was added dropwise to a solution of compound (1) (1 mmol, 0.21 g) in dichloromethane (5 ml) at 273–283 K. K_2CO_3 (1 mmol, 0.14 g) in water (2 ml) was then added dropwise at this temperature. The reaction mixture was kept at room temperature for 2–3 h. The aqueous layer was extracted with dichloromethane and the combined organic layers were washed with saturated brine, dried, filtered and concentrated. The residue was separated by silica gel to afford the title compound. Recrystallization from a solution in ethyl acetate–cyclohexane (1:3) yielded colourless single crystals suitable for X-ray diffraction studies. ^{1}H NMR (300 MHz, $CDCl_3$, δ, p.p.m.): 7.05 (*s*, 1H, H9), 2.75 (*s*, 3H, $-SCH_3$), 1.86 [*s*, 6H, $(CH_3)_2$]. Analysis, calculated for $C_{11}H_{10}N_4S_2O_3$: C 42.57, H 3.25, N 18.05%; found: C 42.40, H 3.39, N 17.98%.

Crystal data

$C_{11}H_{10}N_4O_3S_2$
$M_r = 310.35$
Triclinic, $P\overline{1}$
$a = 7.150\ (2)$ Å
$b = 8.573\ (3)$ Å
$c = 11.171\ (3)$ Å
$\alpha = 98.316\ (4)^\circ$
$\beta = 98.953\ (4)^\circ$
$\gamma = 98.420\ (4)^\circ$
$V = 659.2\ (3)$ Å^3

$Z = 2$
$D_x = 1.564$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1868 reflections
$\theta = 2.4$–27.9°
$\mu = 0.42$ mm^{-1}
$T = 293\ (2)$ K
Prism, colourless
0.38 × 0.16 × 0.10 mm

Data collection

Bruker APEX II CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.784$, $T_{max} = 0.960$
3565 measured reflections

2279 independent reflections
1954 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 25.0^\circ$
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 9$
$l = -13 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.121$
$S = 1.08$
2279 reflections
184 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.9464P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C4	1.315 (4)	N3—C10	1.331 (4)
N1—C7	1.353 (4)	C1—C3	1.513 (5)
N2—C6	1.338 (4)	C4—C5	1.400 (4)
N2—N3	1.374 (3)	C5—C6	1.365 (4)
N2—C7	1.393 (4)	C7—C8	1.377 (4)
O3—S1—C5	93.66 (13)	C6—C5—C4	120.2 (3)
C4—N1—C7	114.5 (2)	C6—C5—S1	130.8 (2)
C6—N2—N3	124.6 (2)	C4—C5—S1	109.0 (2)
C6—N2—C7	123.0 (2)	N2—C6—C5	115.2 (3)
N3—N2—C7	112.4 (2)	N1—C7—C8	132.4 (3)
C10—N3—N2	103.9 (2)	N1—C7—N2	122.1 (2)
O3—C3—C4	102.8 (2)	C8—C7—N2	105.5 (2)
N1—C4—C5	124.9 (3)	C7—C8—C10	105.6 (3)
N1—C4—C3	122.0 (3)	N3—C10—C8	112.6 (3)
C5—C4—C3	113.1 (3)		

All H atoms were placed in calculated positions, with C—H = 0.93 or 0.96 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$ [$1.5U_{eq}$ for methyl].

Data collection: *APEXII* (Bruker, 1998); cell refinement: *APEXII*; data reduction: *SAINT* (Bruker, 1999); 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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