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3,3-Dimethyl-7-phenyl-2-oxa-1,4-dithia-5,6,8,9-tetraaza-3*H*-cyclopenta[*f*]azulene 1,1-dioxide

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The title compound, $C_{14}H_{12}N_4O_3S_2$, has normal bond lengths and angles. The molecule includes a benzene ring, a 1,2,4-triazole ring, a sultone ring and a seven-membered sulfur-containing ring. The crystal packing is mainly stabilized by van der Waals interactions.

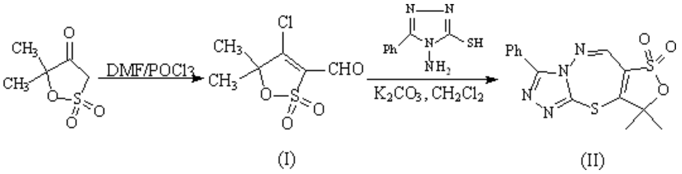
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.056
wR factor = 0.132
Data-to-parameter ratio = 12.9

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

Comment

The Vilsmeier reagent, HCONR_1R_2/$POCl_3$ (R_1 = R_2 = Me), is used extensively in the synthesis of aldehyde derivatives and formamidines (Meth-Cohn, 1991). Recently, we found that the Vilsmeier reaction applied to acetyl phosphonate leads stereospecifically to (*Z*)-β-phosphonyl-β- chlorovinylaldehyde (Qian, 2000). We applied this synthesis to β-carbonyl sultone (Ingate, 1997). Chloroformylation of β-carbonyl sultone 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), (I). Compound (I) is a useful intermediate for the synthesis of heterocyclic compounds. The reaction of (I) with 5-phenyl-4-amino-3-mercapto-(4*H*)-1,2,4-triazole gave the title compound, (II), with a seven-membered ring which is commonly considered unstable because of the high strain in the ring. We report here the crystal structure of (II).



The molecular structure of (II) is shown in Fig. 1. All bond lengths and angles are normal (Table 1) and clearly show that the bonding sequence of the N3/N4/C11/C10/C9/S1/C8 ring is N3—N4═C11—C10═C9—S1—C8. The crystal packing (Fig. 2) is mainly stabilized by van der Waals interactions.

Experimental

To a solution of (I) in dichloromethane (5 ml) was added dropwise a solution of 5-phenyl-4-amino-3-mercapto-(4*H*)-1,2,4-triazole (0.19 g) in water (3 ml) at 283 K, and then K_2CO_3 (0.14 g) in water (2 ml) was added dropwise. 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 separated by silica gel to afford the title compound, which was purified by recrystallization from ethyl acetate–petroleum ether.

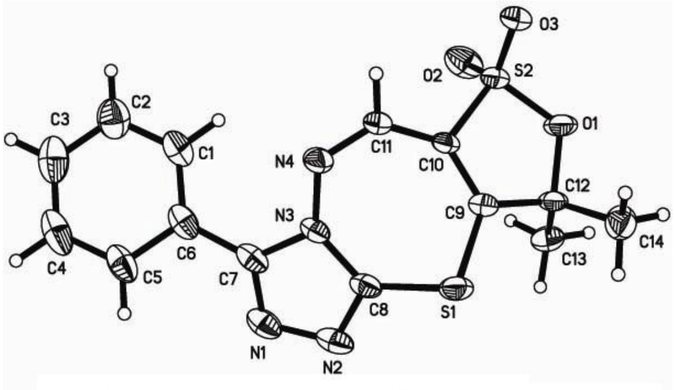


Figure 1
View of the title compound, with 40% probability displacement ellipsoids.

Crystal data

$C_{14}H_{12}N_4O_3S_2$
$M_r = 348.40$
Monoclinic, $P2_1/n$
$a = 7.290$ (2) Å
$b = 22.131$ (7) Å
$c = 9.513$ (3) Å
$\beta = 90.115$ (5)°
$V = 1534.8$ (8) Å^3
$Z = 4$

$D_x = 1.508$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 796 reflections
$\theta = 3.5$–24.2°
$\mu = 0.37$ mm^{-1}
$T = 293$ (2) K
Prism, pale yellow
0.20 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.925$, $T_{\max} = 0.957$
7903 measured reflections

2712 independent reflections
1876 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.045$
$\theta_{\max} = 25.0°$
$h = -8 \rightarrow 4$
$k = -26 \rightarrow 26$
$l = -11 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.132$
$S = 1.07$
2712 reflections
210 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.8529P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.723 (4)	N4—C11	1.275 (4)
S1—C9	1.748 (3)	C9—C10	1.338 (4)
N3—C8	1.380 (4)	C10—C11	1.448 (5)
N3—N4	1.389 (4)		
C8—S1—C9	98.58 (17)	C10—C9—S1	123.9 (3)
O2—S2—O3	117.38 (18)	C12—C9—S1	119.8 (2)
C7—N3—C8	105.7 (3)	C9—C10—C11	131.2 (3)
C7—N3—N4	121.8 (3)	C9—C10—S2	108.2 (3)
C8—N3—N4	132.1 (3)	C11—C10—S2	120.2 (2)
C11—N4—N3	118.2 (3)	N4—C11—C10	131.5 (3)
N2—C8—S1	122.6 (3)	N4—C11—H11	114.2
N3—C8—S1	127.6 (3)	C10—C11—H11	114.2
C10—C9—C12	116.2 (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 $U_{\rm iso}$ (H) = $1.2U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

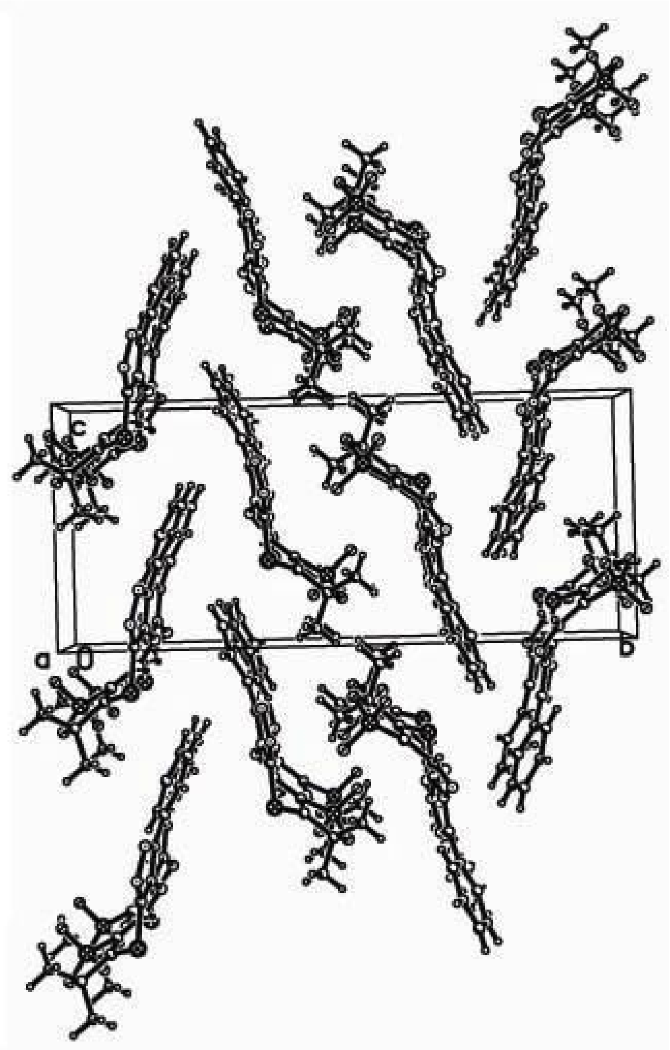


Figure 2
The molecular packing of (II), viewed along the *b* axis.

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