![CrossMark]

(Z)-3-Allyl-5-(4-fluorobenzylidene)-2-sulfanylidene-thiazolidin-4-one

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In the title compound, $C_{13}H_{10}FNOS_2$, the sulfanylidenethiazolidine ring and the benzylidene ring are almost coplanar [dihedral angle between the two planes = 0.1 (2)°]. The mean plane through the allyl group is nearly perpendicular to the sulfanylidenethiazolidine ring, as indicated by the dihedral angle of 69.5 (5)° between them. In the crystal, molecules are linked together by weak C—H⋯O hydrogen bonds involving the same acceptor atom, forming dimers parallel to $(1\bar{2}2)$.

3D view
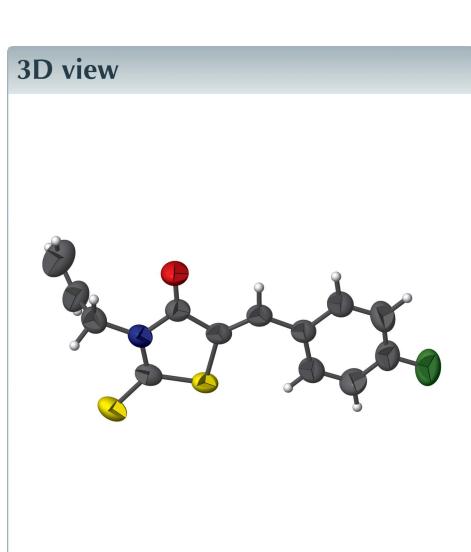
Chemical scheme
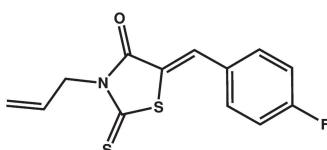
Structure description

5-Arylidene-2-sulfanylidene-1,3-thiazolidinine-4-ones or 5-arylidene rhodanines are considered to be 'privileged scaffolds' in the medicinal chemistry community because they present bioactivity in a large range of derivatives (Mendgen *et al.*, 2012; Khazaei *et al.*, 2014; Coulibaly *et al.*, 2015; Tomašić & Masic *et al.*, 2009). For example, these small molecules have been known to possess a wide range of biological properties such as potent and selective inhibitors of the 'atypical' dual-specificity phosphatase (DSP) family member-JNK-stimulating phosphatase-1 (JSP-1) (Cutshall *et al.*, 2005), as aldose reductase inhibitors in diabetic peripheral neuropathy (Hotta *et al.*, 2006), and as DDX3 inhibitors for HIV replication (Maga *et al.*, 2008). The unusual biological activities displayed by many rhodanine-based molecules have made them attractive synthetic targets.

The molecule of the title compound is build up from a 4-fluorobenzylidene ring linked to a sulfanylidenethiazolidine ring which is attached to an allyl group as shown in Fig. 1. The 4-fluorobenzylidene and the sulfanylidenethiazolidine rings are virtually coplanar with a maximum deviation from the mean plane of 0.067 (3) Å for the C4 atom. The
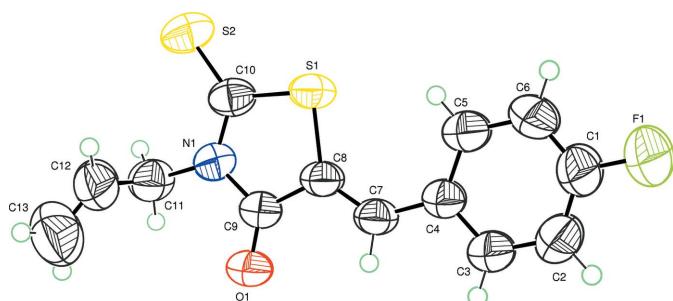
Figure 1
Plot of the molecule of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

sulfanylidenethiazolidine ring makes a dihedral angle of 69.5 (5)° with the plan through the allyl group.

Structure cohesion is ensured by weak C3—H3···O1 and C7—H7···O1 hydrogen bonds, forming dimers parallel to $(1\bar{2}2)$ (see Fig. 2 and Table 1).

Synthesis and crystallization

To a solution of 3-allylrhodanine (1.15 mmol, 0.2 g) in 10 ml of THF, (4-fluorobenzylidene)-4-methyl-5-oxopyrazolidin-2-ium-1-ide (1.38 mmol) was added. The mixture was refluxed for 8 h, monitored by TLC, the reaction completed and a yellow spot (TLC R_f = 0.3, using hexane/ethyl acetate 1:9) was generated cleanly. The solvent was evaporated *in vacuo*. The crude product was purified on silica gel using hexane:ethyl acetate (1:9) as eluent. The title compound was recrystallized from ethanol solution (yield 82%, m.p. 382 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1^i	0.93	2.57	3.442 (3)	157
C3—H3···O1^i	0.93	2.55	3.407 (4)	154

Symmetry code: (i) $-x, -y, -z + 1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{10}FNOS_2$
M_r	279.34
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	296
a, b, c (Å)	4.7764 (10), 11.750 (3), 13.067 (3)
α, β, γ (°)	109.917 (10), 99.766 (11), 101.71 (1)
V (Å^3)	652.0 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.41
Crystal size (mm)	$0.35 \times 0.31 \times 0.22$
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.682, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17965, 2861, 2035
R_{int}	0.037
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.641
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.050, 0.148, 1.07
No. of reflections	2861
No. of parameters	163
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.54, −0.23

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Figure 2
Crystal packing for the title compound showing hydrogen bonds as dashed lines.

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full crystallographic data

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(*Z*)-3-Allyl-5-(4-fluorobenzylidene)-2-sulfanylidenethiazolidin-4-one

Crystal data

$C_{13}H_{10}FNOS_2$	$F(000) = 288$
$M_r = 279.34$	$D_x = 1.423$ Mg m^{-3}
Triclinic, $P\bar{1}$	Melting point: 382 K
$a = 4.7764$ (10) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 11.750$ (3) Å	Cell parameters from 2861 reflections
$c = 13.067$ (3) Å	$\theta = 2.0–27.1°$
$\alpha = 109.917$ (10)°	$\mu = 0.41$ mm^{-1}
$\beta = 99.766$ (11)°	$T = 296$ K
$\gamma = 101.71$ (1)°	Block, colourless
$V = 652.0$ (3) Å^3	$0.35 \times 0.31 \times 0.22$ mm
$Z = 2$	

Data collection

Bruker X8 APEX diffractometer	17965 measured reflections
Radiation source: fine-focus sealed tube	2861 independent reflections
Graphite monochromator	2035 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.037$
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)	$\theta_{max} = 27.1°$, $\theta_{min} = 2.0°$
	$h = -6\rightarrow6$
	$k = -15\rightarrow15$
$T_{min} = 0.682$, $T_{max} = 0.745$	$l = -16\rightarrow16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.050$	H-atom parameters constrained
$wR(F^2) = 0.148$	$w = 1/[\sigma^2(F_o^2) + (0.0706P)^2 + 0.2274P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
2861 reflections	$(\Delta/\sigma)_{max} < 0.001$
163 parameters	$\Delta\rho_{max} = 0.54$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.3197 (8)	0.5284 (3)	0.8728 (3)	0.0779 (9)
C2	0.1461 (9)	0.4097 (3)	0.8479 (3)	0.0892 (10)
H2	0.0260	0.3950	0.8937	0.107*
C3	0.1524 (7)	0.3124 (3)	0.7538 (3)	0.0712 (8)
H3	0.0352	0.2313	0.7363	0.085*
C4	0.3302 (6)	0.3326 (2)	0.6844 (2)	0.0551 (6)
C5	0.4984 (7)	0.4560 (3)	0.7132 (3)	0.0751 (8)
H5	0.6170	0.4729	0.6676	0.090*
C6	0.4931 (8)	0.5531 (3)	0.8072 (3)	0.0825 (9)
H6	0.6075	0.6349	0.8255	0.099*
C7	0.3191 (6)	0.2259 (2)	0.5854 (2)	0.0555 (6)
H7	0.1924	0.1501	0.5771	0.067*
C8	0.4594 (5)	0.2170 (2)	0.5034 (2)	0.0541 (6)
C9	0.4088 (6)	0.0954 (2)	0.4106 (2)	0.0552 (6)
C10	0.7549 (5)	0.2276 (3)	0.3625 (2)	0.0572 (6)
C11	0.5746 (7)	−0.0010 (3)	0.2381 (3)	0.0695 (8)
H11A	0.5181	−0.0773	0.2521	0.083*
H11B	0.7706	0.0073	0.2253	0.083*
C12	0.3616 (9)	−0.0127 (3)	0.1352 (3)	0.0865 (10)
H12	0.3827	0.0586	0.1179	0.104*
C13	0.1561 (11)	−0.1087 (4)	0.0692 (4)	0.1183 (15)
H13A	0.1254	−0.1825	0.0826	0.142*
H13B	0.0348	−0.1060	0.0069	0.142*
N1	0.5847 (4)	0.1081 (2)	0.33787 (18)	0.0548 (5)
O1	0.2415 (5)	−0.00459 (18)	0.39528 (17)	0.0744 (6)
F1	0.3139 (6)	0.6243 (2)	0.9653 (2)	0.1186 (8)
S1	0.71039 (14)	0.33471 (6)	0.48434 (6)	0.0600 (2)
S2	0.97365 (17)	0.26959 (8)	0.29023 (8)	0.0789 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.098 (2)	0.0623 (18)	0.078 (2)	0.0303 (17)	0.0374 (18)	0.0207 (16)
C2	0.113 (3)	0.075 (2)	0.088 (2)	0.0225 (19)	0.058 (2)	0.0293 (18)
C3	0.0802 (19)	0.0576 (16)	0.0754 (19)	0.0085 (14)	0.0317 (16)	0.0262 (14)
C4	0.0559 (14)	0.0505 (13)	0.0600 (15)	0.0112 (11)	0.0151 (11)	0.0248 (12)
C5	0.085 (2)	0.0580 (17)	0.082 (2)	0.0083 (14)	0.0378 (17)	0.0262 (15)
C6	0.100 (2)	0.0478 (16)	0.093 (2)	0.0098 (15)	0.038 (2)	0.0194 (15)
C7	0.0557 (14)	0.0472 (13)	0.0619 (15)	0.0057 (11)	0.0148 (12)	0.0245 (12)
C8	0.0519 (13)	0.0492 (13)	0.0603 (15)	0.0050 (10)	0.0120 (11)	0.0265 (12)
C9	0.0553 (14)	0.0487 (13)	0.0597 (15)	0.0081 (11)	0.0154 (12)	0.0221 (12)
C10	0.0454 (13)	0.0613 (15)	0.0719 (17)	0.0140 (11)	0.0146 (11)	0.0351 (13)
C11	0.0727 (18)	0.0614 (17)	0.080 (2)	0.0218 (14)	0.0346 (16)	0.0255 (15)
C12	0.115 (3)	0.067 (2)	0.068 (2)	0.0179 (19)	0.0249 (19)	0.0192 (16)
C13	0.154 (4)	0.088 (3)	0.092 (3)	0.031 (3)	0.015 (3)	0.020 (2)

N1	0.0520 (11)	0.0556 (12)	0.0603 (13)	0.0130 (9)	0.0187 (10)	0.0261 (10)
O1	0.0830 (13)	0.0523 (11)	0.0776 (13)	−0.0033 (9)	0.0305 (11)	0.0210 (9)
F1	0.168 (2)	0.0730 (13)	0.1117 (17)	0.0337 (13)	0.0750 (16)	0.0134 (11)
S1	0.0534 (4)	0.0515 (4)	0.0731 (5)	0.0033 (3)	0.0190 (3)	0.0271 (3)
S2	0.0669 (5)	0.0865 (6)	0.0954 (6)	0.0129 (4)	0.0382 (4)	0.0469 (5)

Geometric parameters (Å, º)

C1—C6	1.348 (5)	C8—S1	1.754 (2)
C1—F1	1.351 (3)	C9—O1	1.210 (3)
C1—C2	1.367 (5)	C9—N1	1.395 (3)
C2—C3	1.374 (4)	C10—N1	1.370 (3)
C2—H2	0.9300	C10—S2	1.634 (3)
C3—C4	1.388 (4)	C10—S1	1.738 (3)
C3—H3	0.9300	C11—N1	1.470 (3)
C4—C5	1.394 (4)	C11—C12	1.488 (5)
C4—C7	1.446 (4)	C11—H11A	0.9700
C5—C6	1.371 (4)	C11—H11B	0.9700
C5—H5	0.9300	C12—C13	1.257 (5)
C6—H6	0.9300	C12—H12	0.9300
C7—C8	1.343 (4)	C13—H13A	0.9300
C7—H7	0.9300	C13—H13B	0.9300
C8—C9	1.466 (4)		
C6—C1—F1	119.0 (3)	C9—C8—S1	109.58 (18)
C6—C1—C2	122.1 (3)	O1—C9—N1	122.3 (2)
F1—C1—C2	118.9 (3)	O1—C9—C8	127.1 (2)
C1—C2—C3	118.8 (3)	N1—C9—C8	110.6 (2)
C1—C2—H2	120.6	N1—C10—S2	126.4 (2)
C3—C2—H2	120.6	N1—C10—S1	110.99 (19)
C2—C3—C4	121.4 (3)	S2—C10—S1	122.59 (16)
C2—C3—H3	119.3	N1—C11—C12	112.2 (2)
C4—C3—H3	119.3	N1—C11—H11A	109.2
C3—C4—C5	117.1 (3)	C12—C11—H11A	109.2
C3—C4—C7	118.1 (2)	N1—C11—H11B	109.2
C5—C4—C7	124.7 (2)	C12—C11—H11B	109.2
C6—C5—C4	121.5 (3)	H11A—C11—H11B	107.9
C6—C5—H5	119.2	C13—C12—C11	126.9 (4)
C4—C5—H5	119.2	C13—C12—H12	116.6
C1—C6—C5	119.1 (3)	C11—C12—H12	116.6
C1—C6—H6	120.5	C12—C13—H13A	120.0
C5—C6—H6	120.5	C12—C13—H13B	120.0
C8—C7—C4	131.4 (2)	H13A—C13—H13B	120.0
C8—C7—H7	114.3	C10—N1—C9	116.2 (2)
C4—C7—H7	114.3	C10—N1—C11	122.7 (2)
C7—C8—C9	120.7 (2)	C9—N1—C11	121.0 (2)
C7—C8—S1	129.7 (2)	C10—S1—C8	92.50 (12)

Hydrogen-bond geometry (Å, º)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C7—H7⋯O1[i]	0.93	2.57	3.442 (3)	157
C3—H3⋯O1[i]	0.93	2.55	3.407 (4)	154

Symmetry code: (i) −*x*, −*y*, −*z*+1.