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(*Z*)-3-Allyl-5-(3-methoxybenzylidene)-2-sulfanylidene-1,3-thiazolidin-4-one

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Keywords: crystal structure; rhodanine-based molecule; hydrogen bonds.

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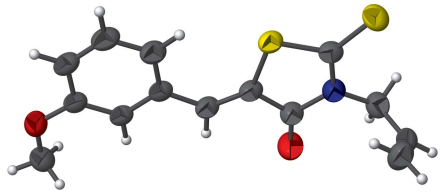
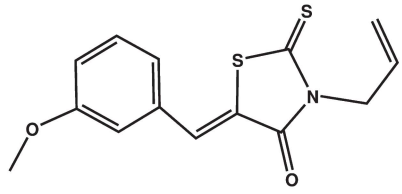
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{14}H_{13}NO_2S_2$, the rhodanine ring and the 3-methoxybenzylidene ring are nearly coplanar, as indicated by the dihedral angle of 1.77 (6)° between their planes. The allyl group is nearly perpendicular to the rhodanine ring, with a dihedral angle of 83.64 (19)°. An intramolecular C—H···S interaction forms an *S*(6) ring motif. In the crystal, molecules are linked by pairs of C—H···O hydrogen bonds into inversion dimers.

3D view



Chemical scheme



Structure description

Compounds containing 2-thioxothiazolidin-4-one (rhodanine) and its derivatives have been reported to exhibit a broad spectrum of biological activities, acting as antidiabetic, anticancer, antitubercular, anti-HIV and antiparasitic agents (Murugan *et al.*, 2009; Chandrappa *et al.*, 2009; Mallikarjuna *et al.*, 2009; Murugesan *et al.*, 2011; Zhang *et al.*, 2009). The unusual biological activity displayed by many rhodanine-based molecules has made them attractive synthetic targets.

The molecule of the title compound is build up from a rhodanine ring (S1/N1/C8–C10) linked to an allyl group (C11–C13) at the nitrogen atom and to a 3-methoxybenzylidene ring (C1–C6) as shown in Fig. 1. In the crystal, molecules are linked by pairs of C—H···O hydrogen bonds (Table 1), forming an inversion dimer as shown in Fig. 2.

Synthesis and crystallization

To a solution of 3-allylrhodanine (1.15 mmol, 0.2 g) in 10 ml of THF, (3-methoxybenzylidene)-4-methyl-5-oxopyrazolidin-2-ium-1-ide (1.38 mmol) was added. The

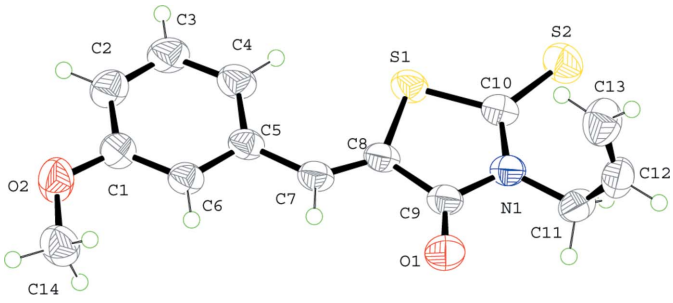


Figure 1
The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

mixture was refluxed for 8 h until the reaction was completed (TLC) 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 (yield 78%, m.p. 364 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The reflection (0 0 1) was affected by the beam-stop and was removed during refinement.

Acknowledgements

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements and the University Sultan Moulay Slimane, Beni-Mellal, Morocco, for financial support.

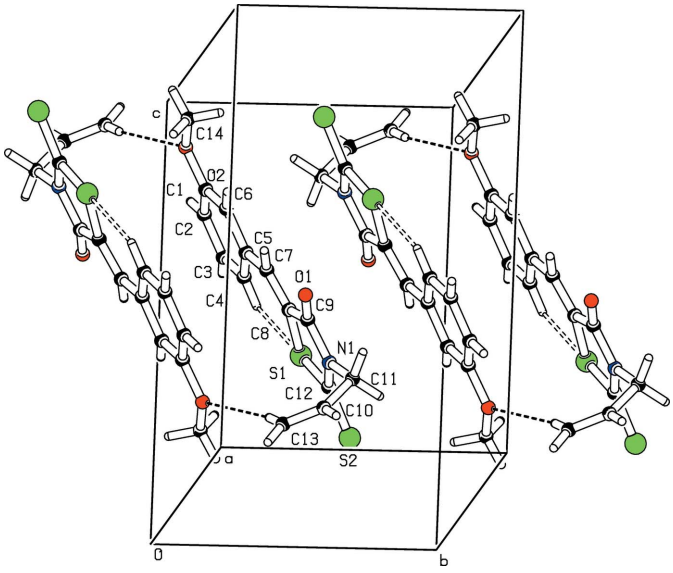


Figure 2
A crystal packing diagram of the title compound, showing the hydrogen bonds as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···S1	0.93	2.55	3.2497 (17)	133
C13—H13A···O2^{i}	0.93	2.57	3.441 (2)	157

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{13}NO_2S_2$
M_r	291.37
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	6.9841 (14), 8.3241 (18), 13.116 (3)
α, β, γ (°)	89.276 (9), 75.614 (9), 72.095 (10)
V (Å^3)	701.2 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.38
Crystal size (mm)	0.31 × 0.27 × 0.21
Data collection	
Diffractometer	Bruker X8 *APEX* diffractometer
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.479, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	28065, 4522, 3618
R_{int}	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.729
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.117, 1.02
No. of reflections	4522
No. of parameters	172
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.26

Computer programs: *APEX2* (Bruker, 2009), *SAINT* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *publCIF* (Westrip, 2010).

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full crystallographic data

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Crystal data

$C_{14}H_{13}NO_2S_2$
M_r = 291.37
Triclinic, $P\overline{1}$
a = 6.9841 (14) Å
b = 8.3241 (18) Å
c = 13.116 (3) Å
α = 89.276 (9)°
β = 75.614 (9)°
γ = 72.095 (10)°
V = 701.2 (3) Å^3
Z = 2
$F(000)$ = 304
D_x = 1.380 Mg m^{-3}
Melting point: 364 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4522 reflections
θ = 2.6–31.2°
μ = 0.38 mm^{-1}
T = 296 K
Block, colourless
0.31 × 0.27 × 0.21 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.479, T_{max} = 0.746
28065 measured reflections
4522 independent reflections
3618 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 31.2°, θ_{min} = 2.6°
h = −10→10
k = −12→12
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.117
S = 1.02
4522 reflections
172 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.1273P]$ 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}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6784 (2)	0.92899 (16)	0.26792 (11)	0.0453 (3)
C2	0.8814 (2)	0.88347 (19)	0.27837 (12)	0.0528 (3)
H2	0.9846	0.9093	0.2274	0.063*
C3	0.9283 (2)	0.8002 (2)	0.36440 (12)	0.0555 (3)
H3	1.0641	0.7700	0.3711	0.067*
C4	0.7776 (2)	0.76011 (19)	0.44141 (11)	0.0512 (3)
H4	0.8122	0.7042	0.4993	0.061*
C5	0.57287 (19)	0.80427 (16)	0.43169 (10)	0.0410 (2)
C6	0.52494 (19)	0.88990 (16)	0.34378 (10)	0.0418 (3)
H6	0.3895	0.9205	0.3365	0.050*
C7	0.40267 (19)	0.76903 (16)	0.50824 (10)	0.0431 (3)
H7	0.2737	0.8106	0.4932	0.052*
C8	0.40122 (19)	0.68638 (16)	0.59698 (10)	0.0408 (2)
C9	0.2049 (2)	0.66565 (17)	0.66206 (10)	0.0450 (3)
C10	0.4403 (2)	0.52824 (16)	0.76057 (10)	0.0434 (3)
C11	0.0714 (2)	0.53236 (19)	0.82335 (12)	0.0520 (3)
H11A	0.1286	0.4223	0.8486	0.062*
H11B	−0.0273	0.5227	0.7851	0.062*
C12	−0.0422 (2)	0.65574 (19)	0.91596 (12)	0.0533 (3)
H12	−0.1612	0.6390	0.9590	0.064*
C13	0.0068 (3)	0.7842 (2)	0.94329 (14)	0.0677 (4)
H13A	0.1244	0.8067	0.9030	0.081*
H13B	−0.0757	0.8538	1.0031	0.081*
C14	0.4469 (3)	1.0541 (2)	0.16012 (13)	0.0616 (4)
H14A	0.4481	1.1117	0.0962	0.092*
H14B	0.4124	0.9525	0.1529	0.092*
H14C	0.3452	1.1265	0.2178	0.092*
N1	0.24062 (17)	0.57615 (14)	0.75017 (9)	0.0435 (2)
O1	0.03559 (17)	0.71622 (17)	0.64424 (9)	0.0660 (3)
O2	0.64706 (17)	1.01213 (16)	0.18002 (9)	0.0643 (3)
S1	0.60384 (5)	0.59150 (4)	0.65517 (3)	0.04671 (11)
S2	0.52699 (7)	0.42768 (6)	0.85588 (3)	0.06213 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0380 (6)	0.0469 (6)	0.0453 (6)	−0.0068 (5)	−0.0083 (5)	−0.0009 (5)
C2	0.0346 (6)	0.0607 (8)	0.0560 (8)	−0.0105 (5)	−0.0042 (5)	−0.0015 (6)
C3	0.0317 (6)	0.0695 (9)	0.0612 (9)	−0.0085 (6)	−0.0135 (6)	−0.0020 (7)
C4	0.0391 (6)	0.0636 (8)	0.0488 (7)	−0.0090 (6)	−0.0162 (5)	0.0004 (6)
C5	0.0352 (5)	0.0458 (6)	0.0395 (6)	−0.0076 (4)	−0.0110 (4)	−0.0062 (5)
C6	0.0332 (5)	0.0470 (6)	0.0422 (6)	−0.0077 (4)	−0.0100 (4)	−0.0035 (5)
C7	0.0354 (5)	0.0534 (7)	0.0409 (6)	−0.0113 (5)	−0.0135 (5)	−0.0028 (5)
C8	0.0349 (5)	0.0488 (6)	0.0392 (6)	−0.0105 (5)	−0.0132 (4)	−0.0048 (5)
C9	0.0406 (6)	0.0549 (7)	0.0441 (6)	−0.0171 (5)	−0.0166 (5)	0.0017 (5)

C10	0.0416 (6)	0.0445 (6)	0.0449 (6)	−0.0107 (5)	−0.0161 (5)	−0.0023 (5)
C11	0.0508 (7)	0.0571 (8)	0.0593 (8)	−0.0288 (6)	−0.0195 (6)	0.0116 (6)
C12	0.0428 (7)	0.0607 (8)	0.0539 (8)	−0.0159 (6)	−0.0095 (6)	0.0197 (6)
C13	0.0682 (10)	0.0646 (9)	0.0590 (9)	−0.0200 (8)	0.0030 (8)	−0.0032 (7)
C14	0.0578 (9)	0.0692 (9)	0.0569 (9)	−0.0132 (7)	−0.0219 (7)	0.0129 (7)
N1	0.0412 (5)	0.0495 (6)	0.0440 (5)	−0.0169 (4)	−0.0150 (4)	0.0016 (4)
O1	0.0423 (5)	0.0990 (9)	0.0671 (7)	−0.0278 (5)	−0.0264 (5)	0.0233 (6)
O2	0.0462 (6)	0.0820 (8)	0.0585 (6)	−0.0147 (5)	−0.0103 (5)	0.0227 (6)
S1	0.03460 (15)	0.0597 (2)	0.04389 (18)	−0.00911 (13)	−0.01372 (12)	0.00062 (13)
S2	0.0570 (2)	0.0726 (3)	0.0594 (2)	−0.01587 (19)	−0.02600 (18)	0.01825 (18)

Geometric parameters (Å, º)

C1—O2	1.3668 (17)	C9—N1	1.4001 (17)
C1—C6	1.3860 (18)	C10—N1	1.3685 (17)
C1—C2	1.3920 (19)	C10—S2	1.6335 (14)
C2—C3	1.373 (2)	C10—S1	1.7494 (14)
C2—H2	0.9300	C11—N1	1.4639 (17)
C3—C4	1.385 (2)	C11—C12	1.487 (2)
C3—H3	0.9300	C11—H11A	0.9700
C4—C5	1.4008 (18)	C11—H11B	0.9700
C4—H4	0.9300	C12—C13	1.298 (2)
C5—C6	1.4050 (18)	C12—H12	0.9300
C5—C7	1.4564 (18)	C13—H13A	0.9300
C6—H6	0.9300	C13—H13B	0.9300
C7—C8	1.3441 (19)	C14—O2	1.422 (2)
C7—H7	0.9300	C14—H14A	0.9600
C8—C9	1.4827 (18)	C14—H14B	0.9600
C8—S1	1.7445 (13)	C14—H14C	0.9600
C9—O1	1.2079 (16)		
O2—C1—C6	124.67 (12)	N1—C10—S2	127.52 (11)
O2—C1—C2	115.21 (13)	N1—C10—S1	110.74 (10)
C6—C1—C2	120.12 (13)	S2—C10—S1	121.74 (8)
C3—C2—C1	119.57 (13)	N1—C11—C12	114.59 (11)
C3—C2—H2	120.2	N1—C11—H11A	108.6
C1—C2—H2	120.2	C12—C11—H11A	108.6
C2—C3—C4	121.39 (13)	N1—C11—H11B	108.6
C2—C3—H3	119.3	C12—C11—H11B	108.6
C4—C3—H3	119.3	H11A—C11—H11B	107.6
C3—C4—C5	119.69 (14)	C13—C12—C11	127.06 (14)
C3—C4—H4	120.2	C13—C12—H12	116.5
C5—C4—H4	120.2	C11—C12—H12	116.5
C4—C5—C6	118.87 (12)	C12—C13—H13A	120.0
C4—C5—C7	124.13 (12)	C12—C13—H13B	120.0
C6—C5—C7	117.00 (11)	H13A—C13—H13B	120.0
C1—C6—C5	120.35 (12)	O2—C14—H14A	109.5
C1—C6—H6	119.8	O2—C14—H14B	109.5

C5—C6—H6	119.8	H14A—C14—H14B	109.5
C8—C7—C5	130.67 (12)	O2—C14—H14C	109.5
C8—C7—H7	114.7	H14A—C14—H14C	109.5
C5—C7—H7	114.7	H14B—C14—H14C	109.5
C7—C8—C9	120.39 (11)	C10—N1—C9	116.48 (11)
C7—C8—S1	130.08 (10)	C10—N1—C11	123.16 (12)
C9—C8—S1	109.53 (9)	C9—N1—C11	120.32 (11)
O1—C9—N1	123.01 (13)	C1—O2—C14	118.75 (12)
O1—C9—C8	126.62 (13)	C8—S1—C10	92.87 (6)
N1—C9—C8	110.37 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···S1	0.93	2.55	3.2497 (17)	133
C13—H13*A*···O2^{i}	0.93	2.57	3.441 (2)	157

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.