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Crystal structure of (*Z*)-3-allyl-5-(4-chlorobenzylidene)-2-sulfanylidene-1,3-thiazolidin-4-one

Rahhal El Ajlaoui,[a]* El Mostapha Rakib,[a] Souad Mojahidi,[a] Mohamed Saadi[b] and Lahcen El Ammari[b]

[a]Laboratoire de Chimie Organique et Analytique, Université Sultan Moulay Slimane, Faculté des Sciences et Techniques, Béni-Mellal, BP 523, Morocco, and [b]Laboratoire de Chimie du Solide Appliquée, Faculté des Sciences, Université Mohammed V de Rabat, Avenue Ibn Battouta, BP 1014, Rabat, Morocco. *Correspondence e-mail: r_elajlaoui@yahoo.fr

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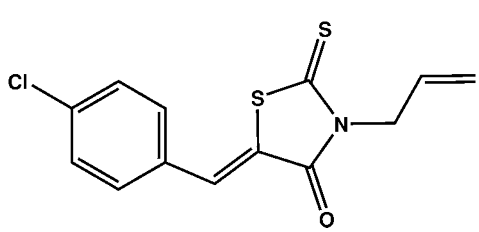
In the title compound, $C_{13}H_{10}ClNOS_2$, the dihedral angle between the rhodanine (r.m.s. deviation = 0.008 Å) and 4-chlorobenzylidene rings is 1.79 (11)°. The allyl group attached to the N atom, which lies almost perpendicular to the rhodanine ring, is disordered over two orientations in a 0.519 (13):0.481 (13) ratio. A short intramolecular C—H⋯S interaction closes an *S*(6) ring. In the crystal, molecules are linked by π–π stacking interactions [centroid–centroid separation = 3.600 (15) Å], generating inversion dimers.

Keywords: crystal structure; rhodanine-based molecules; pharmacological activity; biological activity; 1,3-thiazolidin-4-one.

CCDC reference: 1439050

1. Related literature

For a related structure and background to the pharmacological and biological activities of rhodanine-based molecules, see: El Ajlaoui *et al.* (2015).



2. Experimental

2.1. Crystal data

$C_{13}H_{10}ClNOS_2$
M_r = 295.79
Triclinic, $P\overline{1}$
a = 7.6197 (8) Å
b = 7.9849 (7) Å
c = 13.0624 (14) Å
α = 77.600 (5)°
β = 77.996 (5)°
γ = 61.954 (4)°
V = 679.76 (12) $Å^3$
Z = 2
Mo *K*α radiation
μ = 0.57 mm^{-1}
T = 296 K
0.37 × 0.25 × 0.21 mm

2.2. Data collection

Bruker X8 APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.656, T_{max} = 0.746
24189 measured reflections
3249 independent reflections
2199 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.144
S = 1.04
3249 reflections
182 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.35 e $Å^{-3}$

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C3—H3⋯S1	0.93	2.55	3.254 (3)	133

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7551).

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supporting information

Acta Cryst. (2015). E**71**, o1012 [https://doi.org/10.1107/S2056989015022689]

Crystal structure of (*Z*)-3-allyl-5-(4-chlorobenzylidene)-2-sulfanylidene-1,3-thiazolidin-4-one

Rahhal El Ajlaoui, El Mostapha Rakib, Souad Mojahidi, Mohamed Saadi and Lahcen El Ammari

S1. Comment

As part of our ongoing studies of rhodanine derivatives, we now describe the title compound.

The molecule of the title compound is build up from a rhodanine ring (S1–N1–C8–C9–C10) linked to an disordered allyl group (48%/52%) (C11–C12AC12B–C13AC13B) and at the nitrogen atom and to a 4-chlorobenzylidene ring system (C1 to C6) as shown in Fig.1. The mean plane through the rhodanine ring is almost perpendicular to the allyl group and makes a dihedral angle of 1.79 (11)° with the 4-chlorobenzylidene ring system. Nearly the same structure is observed by El Ajlaoui *et al.* 2015 in (Z)-3-Allyl-5-(4-methyl-benzylidene)-2- thioxothiazolidin-4-one.

The cohesion of the crystal structure is ensured by π—π interaction between molecules forming inversion dimers as shown in Fig.2.

S2. Experimental

To a solution of 3-allylrhodanine (1.15 mmol, 0.2 g) in 10 mL of THF, (4-chlorobenzylidene)-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 Rf = 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: 72%, m.p.: 371 K).

S3. Refinement

H atoms were located in a difference map and treated as riding with C–H = 0.97 Å and C–H = 0.93 Å for methylene and aromatic, respectively. All hydrogen with $U_{iso}(H) = 1.2 U_{eq}$ for methylene and aromatic. The reflection (0 0 1) affected by the beam-stop is removed during refinement.

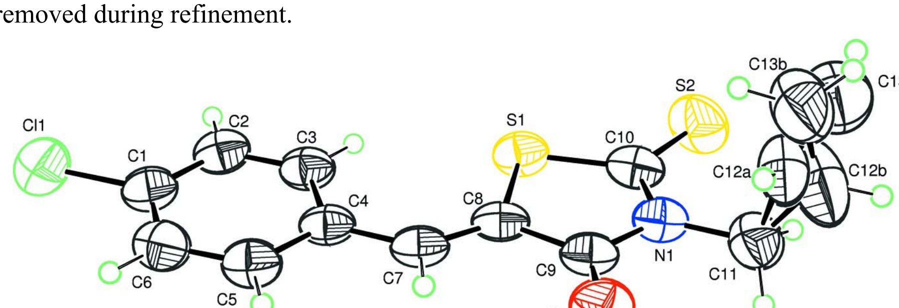

Figure 1

Plot of the molecule of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are represented as small circles.

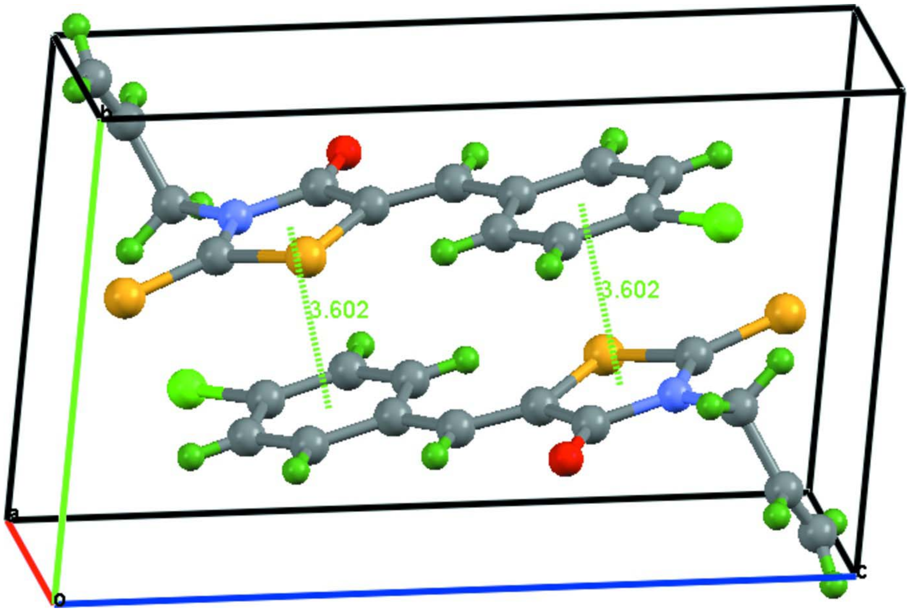

Figure 2

Crystal packing for the title compound showing hydrogen bonds as dashed lines between inversion-related molecules.

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

Crystal data

$C_{13}H_{10}ClNOS_2$
M_r = 295.79
Triclinic, $P\overline{1}$
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b = 7.9849 (7) Å
c = 13.0624 (14) Å
α = 77.600 (5)°
β = 77.996 (5)°
γ = 61.954 (4)°
V = 679.76 (12) Å^3
Z = 2
$F(000)$ = 304
D_x = 1.445 Mg m^{-3}
Melting point: 371 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3249 reflections
θ = 2.9–27.9°
μ = 0.57 mm^{-1}
T = 296 K
Block, colourless
0.37 × 0.25 × 0.21 mm

Data collection

Bruker X8 APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.656, T_{max} = 0.746
24189 measured reflections
3249 independent reflections
2199 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.9°, θ_{min} = 2.9°
h = −10→9
k = −10→10
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.144
S = 1.04
3249 reflections
182 parameters
3 restraints
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 0.2968P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ 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}	Occ. (<1)
C1	−0.1200 (4)	0.7895 (3)	0.6827 (2)	0.0698 (7)	
C2	−0.0835 (4)	0.7296 (4)	0.5860 (2)	0.0709 (7)	
H2	−0.1709	0.6939	0.5668	0.085*	
C3	0.0823 (4)	0.7228 (3)	0.5181 (2)	0.0652 (6)	
H3	0.1060	0.6822	0.4528	0.078*	
C4	0.2174 (3)	0.7752 (3)	0.54439 (19)	0.0574 (6)	
C5	0.1746 (4)	0.8355 (3)	0.6429 (2)	0.0678 (7)	
H5	0.2612	0.8714	0.6627	0.081*	
C6	0.0086 (5)	0.8438 (4)	0.7117 (2)	0.0762 (7)	
H6	−0.0170	0.8853	0.7769	0.091*	
C7	0.3946 (4)	0.7721 (3)	0.4765 (2)	0.0592 (6)	
H4	0.4665	0.8149	0.5045	0.071*	
C8	0.4733 (4)	0.7186 (3)	0.38062 (19)	0.0584 (6)	
C9	0.6604 (4)	0.7264 (3)	0.3269 (2)	0.0634 (6)	
C10	0.5892 (4)	0.6044 (4)	0.2013 (2)	0.0712 (7)	
C11	0.8918 (5)	0.6605 (5)	0.1606 (3)	0.0912 (9)	
H11A	0.9341	0.5757	0.1079	0.109*	
H11B	1.0013	0.6194	0.2015	0.109*	
C12A	0.8259 (17)	0.8701 (16)	0.1084 (6)	0.100 (3)	0.519 (13)
H12A	0.8190	0.9597	0.1463	0.120*	0.519 (13)
C13A	0.779 (2)	0.925 (2)	0.0099 (6)	0.131 (4)	0.519 (13)
H13A	0.7852	0.8369	−0.0287	0.157*	0.519 (13)
H13B	0.7393	1.0524	−0.0202	0.157*	0.519 (13)
C12B	0.8935 (18)	0.8039 (12)	0.0686 (7)	0.144 (6)	0.481 (13)
H12B	1.0008	0.7686	0.0151	0.172*	0.481 (13)
C13B	0.747 (2)	0.9822 (14)	0.0591 (12)	0.140 (6)	0.481 (13)
H13C	0.6384	1.0200	0.1117	0.168*	0.481 (13)
H13D	0.7530	1.0688	−0.0002	0.168*	0.481 (13)
N1	0.7137 (3)	0.6620 (3)	0.22888 (17)	0.0667 (5)	
O1	0.7595 (3)	0.7794 (3)	0.36081 (16)	0.0818 (6)	
S1	0.38675 (10)	0.63202 (10)	0.29984 (6)	0.0703 (2)	
S2	0.61487 (17)	0.51989 (15)	0.09357 (7)	0.1047 (3)	
Cl1	−0.33030 (13)	0.79962 (14)	0.76777 (7)	0.1017 (3)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0656 (15)	0.0542 (13)	0.0814 (17)	−0.0180 (11)	−0.0213 (13)	−0.0017 (12)
C2	0.0635 (15)	0.0670 (15)	0.0856 (18)	−0.0260 (12)	−0.0248 (14)	−0.0091 (13)
C3	0.0675 (15)	0.0583 (13)	0.0731 (15)	−0.0225 (12)	−0.0262 (12)	−0.0119 (11)
C4	0.0618 (13)	0.0388 (10)	0.0715 (14)	−0.0163 (9)	−0.0270 (11)	−0.0041 (10)
C5	0.0759 (17)	0.0565 (13)	0.0795 (17)	−0.0280 (12)	−0.0257 (14)	−0.0138 (12)
C6	0.0841 (19)	0.0632 (15)	0.0758 (17)	−0.0217 (14)	−0.0203 (15)	−0.0153 (13)
C7	0.0652 (14)	0.0444 (11)	0.0755 (15)	−0.0228 (10)	−0.0310 (12)	−0.0052 (10)
C8	0.0647 (14)	0.0441 (11)	0.0727 (15)	−0.0224 (10)	−0.0320 (12)	−0.0019 (10)
C9	0.0704 (15)	0.0502 (12)	0.0743 (16)	−0.0265 (11)	−0.0300 (12)	0.0017 (11)
C10	0.0826 (17)	0.0618 (14)	0.0712 (16)	−0.0280 (13)	−0.0312 (14)	−0.0024 (12)
C11	0.094 (2)	0.101 (2)	0.082 (2)	−0.0513 (19)	−0.0114 (17)	−0.0002 (17)
C12A	0.118 (7)	0.113 (8)	0.074 (5)	−0.068 (7)	0.019 (5)	−0.014 (5)
C13A	0.134 (10)	0.109 (9)	0.136 (9)	−0.044 (8)	−0.018 (8)	−0.011 (7)
C12B	0.221 (15)	0.113 (8)	0.077 (7)	−0.075 (9)	0.033 (8)	−0.022 (6)
C13B	0.147 (9)	0.103 (8)	0.092 (9)	−0.008 (7)	0.022 (7)	−0.009 (6)
N1	0.0715 (13)	0.0601 (11)	0.0714 (13)	−0.0293 (10)	−0.0238 (11)	−0.0003 (10)
O1	0.0901 (13)	0.0898 (13)	0.0922 (13)	−0.0563 (11)	−0.0284 (11)	−0.0093 (10)
S1	0.0730 (4)	0.0733 (4)	0.0787 (4)	−0.0340 (3)	−0.0272 (3)	−0.0173 (3)
S2	0.1285 (8)	0.1284 (8)	0.0783 (5)	−0.0645 (6)	−0.0206 (5)	−0.0303 (5)
Cl1	0.0793 (5)	0.1100 (7)	0.1008 (6)	−0.0346 (5)	−0.0040 (4)	−0.0102 (5)

Geometric parameters (Å, º)

C1—C2	1.374 (4)	C9—N1	1.392 (3)
C1—C6	1.384 (4)	C10—N1	1.366 (3)
C1—Cl1	1.728 (3)	C10—S2	1.626 (3)
C2—C3	1.370 (4)	C10—S1	1.749 (3)
C2—H2	0.9300	C11—N1	1.457 (4)
C3—C4	1.402 (3)	C11—C12B	1.4743 (10)
C3—H3	0.9300	C11—C12A	1.543 (11)
C4—C5	1.392 (3)	C11—H11A	0.9700
C4—C7	1.445 (4)	C11—H11B	0.9700
C5—C6	1.373 (4)	C12A—C13A	1.3334 (10)
C5—H5	0.9300	C12A—H12A	0.9300
C6—H6	0.9300	C13A—H13A	0.9300
C7—C8	1.338 (3)	C13A—H13B	0.9300
C7—H4	0.9300	C12B—C13B	1.3333 (10)
C8—C9	1.475 (4)	C12B—H12B	0.9300
C8—S1	1.749 (2)	C13B—H13C	0.9300
C9—O1	1.211 (3)	C13B—H13D	0.9300
C2—C1—C6	120.6 (3)	N1—C10—S2	127.3 (2)
C2—C1—Cl1	119.4 (2)	N1—C10—S1	110.9 (2)
C6—C1—Cl1	119.9 (2)	S2—C10—S1	121.89 (17)
C3—C2—C1	119.5 (2)	N1—C11—C12B	125.5 (5)

C3—C2—H2	120.2	N1—C11—C12A	104.4 (5)
C1—C2—H2	120.2	N1—C11—H11A	110.9
C2—C3—C4	121.8 (2)	C12A—C11—H11A	110.9
C2—C3—H3	119.1	N1—C11—H11B	110.9
C4—C3—H3	119.1	C12A—C11—H11B	110.9
C5—C4—C3	116.8 (2)	H11A—C11—H11B	108.9
C5—C4—C7	118.5 (2)	C13A—C12A—C11	121.3 (11)
C3—C4—C7	124.7 (2)	C13A—C12A—H12A	119.4
C6—C5—C4	122.1 (2)	C11—C12A—H12A	119.4
C6—C5—H5	119.0	C12A—C13A—H13A	120.0
C4—C5—H5	119.0	C12A—C13A—H13B	120.0
C5—C6—C1	119.1 (3)	H13A—C13A—H13B	120.0
C5—C6—H6	120.4	C13B—C12B—C11	122.4 (11)
C1—C6—H6	120.4	C13B—C12B—H12B	118.8
C8—C7—C4	131.4 (2)	C11—C12B—H12B	118.8
C8—C7—H4	114.3	C12B—C13B—H13C	120.0
C4—C7—H4	114.3	C12B—C13B—H13D	120.0
C7—C8—C9	121.4 (2)	H13C—C13B—H13D	120.0
C7—C8—S1	129.3 (2)	C10—N1—C9	116.3 (2)
C9—C8—S1	109.29 (18)	C10—N1—C11	123.1 (3)
O1—C9—N1	122.6 (3)	C9—N1—C11	120.6 (2)
O1—C9—C8	126.4 (3)	C8—S1—C10	92.62 (12)
N1—C9—C8	110.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···S1	0.93	2.55	3.254 (3)	133