Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(5*S*)-5-Methyl-3-phenyl-2-sulfanylidene-1,3-thiazolidin-4-one

Jun-Rong Jiang,* Feng Xu, Zhong-Lu Ke and Li Li

Department of Biological & Chemical Engineering, Taizhou Vocational & Technical College, Taizhou 318000, People's Republic of China
Correspondence e-mail: jiangjr@tzvtc.com

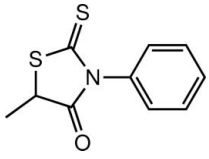
Received 15 November 2011; accepted 29 November 2011

Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.064; data-to-parameter ratio = 21.7.

In the title molecule, $C_{10}H_9NOS_2$, the 2-sulfanylidenethiazolidin-4-one mean plane and phenyl ring form a dihedral angle of 81.7 (1)°. In the crystal, C—H$\cdots\pi$ interactions link molecules into helical chains in [010].

Related literature

For related structures, see: Gattow *et al.* (1983); Rang *et al.* (1997). For applications of 2-sulfanylidenethiazolidin-4-one derivatives, see: Zidar *et al.* (2010); Powers *et al.* (2006).



Experimental

Crystal data

$C_{10}H_9NOS_2$
M_r = 223.30
Orthorhombic, $P2_12_12_1$
a = 6.8527 (4) Å
b = 8.6643 (5) Å
c = 17.5572 (15) Å
V = 1042.44 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.48 mm^{-1}
T = 153 K
0.30 × 0.20 × 0.18 mm

Data collection

Rigaku AFC10/Saturn724+ diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2008)
T_{min} = 0.872, T_{max} = 0.919
9028 measured reflections
2777 independent reflections
2561 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.064
S = 1.00
2777 reflections
128 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Absolute structure: Flack (1983), 1155 Friedel pairs
Flack parameter: −0.01 (6)

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

Cg is the centroid of the C7–C12 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C5—H5$\cdots Cg^{i}$	1.00	2.47	3.4321 (16)	162

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

Data collection: *CrystalClear* (Rigaku/MSC, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear* ; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

We are very grateful to the Foundation of Taizhou Vocational and Technical College (grant No. 2010ZD09) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5203).

References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Gattow, G., Kiel, G. & Rach, W. (1983). *Z. Anorg. Allg. Chem.* **506**, 145-152.
Powers, J. P., Piper, D. E., Li, Y., Mayorga, V., Anzola, J., Chen, J. M., Jaen, J. C., Lee, G., Liu, J., Peterson, M. G., Tonn, G. R., Ye, Q., Walker, N. P. & Wang, Z. (2006). *J. Med. Chem.* **49**,1034–1046.
Rang, K., Liao, F. L., Sandstorm, J. & Wang, S. L. (1997). *Chirality*, **9**, 568-577.
Rigaku/MSC (2008). *CrystalClear*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Zidar, N., Tomašić, T., Šink, R., Rupnik, V., Kovač, A., Turk, S., Patin, D., Blanot, D., Martel, C. C., Dessen, A., Müller-Premru, M., Zega, A., Gobec, S., Mašić, L. P. & Kikelj, D. (2010). *J. Med. Chem.* **53**, 6584-6594.

supporting information

Acta Cryst. (2012). E**68**, o34 [doi:10.1107/S1600536811051312]

(5*S*)-5-Methyl-3-phenyl-2-sulfanylidene-1,3-thiazolidin-4-one

Jun-Rong Jiang, Feng Xu, Zhong-Lu Ke and Li Li

S1. Comment

2-sulfanylidenethiazolidin-4-one derivatives are known as compounds with potential antifungal activities (Zidar *et al.*, 2010) and potential drugs-inhibitors of the HCV-RNA polymerase (Powers *et al.*, 2006). Herewith we present the title compound (I), which is a new 2-sulfanylidenethiazolidin-4-one derivative.

In (I) (Fig. 1), all bond lengths and angles are normal and correspond to those observed in the related compounds 3-(S)-(1-phenylethyl)-5-methyl-2-sulfanylidenethiazolidin-4-one (Rang *et al.*, 1997) and 5-methyl-2-sulfanylidenethiazolidin-4-one (Gattow *et al.*, 1983). The 2-sulfanylidenethiazolidin-4-one and phenyl rings form a dihedral angle of 81.7 (1)°. In the crystal structure, intermolecular C—H···π interactions (Table 1) link molecules into helical chains in [010].

S2. Experimental

To 54 ml of concentrated ammonia in an ice-salt bath was added 13.95 g(0.15 mol) of benzylamine. carbon bisulfide 19.5 ml(24.6 g,0.323 mol) was added dropwise over a period 2 h and stirring continued for 4 h.The dithiocarbamate precipitated was allowed to stand overnight. It was filtered(warning:filtered to be immediately used), washed with cold ether and dried by suction. The sodium 2-bromopropionate solution was prepared by 15.3 g(0.1 mol) of 2-bromopropionic acid in 9 ml of water and 3.5 g(0.0875 mol)of sodium hydroxide in 6 ml of water,and adding saturated $NaHCO_3$ solution until the solution was basic.The sodium 2-bromopropionate solution was stirred, cooled to 273 K and the dithiocarbamate added by batch about 10 min.After the mixture was stirred for 1 h at the same condition,it was allowed to warm up to r.t. and stand 30 min.Then a hot solution of concentrated HCl plus water(40 ml+27 ml)was added to it.The mixture was boiled for 10 min and cooled to r.t.The precipitate was filtered, washed with cold water and little cold ethanol.The crude product was recrystallized from ethanol to yield 13.6 g(61%) yellow needle-like compounds.

S3. Refinement

H atoms were placed in calculated positions [C—H = 0.95-1.00 Å] and refined in riding mode, with U_{iso}(H) = 1.2-1.5 U_{eq}(C).

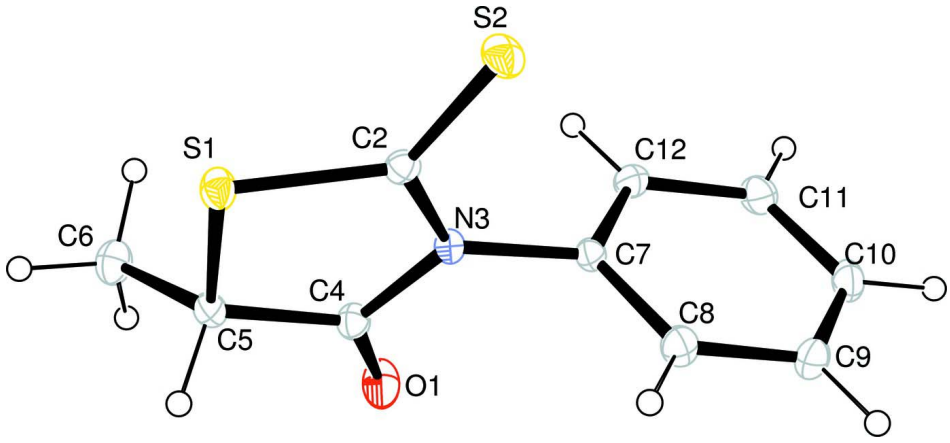


Figure 1

The molecular structure of (I), shown with 30% probability displacement ellipsoids.

(5*S*)-5-Methyl-3-phenyl-2-sulfanylidene-1,3-thiazolidin-4-one

Crystal data

$C_{10}H_9NOS_2$
$M_r = 223.30$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.8527$ (4) Å
$b = 8.6643$ (5) Å
$c = 17.5572$ (15) Å
$V = 1042.44$ (12) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.423$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3919 reflections
$\theta = 2.3$–$29.1°$
$\mu = 0.48$ mm^{-1}
$T = 153$ K
Block, colorless
0.30 × 0.20 × 0.18 mm

Data collection

Rigaku AFC10/Saturn724+ diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 28.5714 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2008)
$T_{min} = 0.872$, $T_{max} = 0.919$
9028 measured reflections
2777 independent reflections
2561 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 29.1°$, $\theta_{min} = 2.6°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 10$
$l = -23 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.064$
$S = 1.00$
2777 reflections
128 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2 + 0.086P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.014$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Absolute structure: Flack (1983), 1155 Friedel pairs
Absolute structure parameter: −0.01 (6)

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.11471 (5)	0.55991 (4)	0.42516 (2)	0.02375 (9)
S2	0.13997 (6)	0.70725 (4)	0.57733 (2)	0.02665 (10)
O1	0.53389 (18)	0.27885 (14)	0.46834 (6)	0.0334 (3)
N3	0.36082 (17)	0.47158 (13)	0.52694 (6)	0.0177 (2)
C2	0.2145 (2)	0.57762 (16)	0.51556 (8)	0.0186 (3)
C4	0.4056 (2)	0.37374 (17)	0.46629 (8)	0.0212 (3)
C5	0.2751 (2)	0.40173 (16)	0.39840 (8)	0.0204 (3)
H5	0.1937	0.3075	0.3899	0.025*
C6	0.3913 (2)	0.4338 (2)	0.32607 (8)	0.0285 (3)
H6A	0.4889	0.3525	0.3188	0.034*
H6B	0.4569	0.5339	0.3306	0.034*
H6C	0.3027	0.4357	0.2823	0.034*
C7	0.4603 (2)	0.45370 (16)	0.59871 (7)	0.0184 (3)
C8	0.3715 (2)	0.36714 (18)	0.65529 (8)	0.0246 (3)
H8	0.2451	0.3249	0.6476	0.030*
C9	0.4698 (3)	0.34305 (19)	0.72335 (9)	0.0285 (4)
H9	0.4112	0.2829	0.7624	0.034*
C10	0.6522 (2)	0.40624 (18)	0.73445 (8)	0.0280 (3)
H10	0.7178	0.3909	0.7815	0.034*
C11	0.7409 (2)	0.4923 (2)	0.67723 (9)	0.0274 (3)
H11	0.8671	0.5348	0.6852	0.033*
C12	0.6448 (2)	0.51620 (16)	0.60833 (8)	0.0226 (3)
H12	0.7045	0.5742	0.5687	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02356 (17)	0.02662 (18)	0.02108 (17)	0.00623 (15)	−0.00460 (16)	−0.00175 (15)
S2	0.02865 (19)	0.02565 (18)	0.02565 (19)	0.00662 (16)	0.00106 (18)	−0.00678 (16)
O1	0.0377 (7)	0.0365 (7)	0.0259 (6)	0.0192 (6)	−0.0044 (5)	−0.0058 (5)
N3	0.0179 (5)	0.0197 (5)	0.0155 (5)	0.0002 (5)	−0.0008 (5)	−0.0004 (4)
C2	0.0180 (6)	0.0183 (6)	0.0194 (6)	−0.0016 (5)	0.0012 (5)	0.0008 (5)
C4	0.0225 (8)	0.0226 (7)	0.0186 (7)	0.0013 (6)	0.0004 (6)	−0.0003 (6)
C5	0.0230 (7)	0.0198 (7)	0.0186 (6)	0.0002 (6)	−0.0007 (6)	−0.0027 (6)
C6	0.0301 (8)	0.0347 (8)	0.0208 (7)	0.0031 (8)	0.0021 (6)	0.0006 (7)
C7	0.0216 (6)	0.0182 (7)	0.0154 (6)	0.0014 (5)	−0.0005 (5)	−0.0012 (5)

C8	0.0227 (7)	0.0286 (7)	0.0227 (7)	−0.0028 (7)	0.0021 (6)	−0.0003 (6)
C9	0.0374 (9)	0.0291 (8)	0.0189 (7)	−0.0009 (7)	0.0043 (7)	0.0031 (6)
C10	0.0355 (9)	0.0292 (8)	0.0193 (7)	0.0057 (7)	−0.0065 (7)	−0.0017 (6)
C11	0.0254 (7)	0.0281 (8)	0.0286 (8)	−0.0028 (6)	−0.0078 (6)	−0.0007 (6)
C12	0.0243 (7)	0.0205 (6)	0.0231 (7)	−0.0025 (6)	−0.0008 (6)	0.0023 (6)

Geometric parameters (Å, º)

S1—C2	1.7350 (14)	C6—H6C	0.9800
S1—C5	1.8184 (15)	C7—C12	1.3856 (19)
S2—C2	1.6427 (14)	C7—C8	1.386 (2)
O1—C4	1.2043 (17)	C8—C9	1.387 (2)
N3—C2	1.3746 (17)	C8—H8	0.9500
N3—C4	1.3951 (18)	C9—C10	1.379 (2)
N3—C7	1.4412 (17)	C9—H9	0.9500
C4—C5	1.510 (2)	C10—C11	1.391 (2)
C5—C6	1.5245 (19)	C10—H10	0.9500
C5—H5	1.0000	C11—C12	1.393 (2)
C6—H6A	0.9800	C11—H11	0.9500
C6—H6B	0.9800	C12—H12	0.9500
C2—S1—C5	93.72 (7)	H6A—C6—H6C	109.5
C2—N3—C4	117.10 (11)	H6B—C6—H6C	109.5
C2—N3—C7	122.96 (11)	C12—C7—C8	121.67 (13)
C4—N3—C7	119.87 (12)	C12—C7—N3	119.76 (12)
N3—C2—S2	126.00 (10)	C8—C7—N3	118.49 (13)
N3—C2—S1	111.18 (10)	C7—C8—C9	119.05 (15)
S2—C2—S1	122.82 (9)	C7—C8—H8	120.5
O1—C4—N3	123.54 (13)	C9—C8—H8	120.5
O1—C4—C5	124.46 (13)	C10—C9—C8	120.14 (15)
N3—C4—C5	112.01 (12)	C10—C9—H9	119.9
C4—C5—C6	112.18 (12)	C8—C9—H9	119.9
C4—C5—S1	105.97 (10)	C9—C10—C11	120.47 (14)
C6—C5—S1	113.16 (10)	C9—C10—H10	119.8
C4—C5—H5	108.5	C11—C10—H10	119.8
C6—C5—H5	108.5	C10—C11—C12	120.01 (15)
S1—C5—H5	108.5	C10—C11—H11	120.0
C5—C6—H6A	109.5	C12—C11—H11	120.0
C5—C6—H6B	109.5	C7—C12—C11	118.64 (14)
H6A—C6—H6B	109.5	C7—C12—H12	120.7
C5—C6—H6C	109.5	C11—C12—H12	120.7
C4—N3—C2—S2	−178.34 (11)	C2—S1—C5—C4	−0.78 (10)
C7—N3—C2—S2	4.66 (19)	C2—S1—C5—C6	−124.12 (11)
C4—N3—C2—S1	1.17 (15)	C2—N3—C7—C12	−102.04 (16)
C7—N3—C2—S1	−175.83 (10)	C4—N3—C7—C12	81.04 (17)
C5—S1—C2—N3	−0.14 (11)	C2—N3—C7—C8	81.10 (18)
C5—S1—C2—S2	179.39 (9)	C4—N3—C7—C8	−95.83 (16)

C2—N3—C4—O1	177.98 (14)	C12—C7—C8—C9	0.2 (2)
C7—N3—C4—O1	−4.9 (2)	N3—C7—C8—C9	177.02 (13)
C2—N3—C4—C5	−1.81 (17)	C7—C8—C9—C10	0.8 (2)
C7—N3—C4—C5	175.29 (12)	C8—C9—C10—C11	−1.1 (2)
O1—C4—C5—C6	−54.3 (2)	C9—C10—C11—C12	0.5 (2)
N3—C4—C5—C6	125.48 (13)	C8—C7—C12—C11	−0.8 (2)
O1—C4—C5—S1	−178.26 (13)	N3—C7—C12—C11	−177.60 (13)
N3—C4—C5—S1	1.53 (14)	C10—C11—C12—C7	0.5 (2)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C7–C12 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···*Cg*^{i}	1.00	2.47	3.4321 (16)	162

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