

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

# N-[(Z)-3-(4-Chlorobenzoyl)-1,3-thiazolidin-2-ylidene]cyanamide

 Jian-Gang Wang,<sup>a</sup> Li-Hua Huang<sup>b</sup> and Fang-Fang Jian<sup>c\*</sup>

<sup>a</sup>Microscale Science Institute, Biology Department, Weifang University, Weifang 261061, People's Republic of China, <sup>b</sup>New Materials and Functional Coordination Chemistry Laboratory, Qingdao University of Science and Technology, Qingdao 266042 People's Republic of China, and <sup>c</sup>Microscale Science Institute, Weifang University, Weifang 261061, People's Republic of China  
Correspondence e-mail: ffjian2008@163.com

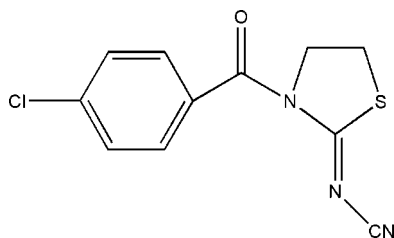
Received 8 October 2008; accepted 7 November 2008

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å; R factor = 0.026; wR factor = 0.066; data-to-parameter ratio = 13.0.

The title compound,  $\text{C}_{11}\text{H}_8\text{ClN}_3\text{OS}$ , was prepared by the reaction of *N*-cyanoiminothiazolidine, 2-aminoethanethiol and triethylamine at 350 K. The dihedral angle between the two rings is  $62.5$  (8)°.

## Related literature

For the biological activities of thiazolidine compounds, see: Iwata *et al.* (1988); Huang & Shi (1990). For related structures, see Jian *et al.* (2006); Schroth *et al.* (1997).



## Experimental

## Crystal data

$\text{C}_{11}\text{H}_8\text{ClN}_3\text{OS}$   
 $M_r = 265.72$   
 Monoclinic,  $P2_1/c$   
 $a = 16.442$  (3) Å  
 $b = 5.6798$  (11) Å  
 $c = 13.313$  (3) Å  
 $\beta = 112.76$  (3)°  
 $V = 1146.5$  (5) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.50$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 $0.34 \times 0.21 \times 0.15$  mm

## Data collection

Bruker SMART CCD area-detector  
 diffractometer  
 Absorption correction: none  
 8343 measured reflections  
 2016 independent reflections  
 1915 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.030$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$   
 $wR(F^2) = 0.066$   
 $S = 1.13$   
 2016 reflections  
 155 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.22$  e Å<sup>-3</sup>

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2128).

## References

- Bruker (2001). *SMART* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin.  
 Huang, Z. T. & Shi, X. (1990). *Synthesis*, **2**, 162–167.  
 Iwata, C., Watanabe, M., Okamoto, S., Fujimoto, M., Sakae, M., Katstrada, M. & Imanishi, T. (1988). *Synthesis*, **3**, 261–262.  
 Jian, F.-F., Zhuang, R.-R., Wang, K.-F., Zhao, P.-S. & Xiao, H.-L. (2006). *Acta Cryst.* **E62**, o3198–o3199.  
 Schroth, W., Hintzsche, E., Jordan, H., Jende, T., Spitzner, R. & Thondorf, I. (1997). *Tetrahedron*, **53**, 7509–7528.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

## supporting information

*Acta Cryst.* (2008). E64, o2321 [doi:10.1107/S1600536808036568]

***N*-[(*Z*)-3-(4-Chlorobenzoyl)-1,3-thiazolidin-2-ylidene]cyanamide****Jian-Gang Wang, Li-Hua Huang and Fang-Fang Jian****S1. Comment**

Thiazolidine is an important group in organic chemistry. Many compounds containing thiazolidine groups possess a broad spectrum of biological activities (Iwata *et al.*, 1988; Huang & Shi, 1990).

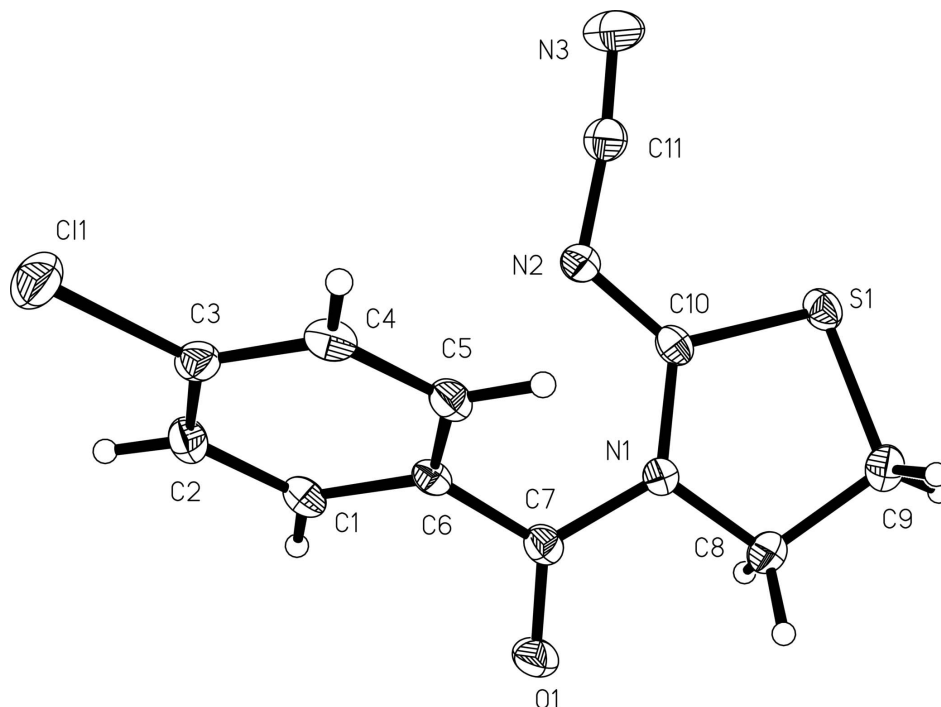
In the crystal structure (Fig. 1), the torsion angle formed by atoms N1, C8, C9 and S1 was 34.5 (9)°. The dihedral angle formed by the the ring (N1, C8, C9, C10 and S1) and the phenyl ring (C1-C6) was 62.5 (8)°. The C=N bond length (1.299 (2) Å) is in agreement with that observed before (Jian *et al.*, 2006). The C—S bond length (1.734 (7) and 1.808 (2) Å) are in agreement with those observed before (Schroth *et al.*, 1997). Intermolecular C—H···N interactions help to stabilize the crystal structure.

**S2. Experimental**

A mixture of *N*-cyanoiminothiazolidine 10 mmol (1.27 g), 2-amino-ethanethiol (1.75 g, 10 mmol) and (1.01 g, 10 mmol) triethylamine was refluxed in absolute acetone (25 ml) for 4 h. On cooling, the product crystallized, was filtered, and recrystallized from absolute EtOH (yield 2.42 g (91%)). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

**S3. Refinement**

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H distances of 0.93–0.97 Å, respectively, and  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}$  of the parent atoms.

**Figure 1**

The molecular structure of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 40% probability level.

### N-[(Z)-3-(4-Chlorobenzoyl)-1,3-thiazolidin-2-ylidene]cyanamide

#### Crystal data

$C_{11}H_8ClN_3OS$

$M_r = 265.72$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2ybc$

$a = 16.442\ (3)\ \text{\AA}$

$b = 5.6798\ (11)\ \text{\AA}$

$c = 13.313\ (3)\ \text{\AA}$

$\beta = 112.76\ (3)^\circ$

$V = 1146.5\ (5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 544$

$D_x = 1.539\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1021 reflections

$\theta = 2.9\text{--}26.4^\circ$

$\mu = 0.50\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, colorless

$0.34 \times 0.21 \times 0.15\ \text{mm}$

#### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

8343 measured reflections

2016 independent reflections

1915 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\text{max}} = 25.0^\circ$ ,  $\theta_{\text{min}} = 3.1^\circ$

$h = -19 \rightarrow 19$

$k = -6 \rightarrow 6$

$l = -15 \rightarrow 15$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.026$   
 $wR(F^2) = 0.066$   
 $S = 1.13$   
 2016 reflections  
 155 parameters  
 0 restraints  
 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  
 $w = 1/[\sigma^2(F_o^2) + (0.017P)^2 + 0.7287P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$   
 Extinction correction: *SHELXL97* (Sheldrick,  
 2008),  $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
 Extinction coefficient: 0.031 (2)

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 ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.07965 (3)	0.52934 (7)	0.33331 (3)	0.02469 (14)
Cl1	0.46689 (3)	0.71315 (8)	0.06338 (3)	0.03512 (15)
O1	0.27367 (8)	1.17756 (19)	0.37317 (10)	0.0302 (3)
N2	0.16368 (9)	0.5912 (2)	0.19639 (11)	0.0244 (3)
C3	0.40564 (10)	0.7898 (3)	0.13961 (12)	0.0229 (3)
N1	0.20390 (8)	0.8283 (2)	0.35254 (10)	0.0216 (3)
C10	0.15500 (10)	0.6503 (3)	0.28595 (12)	0.0201 (3)
C4	0.40148 (10)	0.6326 (3)	0.21690 (12)	0.0232 (3)
H4A	0.4315	0.4901	0.2281	0.028*
N3	0.08333 (10)	0.2509 (3)	0.08619 (14)	0.0426 (4)
C7	0.26182 (10)	0.9843 (3)	0.33103 (12)	0.0220 (3)
C6	0.30963 (9)	0.9060 (3)	0.26219 (12)	0.0197 (3)
C8	0.16984 (11)	0.9017 (3)	0.43566 (13)	0.0263 (4)
H8A	0.1270	1.0272	0.4077	0.032*
H8B	0.2176	0.9574	0.5007	0.032*
C1	0.31667 (10)	1.0641 (3)	0.18625 (13)	0.0227 (3)
H1A	0.2896	1.2106	0.1779	0.027*
C9	0.12728 (11)	0.6865 (3)	0.46101 (13)	0.0268 (4)
H9A	0.0820	0.7312	0.4872	0.032*
H9B	0.1708	0.5901	0.5158	0.032*
C11	0.11795 (11)	0.4075 (3)	0.14112 (14)	0.0275 (4)
C5	0.35225 (10)	0.6895 (3)	0.27740 (12)	0.0222 (3)
H5A	0.3477	0.5833	0.3281	0.027*

C2	0.36370 (10)	1.0057 (3)	0.12284 (13)	0.0244 (3)
H2B	0.3669	1.1093	0.0704	0.029*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0224 (2)	0.0236 (2)	0.0314 (2)	-0.00304 (16)	0.01415 (17)	0.00002 (16)
C11	0.0368 (3)	0.0412 (3)	0.0340 (2)	0.00200 (19)	0.02096 (19)	-0.00607 (19)
O1	0.0369 (7)	0.0210 (6)	0.0374 (7)	-0.0065 (5)	0.0194 (5)	-0.0072 (5)
N2	0.0252 (7)	0.0244 (7)	0.0252 (7)	-0.0060 (6)	0.0115 (6)	-0.0039 (6)
C3	0.0187 (7)	0.0266 (8)	0.0226 (8)	-0.0038 (6)	0.0071 (6)	-0.0060 (6)
N1	0.0246 (7)	0.0207 (7)	0.0217 (6)	-0.0036 (5)	0.0114 (5)	-0.0012 (5)
C10	0.0185 (7)	0.0167 (7)	0.0243 (8)	0.0018 (6)	0.0075 (6)	0.0039 (6)
C4	0.0194 (8)	0.0190 (7)	0.0273 (8)	0.0006 (6)	0.0047 (6)	-0.0016 (6)
N3	0.0337 (8)	0.0427 (10)	0.0586 (11)	-0.0120 (8)	0.0257 (8)	-0.0256 (9)
C7	0.0218 (8)	0.0201 (8)	0.0229 (8)	-0.0012 (6)	0.0072 (6)	0.0024 (6)
C6	0.0171 (7)	0.0187 (7)	0.0219 (7)	-0.0047 (6)	0.0062 (6)	-0.0019 (6)
C8	0.0293 (8)	0.0284 (8)	0.0244 (8)	-0.0013 (7)	0.0140 (7)	-0.0026 (7)
C1	0.0209 (8)	0.0171 (7)	0.0289 (8)	-0.0013 (6)	0.0082 (6)	0.0017 (6)
C9	0.0248 (8)	0.0326 (9)	0.0236 (8)	-0.0002 (7)	0.0101 (7)	0.0028 (7)
C11	0.0230 (8)	0.0303 (9)	0.0348 (9)	-0.0025 (7)	0.0172 (7)	-0.0056 (8)
C5	0.0226 (8)	0.0192 (8)	0.0227 (8)	-0.0029 (6)	0.0064 (6)	0.0022 (6)
C2	0.0242 (8)	0.0248 (8)	0.0245 (8)	-0.0030 (7)	0.0099 (6)	0.0040 (7)

*Geometric parameters (Å, °)*

S1—C10	1.7347 (15)	N3—C11	1.152 (2)
S1—C9	1.8082 (17)	C7—C6	1.488 (2)
C11—C3	1.7390 (16)	C6—C1	1.390 (2)
O1—C7	1.2136 (19)	C6—C5	1.391 (2)
N2—C10	1.299 (2)	C8—C9	1.510 (2)
N2—C11	1.329 (2)	C8—H8A	0.9700
C3—C2	1.382 (2)	C8—H8B	0.9700
C3—C4	1.384 (2)	C1—C2	1.388 (2)
N1—C10	1.379 (2)	C1—H1A	0.9300
N1—C7	1.409 (2)	C9—H9A	0.9700
N1—C8	1.4804 (19)	C9—H9B	0.9700
C4—C5	1.383 (2)	C5—H5A	0.9300
C4—H4A	0.9300	C2—H2B	0.9300
C10—S1—C9	92.05 (8)	N1—C8—H8A	110.5
C10—N2—C11	118.14 (14)	C9—C8—H8A	110.5
C2—C3—C4	121.83 (14)	N1—C8—H8B	110.5
C2—C3—C11	119.55 (12)	C9—C8—H8B	110.5
C4—C3—C11	118.61 (12)	H8A—C8—H8B	108.7
C10—N1—C7	127.01 (13)	C2—C1—C6	120.72 (14)
C10—N1—C8	113.06 (12)	C2—C1—H1A	119.6
C7—N1—C8	117.11 (13)	C6—C1—H1A	119.6

---

N2—C10—N1	122.40 (14)	C8—C9—S1	105.01 (11)
N2—C10—S1	125.37 (12)	C8—C9—H9A	110.7
N1—C10—S1	112.17 (11)	S1—C9—H9A	110.7
C5—C4—C3	119.40 (14)	C8—C9—H9B	110.7
C5—C4—H4A	120.3	S1—C9—H9B	110.7
C3—C4—H4A	120.3	H9A—C9—H9B	108.8
O1—C7—N1	118.36 (14)	N3—C11—N2	172.67 (18)
O1—C7—C6	121.78 (14)	C4—C5—C6	119.80 (14)
N1—C7—C6	119.80 (13)	C4—C5—H5A	120.1
C1—C6—C5	119.87 (14)	C6—C5—H5A	120.1
C1—C6—C7	117.87 (14)	C3—C2—C1	118.32 (14)
C5—C6—C7	122.08 (14)	C3—C2—H2B	120.8
N1—C8—C9	106.32 (13)	C1—C2—H2B	120.8
C11—N2—C10—N1	175.68 (14)	N1—C7—C6—C1	138.53 (15)
C11—N2—C10—S1	-7.2 (2)	O1—C7—C6—C5	130.83 (17)
C7—N1—C10—N2	6.7 (2)	N1—C7—C6—C5	-46.3 (2)
C8—N1—C10—N2	167.01 (14)	C10—N1—C8—C9	29.96 (17)
C7—N1—C10—S1	-170.72 (12)	C7—N1—C8—C9	-167.65 (13)
C8—N1—C10—S1	-10.44 (16)	C5—C6—C1—C2	1.9 (2)
C9—S1—C10—N2	172.92 (14)	C7—C6—C1—C2	177.18 (14)
C9—S1—C10—N1	-9.73 (12)	N1—C8—C9—S1	-34.60 (15)
C2—C3—C4—C5	1.7 (2)	C10—S1—C9—C8	25.81 (12)
C11—C3—C4—C5	-179.26 (11)	C3—C4—C5—C6	-1.8 (2)
C10—N1—C7—O1	152.51 (15)	C1—C6—C5—C4	0.0 (2)
C8—N1—C7—O1	-7.1 (2)	C7—C6—C5—C4	-175.00 (14)
C10—N1—C7—C6	-30.2 (2)	C4—C3—C2—C1	0.2 (2)
C8—N1—C7—C6	170.18 (13)	C11—C3—C2—C1	-178.80 (12)
O1—C7—C6—C1	-44.3 (2)	C6—C1—C2—C3	-2.1 (2)

---