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(E)-Ethyl 2-cyano-2-(thiazolidin-2-ylidene)acetate

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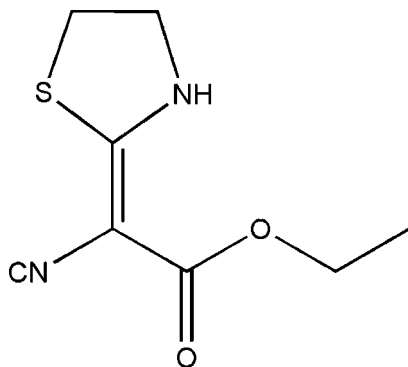
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.051; wR factor = 0.112; data-to-parameter ratio = 13.3.

The title compound, $\text{C}_8\text{H}_{10}\text{N}_2\text{O}_2\text{S}$, was prepared by the reaction of 2-cyano-3,3-bis(methylsulfanyl)acrylate and 2-aminoethanethiol at 350 K. The molecular structure and packing are stabilized by $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bond interactions. All the non-H atoms are nearly in the same plane with the maximum deviation being 0.08 Å.

Related literature

For biological properties of compounds containing thiazolidine groups, see: Huang & Shi (1990); Iwata *et al.* (1988). For related compounds, see: Schroth *et al.* (1997).



Experimental

Crystal data

$\text{C}_8\text{H}_{10}\text{N}_2\text{O}_2\text{S}$	$V = 916.9$ (3) Å ³
$M_r = 198.24$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 4.0676$ (8) Å	$\mu = 0.32$ mm ⁻¹
$b = 15.460$ (3) Å	$T = 293$ (2) K
$c = 14.581$ (3) Å	$0.20 \times 0.12 \times 0.09$ mm
$\beta = 90.03$ (3)°	

Data collection

Bruker SMART CCD area-detector diffractometer	1577 independent reflections
Absorption correction: none	1484 reflections with $I > 2\sigma(I)$
6717 measured reflections	$R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$	119 parameters
$wR(F^2) = 0.112$	H-atom parameters constrained
$S = 1.24$	$\Delta\rho_{\text{max}} = 0.26$ e Å ⁻³
1577 reflections	$\Delta\rho_{\text{min}} = -0.33$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}^i$	0.86	2.33	2.955 (3)	129
$\text{N1}-\text{H1A}\cdots\text{O1}$	0.86	2.12	2.723 (3)	127

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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: AT2647).

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supporting information

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(E)-Ethyl 2-cyano-2-(thiazolidin-2-ylidene)acetate**Jian-Gang Wang and Fang-Fang Jian****S1. Comment**

Thiazolidine is an important kind of group in organic chemistry. Many compounds containing thiazolidine groups possess a broad spectrum of biological activities (Iwata *et al.*, 1988; Huang & Shi, 1990). Here, we report the crystal structure of the title compound (I).

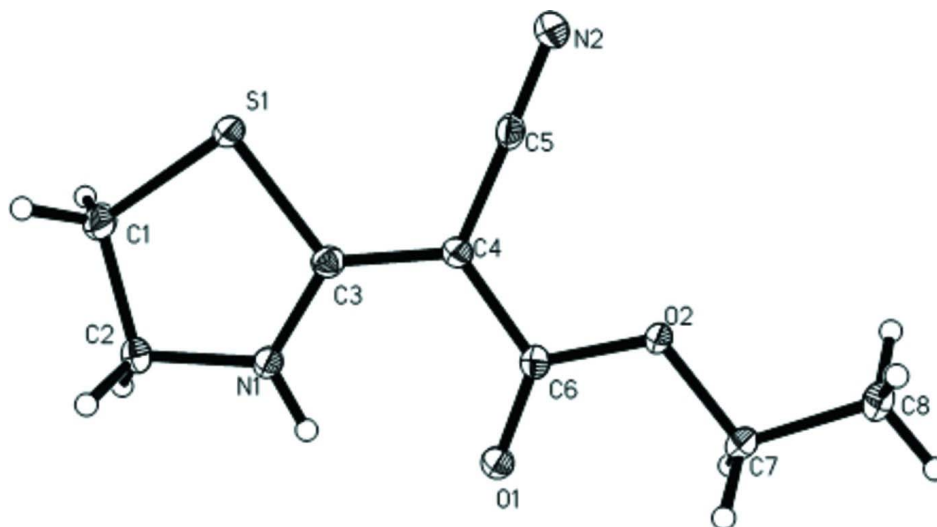
In the crystal structure of (I) (Fig. 1), the torsion angle formed by the N1, C3, S1 and C1 is 4.5 (3)°. All the non-H atoms are nearly the same plane with the maximum deviation of atoms being 0.08 Å. The C—S bond lengths of 1.745 (3) and 1.806 (3) Å are in agreement with those observed before (Schroth *et al.*, 1997). In the crystal structure, there are N—H···O hydrogen-bond interactions to stabilize the crystal structure (Table 12).

S2. Experimental

A mixture of ethyl 2-cyano-3,3-bis(methylthio)acrylate 4 mmol (0.87 g) and 2-amino-ethanethiol (0.32 g, 4.1 mmol) is refluxed in absolute EtOH (25 ml) for 4 h. On cooling, the product crystallized and is filtered, and recrystallized from absolute EtOH [yield 0.67 g (85%)]. 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 N—H and C—H distances of 0.86 and 0.93–0.96 Å, respectively, and with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}$ of the parent atoms.

**Figure 1**

The molecular structure of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

(E)-Ethyl 2-cyano-2-(thiazolidin-2-ylidene)acetate*Crystal data*C₈H₁₀N₂O₂S $M_r = 198.24$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 4.0676$ (8) Å $b = 15.460$ (3) Å $c = 14.581$ (3) Å $\beta = 90.03$ (3)° $V = 916.9$ (3) Å³ $Z = 4$ $F(000) = 416$ $D_x = 1.436$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2422 reflections

 $\theta = 2.3$ – 25.1 ° $\mu = 0.32$ mm⁻¹ $T = 293$ K

Needle, colourless

 $0.20 \times 0.12 \times 0.09$ mm*Data collection*Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

6717 measured reflections

1577 independent reflections

1484 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.025$ $\theta_{\text{max}} = 25.0$ °, $\theta_{\text{min}} = 3.1$ ° $h = -4 \rightarrow 4$ $k = -18 \rightarrow 18$ $l = -17 \rightarrow 17$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.051$ $wR(F^2) = 0.112$ $S = 1.24$

1577 reflections

119 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + 2.059P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.041 (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.

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.2265 (2)	0.70109 (5)	0.01523 (5)	0.0285 (3)
O2	-0.2856 (6)	0.91026 (13)	0.24156 (14)	0.0250 (5)
O1	-0.1022 (6)	0.97114 (14)	0.11080 (15)	0.0325 (6)
N2	-0.1952 (7)	0.68892 (17)	0.23236 (18)	0.0304 (7)
N1	0.1928 (7)	0.86360 (17)	-0.01208 (17)	0.0279 (7)
H1A	0.1440	0.9171	-0.0037	0.034*
C3	0.1091 (7)	0.80489 (19)	0.0489 (2)	0.0209 (6)
C6	-0.1433 (8)	0.9071 (2)	0.1582 (2)	0.0226 (7)
C7	-0.3933 (9)	0.9959 (2)	0.2710 (2)	0.0270 (7)
H7A	-0.5582	1.0182	0.2291	0.032*
H7B	-0.2088	1.0356	0.2717	0.032*
C1	0.3729 (10)	0.7390 (2)	-0.0946 (2)	0.0337 (8)
H1B	0.5946	0.7182	-0.1053	0.040*
H1C	0.2324	0.7174	-0.1433	0.040*
C4	-0.0547 (8)	0.82003 (19)	0.13177 (19)	0.0213 (7)
C5	-0.1330 (7)	0.7482 (2)	0.18828 (19)	0.0220 (7)
C2	0.3686 (8)	0.8366 (2)	-0.0937 (2)	0.0263 (7)
H2A	0.5914	0.8590	-0.0926	0.032*
H2B	0.2593	0.8584	-0.1481	0.032*
C8	-0.5358 (9)	0.9868 (2)	0.3661 (2)	0.0330 (8)
H8A	-0.6097	1.0423	0.3873	0.049*
H8B	-0.3703	0.9650	0.4069	0.049*
H8C	-0.7180	0.9474	0.3645	0.049*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0436 (5)	0.0177 (4)	0.0241 (4)	0.0040 (4)	0.0121 (3)	0.0002 (3)
O2	0.0389 (13)	0.0167 (10)	0.0192 (10)	0.0020 (9)	0.0100 (9)	-0.0013 (8)
O1	0.0527 (15)	0.0197 (11)	0.0251 (12)	0.0021 (11)	0.0141 (11)	0.0033 (9)
N2	0.0444 (17)	0.0255 (14)	0.0215 (14)	0.0008 (13)	0.0075 (12)	0.0017 (12)
N1	0.0421 (17)	0.0182 (13)	0.0234 (14)	0.0058 (12)	0.0128 (12)	0.0028 (10)

C3	0.0237 (15)	0.0194 (14)	0.0197 (15)	0.0003 (13)	-0.0012 (12)	0.0001 (12)
C6	0.0272 (17)	0.0223 (16)	0.0183 (14)	-0.0010 (13)	0.0039 (12)	-0.0021 (12)
C7	0.0385 (19)	0.0182 (15)	0.0244 (16)	0.0012 (14)	0.0057 (14)	-0.0035 (12)
C1	0.051 (2)	0.0258 (17)	0.0245 (17)	0.0088 (16)	0.0135 (15)	0.0034 (13)
C4	0.0274 (16)	0.0180 (15)	0.0183 (14)	-0.0005 (13)	0.0030 (12)	0.0010 (11)
C5	0.0254 (16)	0.0240 (16)	0.0166 (14)	0.0029 (13)	0.0033 (12)	-0.0030 (12)
C2	0.0312 (17)	0.0266 (17)	0.0212 (15)	0.0000 (14)	0.0076 (13)	0.0001 (13)
C8	0.045 (2)	0.0264 (17)	0.0275 (17)	0.0059 (16)	0.0109 (15)	-0.0042 (13)

Geometric parameters (Å, °)

S1—C3	1.745 (3)	C7—C8	1.510 (4)
S1—C1	1.806 (3)	C7—H7A	0.9700
O2—C6	1.348 (3)	C7—H7B	0.9700
O2—C7	1.459 (4)	C1—C2	1.510 (4)
O1—C6	1.218 (4)	C1—H1B	0.9700
N2—C5	1.147 (4)	C1—H1C	0.9700
N1—C3	1.315 (4)	C4—C5	1.420 (4)
N1—C2	1.450 (4)	C2—H2A	0.9700
N1—H1A	0.8600	C2—H2B	0.9700
C3—C4	1.400 (4)	C8—H8A	0.9600
C6—O1	1.218 (4)	C8—H8B	0.9600
C6—C4	1.446 (4)	C8—H8C	0.9600
C3—S1—C1	92.39 (14)	S1—C1—H1B	110.1
C6—O2—C7	115.3 (2)	C2—C1—H1C	110.1
C3—N1—C2	119.0 (3)	S1—C1—H1C	110.1
C3—N1—H1A	120.5	H1B—C1—H1C	108.4
C2—N1—H1A	120.5	C3—C4—C5	118.5 (3)
N1—C3—C4	126.3 (3)	C3—C4—C6	120.3 (3)
N1—C3—S1	111.9 (2)	C5—C4—C6	121.2 (3)
C4—C3—S1	121.8 (2)	N2—C5—C4	178.5 (3)
O1—C6—O2	122.8 (3)	N1—C2—C1	107.5 (2)
O1—C6—O2	122.8 (3)	N1—C2—H2A	110.2
O1—C6—C4	124.8 (3)	C1—C2—H2A	110.2
O1—C6—C4	124.8 (3)	N1—C2—H2B	110.2
O2—C6—C4	112.4 (3)	C1—C2—H2B	110.2
O2—C7—C8	107.5 (2)	H2A—C2—H2B	108.5
O2—C7—H7A	110.2	C7—C8—H8A	109.5
C8—C7—H7A	110.2	C7—C8—H8B	109.5
O2—C7—H7B	110.2	H8A—C8—H8B	109.5
C8—C7—H7B	110.2	C7—C8—H8C	109.5
H7A—C7—H7B	108.5	H8A—C8—H8C	109.5
C2—C1—S1	108.2 (2)	H8B—C8—H8C	109.5
C2—C1—H1B	110.1		
C2—N1—C3—C4	178.7 (3)	S1—C3—C4—C5	-1.6 (4)
C2—N1—C3—S1	-1.4 (4)	N1—C3—C4—C6	-1.1 (5)

C1—S1—C3—N1	-4.5 (3)	S1—C3—C4—C6	178.9 (2)
C1—S1—C3—C4	175.4 (3)	O1—C6—C4—C3	4.2 (5)
O1—O1—C6—O2	0.0 (3)	O1—C6—C4—C3	4.2 (5)
O1—O1—C6—C4	0.0 (3)	O2—C6—C4—C3	-177.2 (3)
C7—O2—C6—O1	1.0 (4)	O1—C6—C4—C5	-175.3 (3)
C7—O2—C6—O1	1.0 (4)	O1—C6—C4—C5	-175.3 (3)
C7—O2—C6—C4	-177.6 (3)	O2—C6—C4—C5	3.3 (4)
C6—O2—C7—C8	-178.4 (3)	C3—N1—C2—C1	8.0 (4)
C3—S1—C1—C2	8.7 (3)	S1—C1—C2—N1	-10.3 (4)
N1—C3—C4—C5	178.4 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots O1 ⁱ	0.86	2.33	2.955 (3)	129
N1—H1A \cdots O1	0.86	2.12	2.723 (3)	127

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