Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*S*)-1-[(*S*)-4-Benzyl-2-thioxothiazolidin-3-yl]-3-hydroxybutan-1-one

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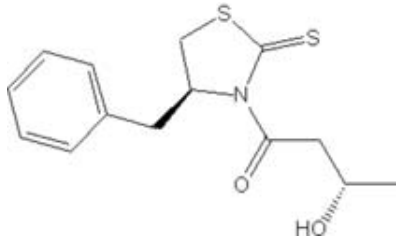
Received 13 November 2007; accepted 29 November 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.040; wR factor = 0.099; data-to-parameter ratio = 16.4.

The title compound, $C_{14}H_{17}NO_2S_2$, was synthesized by asymmetric aldol condensation of *N*-acylthiazolidinethione with acetaldehyde. In the molecule, the thiazolidine five-membered ring assumes an envelope conformation. Intermolecular C—H···O and intramolecular O—H···O and C—H···S hydrogen bonding helps to stabilize the structure.

Related literature

For related literature, see: Crimmins *et al.* (2001); Drück & Littke (1980); Hodge & Olivo (2004).



Experimental

Crystal data

$C_{14}H_{17}NO_2S_2$
M_r = 295.41
Orthorhombic, $P2_12_12_1$
a = 8.0278 (4) Å
b = 8.2637 (4) Å
c = 22.0158 (10) Å
V = 1460.51 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
9324 measured reflections
2845 independent reflections
2648 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.099
S = 1.10
2845 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Absolute structure: Flack (1983), 1175 Friedel pairs
Flack parameter: −0.03 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O1	0.82	2.16	2.765 (3)	131
C4—H4···O2^{i}	0.93	2.45	3.325 (3)	158
C9—H9B···O1^{ii}	0.97	2.48	3.261 (3)	137
C12—H12A···S2	0.97	2.64	3.131 (2)	112

Symmetry codes: (i) $-x+\frac{3}{2}, -y, z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+2$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2003); software used to prepare material for publication: *SHELXTL*.

This work was supported financially by the Science Foundation of China (grant No. 20772026).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2370).

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supplementary materials

Acta Cryst. (2008). E**64**, o228 [**doi:10.1107/S1600536807064355**]

(*S*)-1-[(*S*)-4-Benzyl-2-thioxothiazolidin-3-yl]-3-hydroxybutan-1-one

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Comment

The acyl thiazolidinethione enolates mediated aldol reaction is a well accepted and useful method for the preparation of β-hydroxy acids and their derivatives in high enantiomeric purity. As an important chiral intermediates in the synthesis of our target products, the title compound was synthesized and its crystal structure was determined. The configuration of (I) is in accordance with the model for diastereoselective aldol reaction of acylated chiral thiazolidinethiones derived from amino acids (Crimmins *et al.*, 2001; Hodge & Olivo, 2004).

In the molecule the thiazolidine five membered ring assumes an envelope conformation (Fig. 1). The carbonyl group and the thiocarbonyl group adopt a a S-shaped conformation. The crystal packing is stabilized by the C—H···O, O—H···O and C—H···S hydrogen bonds (Table 1).

Experimental

A solution of *N*-acetyl (4*S*)-benzylthiazolidinethion (1.25 g, 4.98 mmol) in freshly distilled CH_2Cl_2 (30 ml) at 273 K, was treated dropwise with a solution of $TiCl_4$ (5.5 ml, 1 *M* solution in CH_2Cl_2, 5.48 mmol) under nitrogen atmosphere, and the solution allowed to stir for 20 min. To the yellow slurry or suspension was added diisopropylethylamine (4.98 mmol, 0.83 ml). The dark red titanium enolate stirred for 40 min at 273 K. A solution of acetaldehyde (5.5 ml, 1.36 *M* in CH_2Cl_2, 7.47 mmol) was transferred *via* cannula to the reaction mixture, which was then stirred for 1 h at 273 K. The reaction was quenched with half-saturated ammonium chloride (30 ml), and the layers were separated. The organic layer was dried over sodium sulfate, filtered, and concentrated. Purification of the crude material by column chromatography afforded the major diastereomer (0.78 g, 55.8%).

Refinement

H atoms were placed in calculated positions with C—H = 0.93 (aromatic), 0.97 (methylene), 0.96 (methyl), 0.98 Å (methine) and 0.82 Å (hydroxyl), and refined in riding mode with $U_{iso}(H) = xU_{eq}(C)$, $x = 1.5$ for methyl and hydroxyl, $x = 1.2$ for others.

Figures

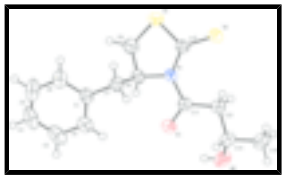

Fig. 1. A view of the molecular structure of (I), with displacement ellipsoids at the 30% probability.

(*S*)-1-[(*S*)-4-Benzyl-2-thioxothiazolidin-3-yl]-3-hydroxybutan-1-one

Crystal data

$C_{14}H_{17}NO_2S_2$	F_{000} = 624
M_r = 295.41	D_x = 1.343 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 4884 reflections
a = 8.0278 (4) Å	θ = 2.5–28.0º
b = 8.2637 (4) Å	μ = 0.36 mm^{-1}
c = 22.0158 (10) Å	T = 294 (2) K
V = 1460.51 (12) Å^3	Plate, yellow
Z = 4	0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2648 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.059
Monochromator: graphite	θ_{max} = 26.0º
T = 294(2) K	θ_{min} = 1.9º
φ and ω scans	h = −9→8
Absorption correction: none	k = −9→10
9324 measured reflections	l = −27→27
2845 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.2097P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.099	$(\Delta/\sigma)_{max}$ = 0.002
S = 1.10	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
2845 reflections	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
174 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1175 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.03 (9)

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8868 (3)	0.3026 (3)	1.10814 (10)	0.0369 (5)
C2	0.8600 (4)	0.1376 (3)	1.09897 (12)	0.0493 (6)
H2	0.8539	0.0965	1.0597	0.059*
C3	0.8424 (4)	0.0351 (4)	1.14808 (15)	0.0638 (8)
H3	0.8246	−0.0748	1.1416	0.077*
C4	0.8510 (4)	0.0937 (4)	1.20634 (14)	0.0666 (9)
H4	0.8376	0.0243	1.2392	0.080*
C5	0.8794 (4)	0.2547 (5)	1.21564 (11)	0.0665 (9)
H5	0.8868	0.2943	1.2551	0.080*
C6	0.8972 (3)	0.3595 (4)	1.16711 (12)	0.0498 (6)
H6	0.9163	0.4689	1.1742	0.060*
C7	0.9009 (3)	0.4147 (3)	1.05432 (10)	0.0385 (5)
H7A	0.9833	0.3728	1.0262	0.046*
H7B	0.9377	0.5204	1.0679	0.046*
C8	0.7330 (3)	0.4310 (3)	1.02180 (9)	0.0347 (5)
H8	0.6802	0.3241	1.0206	0.042*
C9	0.6146 (3)	0.5474 (3)	1.05289 (11)	0.0462 (6)
H9A	0.6386	0.5542	1.0960	0.055*
H9B	0.5000	0.5130	1.0476	0.055*
C10	0.7229 (3)	0.6529 (3)	0.94970 (10)	0.0331 (5)
C11	0.8081 (3)	0.3718 (3)	0.91621 (10)	0.0387 (5)
C12	0.8266 (3)	0.4103 (3)	0.85002 (10)	0.0416 (6)
H12A	0.8955	0.5060	0.8456	0.050*
H12B	0.7178	0.4345	0.8331	0.050*
C13	0.9047 (3)	0.2712 (3)	0.81438 (10)	0.0409 (5)
H13	1.0170	0.2505	0.8299	0.049*
C14	0.9151 (4)	0.3145 (4)	0.74761 (13)	0.0630 (8)
H14A	0.9682	0.2281	0.7258	0.094*
H14B	0.9788	0.4120	0.7429	0.094*
H14C	0.8049	0.3308	0.7318	0.094*
N1	0.7527 (2)	0.4893 (2)	0.95819 (7)	0.0320 (4)
O1	0.8358 (3)	0.2380 (2)	0.93639 (8)	0.0578 (5)
O2	0.8091 (3)	0.1270 (2)	0.81833 (9)	0.0568 (5)
H2A	0.7974	0.1018	0.8541	0.085*
S1	0.65019 (9)	0.74034 (8)	1.01625 (3)	0.04757 (18)
S2	0.74822 (9)	0.76653 (7)	0.88919 (3)	0.04718 (18)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0316 (11)	0.0443 (13)	0.0348 (11)	0.0028 (9)	0.0001 (9)	0.0052 (10)
C2	0.0572 (15)	0.0449 (14)	0.0459 (14)	0.0026 (13)	−0.0003 (12)	0.0050 (11)
C3	0.0629 (18)	0.0514 (16)	0.077 (2)	0.0004 (15)	0.0006 (16)	0.0263 (15)
C4	0.0532 (16)	0.090 (3)	0.0562 (18)	0.0013 (17)	0.0018 (14)	0.0398 (17)
C5	0.0554 (16)	0.111 (3)	0.0331 (12)	−0.003 (2)	−0.0005 (11)	0.0098 (16)
C6	0.0465 (15)	0.0622 (17)	0.0408 (13)	−0.0039 (13)	−0.0024 (11)	0.0038 (12)
C7	0.0402 (12)	0.0407 (13)	0.0347 (11)	−0.0045 (11)	0.0017 (10)	0.0029 (10)
C8	0.0369 (12)	0.0323 (11)	0.0349 (11)	−0.0029 (9)	0.0003 (10)	0.0053 (9)
C9	0.0474 (14)	0.0459 (14)	0.0455 (13)	0.0038 (12)	0.0112 (11)	0.0064 (11)
C10	0.0335 (11)	0.0288 (11)	0.0369 (11)	−0.0006 (9)	−0.0035 (9)	−0.0008 (8)
C11	0.0486 (14)	0.0305 (12)	0.0370 (11)	−0.0002 (10)	−0.0043 (10)	−0.0009 (9)
C12	0.0526 (15)	0.0340 (12)	0.0381 (12)	0.0019 (11)	0.0000 (11)	−0.0004 (9)
C13	0.0426 (12)	0.0385 (13)	0.0417 (11)	−0.0012 (11)	0.0040 (10)	−0.0058 (11)
C14	0.087 (2)	0.0565 (17)	0.0458 (15)	0.0036 (16)	0.0170 (16)	−0.0079 (13)
N1	0.0394 (10)	0.0258 (8)	0.0309 (8)	0.0002 (8)	−0.0015 (8)	0.0029 (7)
O1	0.1009 (15)	0.0318 (9)	0.0407 (8)	0.0148 (11)	−0.0024 (9)	0.0020 (8)
O2	0.0795 (14)	0.0428 (10)	0.0479 (10)	−0.0154 (10)	0.0071 (10)	−0.0102 (8)
S1	0.0624 (4)	0.0364 (3)	0.0439 (3)	0.0114 (3)	0.0089 (3)	−0.0010 (3)
S2	0.0714 (4)	0.0304 (3)	0.0397 (3)	0.0015 (3)	−0.0005 (3)	0.0067 (2)

Geometric parameters (Å, °)

C1—C6	1.383 (3)	C9—H9A	0.9700
C1—C2	1.395 (4)	C9—H9B	0.9700
C1—C7	1.508 (3)	C10—N1	1.386 (3)
C2—C3	1.381 (4)	C10—S2	1.643 (2)
C2—H2	0.9300	C10—S1	1.735 (2)
C3—C4	1.373 (5)	C11—O1	1.212 (3)
C3—H3	0.9300	C11—N1	1.412 (3)
C4—C5	1.365 (5)	C11—C12	1.499 (3)
C4—H4	0.9300	C12—C13	1.526 (3)
C5—C6	1.383 (4)	C12—H12A	0.9700
C5—H5	0.9300	C12—H12B	0.9700
C6—H6	0.9300	C13—O2	1.421 (3)
C7—C8	1.532 (3)	C13—C14	1.515 (4)
C7—H7A	0.9700	C13—H13	0.9800
C7—H7B	0.9700	C14—H14A	0.9600
C8—N1	1.489 (2)	C14—H14B	0.9600
C8—C9	1.515 (3)	C14—H14C	0.9600
C8—H8	0.9800	O2—H2A	0.8200
C9—S1	1.809 (2)		
C6—C1—C2	118.5 (2)	C8—C9—H9B	110.7
C6—C1—C7	121.6 (2)	S1—C9—H9B	110.7
C2—C1—C7	119.9 (2)	H9A—C9—H9B	108.8

C3—C2—C1	120.1 (3)	N1—C10—S2	130.26 (17)
C3—C2—H2	119.9	N1—C10—S1	110.52 (16)
C1—C2—H2	119.9	S2—C10—S1	119.22 (13)
C4—C3—C2	120.6 (3)	O1—C11—N1	116.4 (2)
C4—C3—H3	119.7	O1—C11—C12	122.1 (2)
C2—C3—H3	119.7	N1—C11—C12	121.4 (2)
C5—C4—C3	119.5 (3)	C11—C12—C13	112.4 (2)
C5—C4—H4	120.2	C11—C12—H12A	109.1
C3—C4—H4	120.2	C13—C12—H12A	109.1
C4—C5—C6	120.8 (3)	C11—C12—H12B	109.1
C4—C5—H5	119.6	C13—C12—H12B	109.1
C6—C5—H5	119.6	H12A—C12—H12B	107.9
C5—C6—C1	120.4 (3)	O2—C13—C14	106.7 (2)
C5—C6—H6	119.8	O2—C13—C12	112.25 (19)
C1—C6—H6	119.8	C14—C13—C12	110.1 (2)
C1—C7—C8	110.84 (18)	O2—C13—H13	109.2
C1—C7—H7A	109.5	C14—C13—H13	109.2
C8—C7—H7A	109.5	C12—C13—H13	109.2
C1—C7—H7B	109.5	C13—C14—H14A	109.5
C8—C7—H7B	109.5	C13—C14—H14B	109.5
H7A—C7—H7B	108.1	H14A—C14—H14B	109.5
N1—C8—C9	106.63 (17)	C13—C14—H14C	109.5
N1—C8—C7	112.01 (18)	H14A—C14—H14C	109.5
C9—C8—C7	113.4 (2)	H14B—C14—H14C	109.5
N1—C8—H8	108.2	C10—N1—C11	129.54 (18)
C9—C8—H8	108.2	C10—N1—C8	115.08 (17)
C7—C8—H8	108.2	C11—N1—C8	115.27 (17)
C8—C9—S1	105.01 (15)	C13—O2—H2A	109.5
C8—C9—H9A	110.7	C10—S1—C9	93.58 (11)
S1—C9—H9A	110.7		
C6—C1—C2—C3	0.7 (4)	C11—C12—C13—C14	178.1 (2)
C7—C1—C2—C3	−178.2 (3)	S2—C10—N1—C11	−2.5 (4)
C1—C2—C3—C4	0.0 (5)	S1—C10—N1—C11	178.28 (19)
C2—C3—C4—C5	−0.9 (5)	S2—C10—N1—C8	173.37 (18)
C3—C4—C5—C6	0.9 (5)	S1—C10—N1—C8	−5.8 (2)
C4—C5—C6—C1	−0.1 (4)	O1—C11—N1—C10	174.9 (2)
C2—C1—C6—C5	−0.7 (4)	C12—C11—N1—C10	−6.8 (4)
C7—C1—C6—C5	178.2 (2)	O1—C11—N1—C8	−1.0 (3)
C6—C1—C7—C8	−110.2 (3)	C12—C11—N1—C8	177.3 (2)
C2—C1—C7—C8	68.7 (3)	C9—C8—N1—C10	24.8 (3)
C1—C7—C8—N1	−159.54 (18)	C7—C8—N1—C10	−99.7 (2)
C1—C7—C8—C9	79.7 (2)	C9—C8—N1—C11	−158.7 (2)
N1—C8—C9—S1	−30.8 (2)	C7—C8—N1—C11	76.8 (2)
C7—C8—C9—S1	92.89 (19)	N1—C10—S1—C9	−11.90 (18)
O1—C11—C12—C13	−7.6 (4)	S2—C10—S1—C9	168.79 (15)
N1—C11—C12—C13	174.2 (2)	C8—C9—S1—C10	25.09 (19)
C11—C12—C13—O2	59.4 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2A···O1	0.82	2.16	2.765 (3)	131
C4—H4···O2^{i}	0.93	2.45	3.325 (3)	158
C9—H9B···O1^{ii}	0.97	2.48	3.261 (3)	137
C12—H12A···S2	0.97	2.64	3.131 (2)	112

Symmetry codes: (i) −*x*+3/2, −*y*, *z*+1/2; (ii) *x*−1/2, −*y*+1/2, −*z*+2.

Fig. 1