Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

rac-Ethyl 2-amino-3-hydroxy-3-[4-(methylsulfonyl)phenyl]propanoate

Hao Hu,* Yue-Hu Chen, Shao-Song Qian and Shou-Kai Kang

School of Life Science, ShanDong University of Technology, ZiBo 255049, People's Republic of China
Correspondence e-mail: huhao@sdut.edu.cn

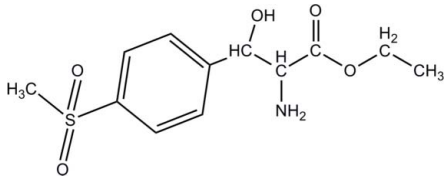
Received 14 November 2010; accepted 16 December 2010

Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.004 Å; R factor = 0.060; wR factor = 0.179; data-to-parameter ratio = 13.6.

In the title compound, $C_{12}H_{17}NO_5S$, the orientations of the 2-ethyl-2-amino-3-hydroxypropanoate group and the 4-methylsulfonyl moiety towards the aromatic ring are periplanar and (−)-anticlinal, respectively. In the crystal packing, the dominant interaction is O—H···N hydrogen bonding, which generates a chain running along [100]. N—H···O and C—H···O interactions are also observed.

Related literature

The title compound is an intermediate in the synthesis of florfenicol, a broad spectrum antibiotic currently used in veterinary medicine, see: Gregory (1957); Syriopoulou & Harding (1981).



Experimental

Crystal data

$C_{12}H_{17}NO_5S$
M_r = 287.33
Triclinic, $P\overline{1}$
a = 4.8123 (11) Å
b = 11.382 (3) Å
c = 12.637 (3) Å
α = 94.952 (4)°
β = 100.530 (4)°
γ = 94.298 (4)°
V = 675.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 273 K
0.18 × 0.16 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
3543 measured reflections
2363 independent reflections
1786 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.179
S = 1.04
2363 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···N1^{i}	0.82	1.97	2.784 (3)	174
N1—H1A···O3^{ii}	0.89	2.25	3.088 (3)	158
C7—H7A···O2^{i}	0.96	2.57	3.227 (4)	126

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+2, -z+1$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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*.

This project was sponsored by Doctoral Research Foundation, Shandong University of Technology, People's Republic of China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2292).

References

Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Gregory, W. A. (1957). US Patent 2 816 915.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Syriopoulou, V. P. & Harding, A. L. (1981). *Antimicrob. Agents Chemother.* **19**, 294–297.

supporting information

Acta Cryst. (2011). E**67**, o258 [doi:10.1107/S1600536810052827]

rac-Ethyl 2-amino-3-hydroxy-3-[4-(methylsulfonyl)phenyl]propanoate

Hao Hu, Yue-Hu Chen, Shao-Song Qian and Shou-Kai Kang

S1. Comment

The title compound (Fig. 1)is an intermediate of florfenicol, a fluorinated synthetic analog of thiamphenicol, which is currently indicated for the treatment of bovine respiratory disease (BRD) associated with Mannheimia (Pasteurella) haemolytica, Pasteurella multocida, and Haemophilus somnus, and for treatment of bovine interdigital phlegmon (foot rot, acute interdigital necrobacillosis, infectious pododermatitis) associated with Fusobacterium necrophorum and Bacteroides melaninogenicus (Syriopoulou, *et al.*, 1981). The chiral title molecule crystallises in centrosymmetric triclinic space group implying a racemic crystal. The two stereogenic centres C8 and C9 are of opposite chirality. The dominant interaction is hydrogen bond O3-H···N1 which generates a chain along the direction [100] (Fig. 2).

S2. Experimental

The compound (rac)-ethyl 2-amino-3-hydroxy-3-(4-(methylsulfonyl)phenyl) propanoate was synthesised according to a US patent (Gregory, 1957). 4-(Methylsulfonyl)benzaldehyde (18.4 g, 0.1 mol) reacted with 2-aminoacetic acid (7.5 g, 0.1 mol) and potassium carbonate (29.2 g, 0.21 mol). As a result, 14.2 g of (rac)-2-amino-3-hydroxy-3-(4-(methylsulfonyl)-phenyl)propanoic acid was obtained (yield, 55%). The amount of 9.3 g of the title compound was obtained through the reaction of (rac)-2-amino-3-hydroxy-3-(4-(methylsulfonyl)phenyl)propanoic acid (14.2 g, 0.055 mol), ethanol (3.3 mL) and 20 mL of sulfuric acid under reflux for 30 min. Subsequently, 0.1 g of the title compound was dissolved in tetra-hydrofurane (5 mL) and single crystals suitable for X-ray diffraction were obtained by spontaneous evaporation of the solution.

S3. Refinement

All the H atoms attached to C atoms were placed in geometrical positions and constrained to ride on their parent atoms with C—H distance in the range 0.93–0.98 Å, They were treated as riding atoms, with U_{iso}(H) = 1.2U_{eq}(C).

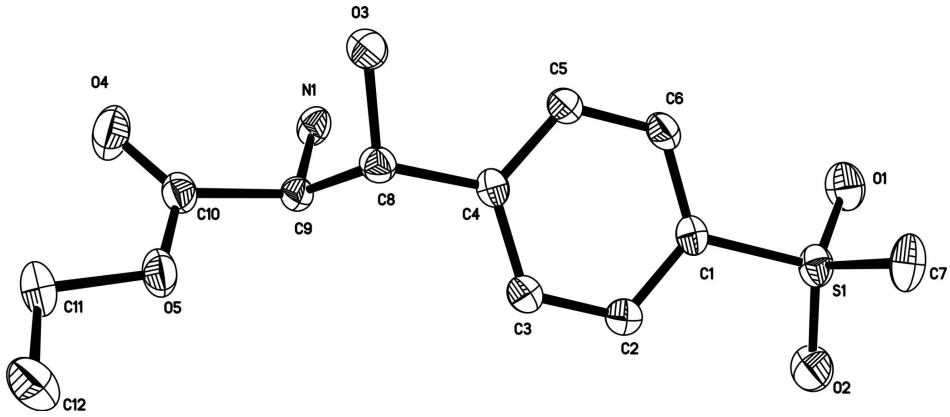


Figure 1

The structure of I showing the atom labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

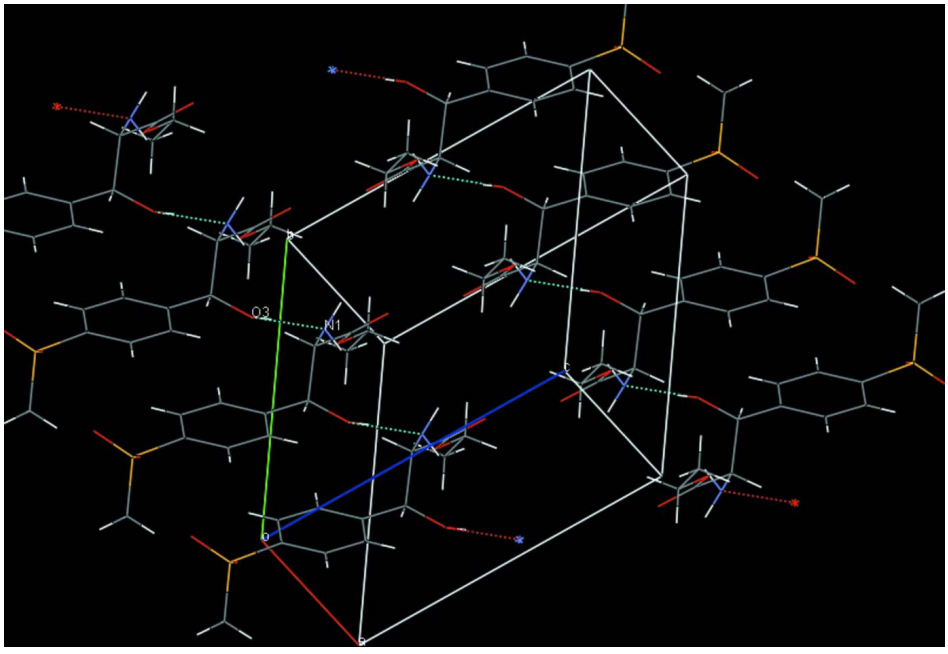


Figure 2

Crystal packing with intermolecular hydrogen bonds generating a chain along [100].

rac-Ethyl 2-amino-3-hydroxy-3-[4-(methylsulfonyl)phenyl]propanoate

Crystal data

$C_{12}H_{17}NO_5S$
$M_r = 287.33$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.8123\ (11)$ Å
$b = 11.382\ (3)$ Å
$c = 12.637\ (3)$ Å
$\alpha = 94.952\ (4)°$
$\beta = 100.530\ (4)°$
$\gamma = 94.298\ (4)°$
$V = 675.1\ (3)$ Å^3
$Z = 2$
$F(000) = 304$
$D_x = 1.414$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1152 reflections
$\theta = 2.3–25.9°$
$\mu = 0.26$ mm^{-1}
$T = 273$ K
Prism, colourless
0.18 × 0.16 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
3543 measured reflections
2363 independent reflections
1786 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 25.1°$, $\theta_{min} = 1.7°$
$h = -4 \rightarrow 5$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.179$
$S = 1.04$
2363 reflections
174 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
$w = 1/[\sigma^2(F_o^2) + (0.119P)^2 + 0.0021P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.75$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	1.45493 (14)	1.26807 (6)	0.93597 (7)	0.0422 (3)
O1	1.5681 (5)	1.3462 (2)	0.8678 (2)	0.0554 (7)
O2	1.6472 (5)	1.2080 (2)	1.0081 (2)	0.0634 (7)
O3	0.3600 (4)	0.95918 (17)	0.60055 (17)	0.0415 (5)
H3	0.2045	0.9207	0.5865	0.050*
O4	0.3671 (6)	0.6609 (2)	0.4925 (2)	0.0669 (8)
O5	0.3457 (5)	0.65735 (18)	0.66724 (18)	0.0505 (6)
N1	0.8171 (5)	0.8421 (2)	0.5424 (2)	0.0385 (6)
H1A	0.7266	0.8992	0.5112	0.046*
H1B	0.8181	0.7816	0.4930	0.046*
C1	1.2030 (6)	1.1614 (2)	0.8552 (2)	0.0355 (7)
C2	1.1603 (6)	1.0510 (3)	0.8907 (3)	0.0422 (7)
H2	1.2735	1.0315	0.9535	0.051*
C3	0.9497 (6)	0.9707 (3)	0.8325 (2)	0.0421 (7)
H3A	0.9218	0.8963	0.8561	0.050*
C4	0.7770 (6)	0.9980 (2)	0.7391 (2)	0.0323 (6)
C5	0.8256 (6)	1.1086 (2)	0.7039 (3)	0.0397 (7)

H5	0.7141	1.1282	0.6408	0.048*
C6	1.0380 (6)	1.1896 (2)	0.7618 (3)	0.0431 (8)
H6	1.0697	1.2634	0.7377	0.052*
C7	1.2543 (7)	1.3513 (3)	1.0121 (3)	0.0558 (9)
H7A	1.1644	1.3006	1.0553	0.084*
H7B	1.1120	1.3860	0.9643	0.084*
H7C	1.3767	1.4128	1.0585	0.084*
C8	0.5434 (6)	0.9065 (2)	0.6795 (2)	0.0348 (7)
H8	0.4342	0.8766	0.7314	0.042*
C9	0.6716 (6)	0.8019 (2)	0.6256 (2)	0.0337 (7)
H9	0.8140	0.7743	0.6814	0.040*
C10	0.4449 (6)	0.6998 (2)	0.5853 (3)	0.0401 (7)
C11	0.1378 (8)	0.5547 (3)	0.6424 (3)	0.0554 (9)
H11A	0.2228	0.4867	0.6148	0.067*
H11B	−0.0212	0.5704	0.5879	0.067*
C12	0.0405 (8)	0.5310 (3)	0.7433 (3)	0.0647 (11)
H12A	0.1986	0.5138	0.7960	0.097*
H12B	−0.1006	0.4645	0.7289	0.097*
H12C	−0.0398	0.5994	0.7705	0.097*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0315 (4)	0.0384 (5)	0.0528 (5)	−0.0036 (3)	0.0039 (3)	−0.0026 (4)
O1	0.0479 (13)	0.0479 (13)	0.0689 (16)	−0.0132 (10)	0.0156 (12)	0.0027 (12)
O2	0.0454 (13)	0.0540 (14)	0.0788 (18)	−0.0011 (11)	−0.0159 (13)	0.0042 (13)
O3	0.0276 (10)	0.0386 (11)	0.0564 (13)	0.0004 (8)	0.0012 (9)	0.0102 (10)
O4	0.0795 (18)	0.0663 (16)	0.0462 (15)	−0.0263 (13)	0.0091 (13)	−0.0087 (13)
O5	0.0611 (14)	0.0390 (12)	0.0473 (13)	−0.0160 (10)	0.0106 (11)	−0.0009 (10)
N1	0.0321 (12)	0.0403 (14)	0.0422 (15)	0.0027 (10)	0.0053 (11)	0.0040 (11)
C1	0.0334 (14)	0.0299 (15)	0.0425 (17)	0.0007 (12)	0.0090 (13)	−0.0010 (13)
C2	0.0459 (17)	0.0370 (16)	0.0407 (17)	0.0028 (13)	0.0004 (14)	0.0052 (14)
C3	0.0475 (17)	0.0328 (15)	0.0438 (18)	−0.0024 (13)	0.0046 (14)	0.0065 (13)
C4	0.0323 (14)	0.0286 (14)	0.0366 (16)	0.0024 (11)	0.0092 (12)	0.0009 (12)
C5	0.0413 (16)	0.0316 (15)	0.0433 (17)	0.0012 (12)	0.0003 (13)	0.0061 (13)
C6	0.0471 (17)	0.0278 (15)	0.0517 (19)	−0.0006 (13)	0.0023 (15)	0.0072 (14)
C7	0.0475 (18)	0.051 (2)	0.064 (2)	−0.0121 (15)	0.0144 (17)	−0.0194 (17)
C8	0.0319 (14)	0.0327 (15)	0.0395 (17)	0.0013 (12)	0.0051 (12)	0.0069 (13)
C9	0.0323 (14)	0.0304 (14)	0.0368 (16)	0.0024 (12)	0.0027 (12)	0.0031 (12)
C10	0.0412 (16)	0.0309 (15)	0.0464 (19)	0.0025 (13)	0.0057 (14)	0.0008 (14)
C11	0.059 (2)	0.0346 (17)	0.068 (2)	−0.0119 (15)	0.0118 (18)	−0.0042 (16)
C12	0.074 (3)	0.049 (2)	0.074 (3)	−0.0032 (18)	0.021 (2)	0.0179 (19)

Geometric parameters (Å, º)

S1—O2	1.432 (2)	C4—C5	1.389 (4)
S1—O1	1.436 (2)	C4—C8	1.515 (4)
S1—C7	1.753 (4)	C5—C6	1.378 (4)

S1—C1	1.760 (3)	C5—H5	0.9300
O3—C8	1.412 (3)	C6—H6	0.9300
O3—H3	0.8200	C7—H7A	0.9600
O4—C10	1.198 (4)	C7—H7B	0.9600
O5—C10	1.329 (4)	C7—H7C	0.9600
O5—C11	1.453 (4)	C8—C9	1.544 (4)
N1—C9	1.452 (4)	C8—H8	0.9800
N1—H1A	0.8900	C9—C10	1.518 (4)
N1—H1B	0.8900	C9—H9	0.9800
C1—C6	1.374 (4)	C11—C12	1.475 (5)
C1—C2	1.384 (4)	C11—H11A	0.9700
C2—C3	1.370 (4)	C11—H11B	0.9700
C2—H2	0.9300	C12—H12A	0.9600
C3—C4	1.387 (4)	C12—H12B	0.9600
C3—H3A	0.9300	C12—H12C	0.9600

O2—S1—O1	118.80 (15)	H7A—C7—H7B	109.5
O2—S1—C7	108.53 (18)	S1—C7—H7C	109.5
O1—S1—C7	107.05 (17)	H7A—C7—H7C	109.5
O2—S1—C1	108.27 (14)	H7B—C7—H7C	109.5
O1—S1—C1	109.22 (14)	O3—C8—C4	109.8 (2)
C7—S1—C1	103.98 (15)	O3—C8—C9	110.1 (2)
C8—O3—H3	109.5	C4—C8—C9	110.3 (2)
C10—O5—C11	117.5 (3)	O3—C8—H8	108.9
C9—N1—H1A	109.3	C4—C8—H8	108.9
C9—N1—H1B	109.2	C9—C8—H8	108.9
H1A—N1—H1B	109.5	N1—C9—C10	113.8 (2)
C6—C1—C2	120.3 (3)	N1—C9—C8	110.1 (2)
C6—C1—S1	120.6 (2)	C10—C9—C8	110.3 (2)
C2—C1—S1	119.0 (2)	N1—C9—H9	107.4
C3—C2—C1	119.3 (3)	C10—C9—H9	107.4
C3—C2—H2	120.4	C8—C9—H9	107.4
C1—C2—H2	120.4	O4—C10—O5	124.1 (3)
C2—C3—C4	121.4 (3)	O4—C10—C9	125.1 (3)
C2—C3—H3A	119.3	O5—C10—C9	110.8 (2)
C4—C3—H3A	119.3	O5—C11—C12	107.6 (3)
C3—C4—C5	118.4 (3)	O5—C11—H11A	110.2
C3—C4—C8	119.0 (2)	C12—C11—H11A	110.2
C5—C4—C8	122.5 (2)	O5—C11—H11B	110.2
C6—C5—C4	120.4 (3)	C12—C11—H11B	110.2
C6—C5—H5	119.8	H11A—C11—H11B	108.5
C4—C5—H5	119.8	C11—C12—H12A	109.5
C1—C6—C5	120.1 (3)	C11—C12—H12B	109.5
C1—C6—H6	119.9	H12A—C12—H12B	109.5
C5—C6—H6	119.9	C11—C12—H12C	109.5
S1—C7—H7A	109.5	H12A—C12—H12C	109.5
S1—C7—H7B	109.5	H12B—C12—H12C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···N1^{i}	0.82	1.97	2.784 (3)	174
N1—H1*A*···O3^{ii}	0.89	2.25	3.088 (3)	158
C7—H7*A*···O2^{i}	0.96	2.57	3.227 (4)	126

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+2, -z+1$.