

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4-(1,3-Thiazolidin-2-yl)phenol

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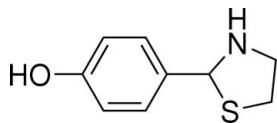
Received 16 September 2009; accepted 14 October 2009

 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.037; wR factor = 0.105; data-to-parameter ratio = 16.7.

In the title compound, $\text{C}_9\text{H}_{11}\text{NOS}$, the thiazolidinyl ring is almost perpendicular to the phenyl ring with $\text{N}-\text{C}-\text{C}-\text{C}$ torsion angles of $71.7(2)$ and $107.1(2)^\circ$. In the crystal, molecules are connected via $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds, forming layers.

Related literature

For the cyclization of 2-amino-ethanthiol Schiff bases, see: Al-Sayyab *et al.* (1968); Stacy & Strong (1967); Thompson & Busch (1964).



Experimental

Crystal data

 $\text{C}_9\text{H}_{11}\text{NOS}$
 $M_r = 181.25$

 Orthorhombic, $Pbca$
 $a = 12.3638(6)$ Å

 $b = 8.9683(5)$ Å

 $c = 15.8249(8)$ Å

 $V = 1754.7(2)$ Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 0.32$ mm⁻¹
 $T = 173$ K

 $0.47 \times 0.45 \times 0.16$ mm

Data collection

Bruker SMART 1000 CCD diffractometer

 Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)

 $T_{\min} = 0.865$, $T_{\max} = 0.951$

9635 measured reflections

1919 independent reflections

 1615 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.105$
 $S = 1.07$

1919 reflections

115 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 0.37$ e Å⁻³
 $\Delta\rho_{\min} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^{\text{i}}$	0.85 (2)	2.28 (2)	3.073 (2)	156 (2)
$\text{O1}-\text{H1A}\cdots\text{N1}^{\text{ii}}$	0.82 (2)	1.91 (2)	2.713 (2)	164 (2)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The author thanks the National Science Foundation of China for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2144).

References

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supporting information

Acta Cryst. (2009). E65, o2799 [https://doi.org/10.1107/S1600536809042135]

4-(1,3-Thiazolidin-2-yl)phenol

Xue-Mei Yang

S1. Comment

In our search for a new synthetic route to imipenem, a carbapenem antibiotic, we got a thiazolidine compound from a reaction of *p*-hydroxybenzaldehyde with 2-amino-ethanthiol, despite of our initial plan to prepare a Schiff base compound. This is consistent with reports that the 2-amino-ethanthiol Schiff base compounds can undergo intramolecular cyclization to form thiazolidines (Al-Sayyab *et al.*, 1968; Thompson & Busch, 1964; Stacy & Strong, 1967).

In the molecular structure (Fig. 1), as it is expected the thiazolidinyl ring is not planar, showing a N(1)—C(1)—C(2)—S(1) torsion angle of $-33.7(2)^\circ$. Furthermore, the thiazolidinyl ring is almost perpendicular to the phenyl ring, with torsion angles N(1)—C(3)—C(4)—C(9) of $71.7(2)^\circ$ and N(1)—C(3)—C(4)—C(5) of $107.1(2)^\circ$. In Fig. 1 the chiral center C(3) adopts *R* configuration. Nevertheless, due to space group symmetry a racemate has been formed and both enantiomers are present in the crystal structure.

In the crystal structure two adjacent molecules are connected *via* N—H \cdots O and O—H \cdots N hydrogen bonds to form centrosymmetric molecule pairs. These pairs are further linked by additional N—H \cdots O and O—H \cdots N intermolecular hydrogen bonds leading to the observed layered supramolecular (Fig. 2).

S2. Experimental

2-Amino-ethanthiol 0.77 g (0.001 mol) was mixed with *p*-hydroxybenzaldehyde 1.22 g (0.001 mol) in ethanol (10 ml) and the mixture refluxed for 2 h. The solvent was evaporated to dryness under reduced pressure and the remaining residue recrystallized from ethanol to afford 1.5 g of yellow block crystals. (Yield 85%). Crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanolic solution. Spectroscopic analysis: ^1H NMR (DMSO- d_6 , δ , p.p.m.): 2.75–2.90 (m, 2H), 2.85–3.05 (m, 2H), 3.50 (m, 1H), 5.35 (s, 1H), 6.70 (d, 2H), 7.25 (d, 2H), 9.35 (s, 1H); elemental analysis, calculated for $\text{C}_9\text{H}_{11}\text{NOS}$: C, 59.67; H, 6.08; N, 7.73; found: C, 59.33; H, 5.93; N 7.41%.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model with $d(\text{C—H}) = 0.95 \text{ \AA}$, $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for aromatic 1.00 \AA , $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for CH, 0.99 \AA , $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ for CH_2 and 0.88 \AA , $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{N})$ for the NH atoms.

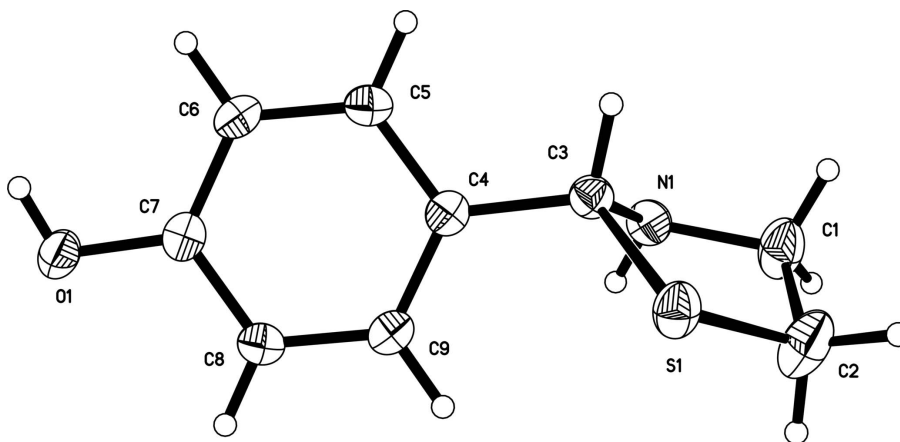


Figure 1

The molecular structure with thermal ellipsoids drawn at the 30% probability level.

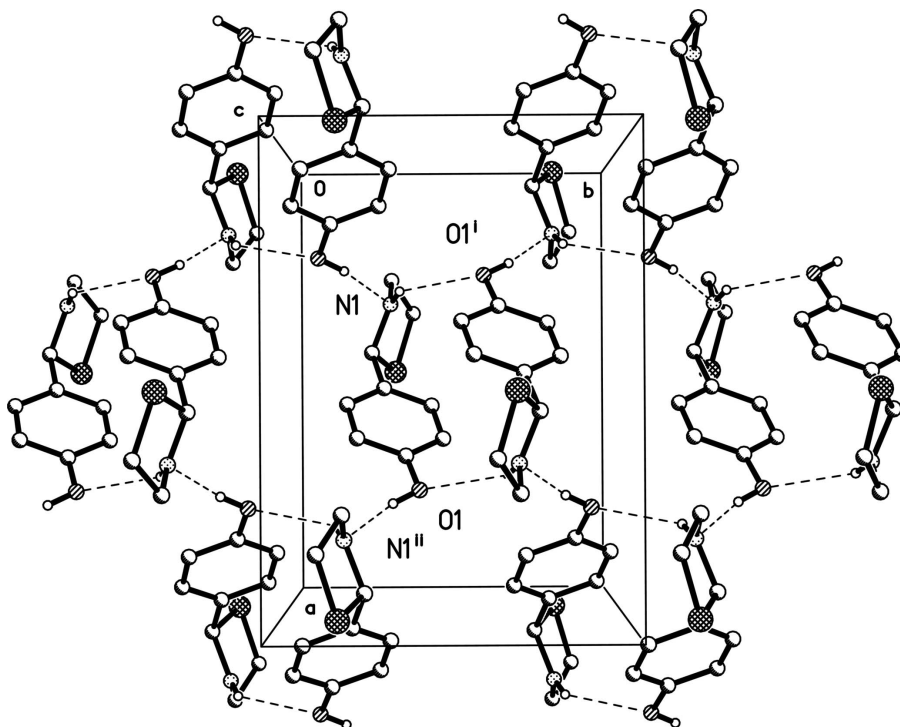


Figure 2

Crystal lattice along *c* axis. H atoms not involved in hydrogen bonds have been omitted for clarity.

4-(1,3-Thiazolidin-2-yl)phenol

Crystal data

$C_9H_{11}NOS$

$M_r = 181.25$

Orthorhombic, *Pbca*

Hall symbol: $-P\ 2ac\ 2ab$

$a = 12.3638\ (6)\ \text{\AA}$

$b = 8.9683\ (5)\ \text{\AA}$

$c = 15.8249\ (8)\ \text{\AA}$

$V = 1754.7\ (2)\ \text{\AA}^3$

$Z = 8$

$F(000) = 768$

$D_x = 1.372\ \text{Mg m}^{-3}$

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

Cell parameters from 4931 reflections

$\theta = 2.6\text{--}27.0^\circ$

$\mu = 0.32 \text{ mm}^{-1}$
 $T = 173 \text{ K}$

Block, colorless
 $0.47 \times 0.45 \times 0.16 \text{ mm}$

Data collection

Bruker SMART 1000 CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2004)
 $T_{\min} = 0.865, T_{\max} = 0.951$

9635 measured reflections
 1919 independent reflections
 1615 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\max} = 27.0^\circ, \theta_{\min} = 2.6^\circ$
 $h = -15 \rightarrow 15$
 $k = -11 \rightarrow 8$
 $l = -20 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.105$
 $S = 1.07$
 1919 reflections
 115 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 atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 0.7197P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.37 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.47979 (3)	0.18010 (5)	0.70817 (2)	0.02679 (16)
C1	0.27121 (14)	0.1895 (2)	0.74816 (11)	0.0355 (4)
H1B	0.2501	0.2924	0.7327	0.043*
H1C	0.2046	0.1288	0.7530	0.043*
C2	0.34426 (15)	0.1245 (3)	0.67981 (12)	0.0417 (5)
H2A	0.3245	0.1646	0.6236	0.050*
H2B	0.3381	0.0145	0.6783	0.050*
C3	0.43704 (12)	0.24880 (18)	0.81446 (9)	0.0217 (3)
H3	0.4323	0.3600	0.8119	0.026*
C4	0.51646 (12)	0.20804 (17)	0.88292 (9)	0.0209 (3)
C5	0.55139 (13)	0.31669 (17)	0.93977 (10)	0.0235 (3)
H5	0.5255	0.4159	0.9342	0.028*
C6	0.62307 (13)	0.28297 (18)	1.00422 (10)	0.0248 (4)

H6	0.6461	0.3587	1.0421	0.030*
C7	0.66115 (13)	0.13804 (18)	1.01328 (9)	0.0227 (3)
C8	0.62657 (13)	0.02820 (18)	0.95705 (10)	0.0239 (3)
H8	0.6521	-0.0712	0.9630	0.029*
C9	0.55519 (12)	0.06344 (18)	0.89265 (10)	0.0229 (3)
H9	0.5323	-0.0122	0.8546	0.027*
N1	0.32818 (11)	0.19058 (16)	0.82922 (9)	0.0249 (3)
H1	0.3303 (16)	0.103 (3)	0.8493 (12)	0.030*
O1	0.73103 (10)	0.09658 (14)	1.07553 (7)	0.0302 (3)
H1A	0.7567 (19)	0.172 (3)	1.0971 (13)	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0253 (2)	0.0357 (3)	0.0194 (2)	0.00118 (16)	0.00148 (14)	-0.00048 (16)
C1	0.0239 (9)	0.0512 (12)	0.0313 (9)	0.0019 (8)	-0.0045 (7)	-0.0055 (8)
C2	0.0308 (9)	0.0616 (14)	0.0326 (9)	-0.0052 (9)	-0.0028 (8)	-0.0139 (9)
C3	0.0218 (7)	0.0221 (8)	0.0213 (7)	0.0008 (6)	0.0012 (6)	-0.0004 (6)
C4	0.0213 (7)	0.0232 (8)	0.0182 (7)	-0.0019 (6)	0.0023 (6)	0.0005 (6)
C5	0.0265 (8)	0.0188 (7)	0.0252 (8)	-0.0003 (6)	0.0013 (6)	-0.0004 (6)
C6	0.0283 (8)	0.0229 (8)	0.0233 (7)	-0.0042 (6)	-0.0009 (6)	-0.0046 (6)
C7	0.0214 (7)	0.0271 (8)	0.0197 (7)	-0.0034 (6)	0.0012 (6)	0.0013 (6)
C8	0.0259 (8)	0.0204 (7)	0.0254 (8)	0.0011 (6)	0.0009 (6)	-0.0003 (6)
C9	0.0236 (7)	0.0233 (8)	0.0218 (7)	-0.0033 (6)	0.0008 (6)	-0.0027 (6)
N1	0.0219 (7)	0.0266 (7)	0.0262 (7)	0.0003 (5)	0.0013 (5)	0.0001 (6)
O1	0.0337 (7)	0.0275 (6)	0.0293 (6)	-0.0026 (5)	-0.0117 (5)	-0.0012 (5)

Geometric parameters (Å, °)

S1—C2	1.8049 (19)	C4—C5	1.395 (2)
S1—C3	1.8676 (15)	C5—C6	1.385 (2)
C1—N1	1.463 (2)	C5—H5	0.9500
C1—C2	1.525 (3)	C6—C7	1.390 (2)
C1—H1B	0.9900	C6—H6	0.9500
C1—H1C	0.9900	C7—O1	1.3620 (19)
C2—H2A	0.9900	C7—C8	1.395 (2)
C2—H2B	0.9900	C8—C9	1.385 (2)
C3—N1	1.462 (2)	C8—H8	0.9500
C3—C4	1.507 (2)	C9—H9	0.9500
C3—H3	1.0000	N1—H1	0.85 (2)
C4—C9	1.391 (2)	O1—H1A	0.82 (2)
C2—S1—C3	93.00 (8)	C5—C4—C3	119.73 (14)
N1—C1—C2	109.83 (14)	C6—C5—C4	121.38 (15)
N1—C1—H1B	109.7	C6—C5—H5	119.3
C2—C1—H1B	109.7	C4—C5—H5	119.3
N1—C1—H1C	109.7	C5—C6—C7	119.81 (14)
C2—C1—H1C	109.7	C5—C6—H6	120.1

H1B—C1—H1C	108.2	C7—C6—H6	120.1
C1—C2—S1	105.55 (12)	O1—C7—C6	123.01 (14)
C1—C2—H2A	110.6	O1—C7—C8	117.59 (14)
S1—C2—H2A	110.6	C6—C7—C8	119.40 (14)
C1—C2—H2B	110.6	C9—C8—C7	120.25 (15)
S1—C2—H2B	110.6	C9—C8—H8	119.9
H2A—C2—H2B	108.8	C7—C8—H8	119.9
N1—C3—C4	113.46 (13)	C8—C9—C4	120.91 (14)
N1—C3—S1	106.65 (10)	C8—C9—H9	119.5
C4—C3—S1	112.52 (11)	C4—C9—H9	119.5
N1—C3—H3	108.0	C3—N1—C1	107.78 (13)
C4—C3—H3	108.0	C3—N1—H1	111.2 (14)
S1—C3—H3	108.0	C1—N1—H1	109.8 (13)
C9—C4—C5	118.25 (14)	C7—O1—H1A	108.7 (15)
C9—C4—C3	122.01 (14)		
N1—C1—C2—S1	-33.3 (2)	C5—C6—C7—O1	-179.46 (15)
C3—S1—C2—C1	10.32 (15)	C5—C6—C7—C8	0.1 (2)
C2—S1—C3—N1	14.01 (13)	O1—C7—C8—C9	179.74 (14)
C2—S1—C3—C4	139.03 (13)	C6—C7—C8—C9	0.2 (2)
N1—C3—C4—C9	71.65 (19)	C7—C8—C9—C4	-0.2 (2)
S1—C3—C4—C9	-49.55 (18)	C5—C4—C9—C8	0.0 (2)
N1—C3—C4—C5	-107.07 (17)	C3—C4—C9—C8	-178.77 (14)
S1—C3—C4—C5	131.73 (13)	C4—C3—N1—C1	-159.92 (14)
C9—C4—C5—C6	0.3 (2)	S1—C3—N1—C1	-35.47 (15)
C3—C4—C5—C6	179.05 (14)	C2—C1—N1—C3	45.7 (2)
C4—C5—C6—C7	-0.3 (2)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1...O1 ⁱ	0.85 (2)	2.28 (2)	3.073 (2)	156 (2)
O1—H1A...N1 ⁱⁱ	0.82 (2)	1.91 (2)	2.713 (2)	164 (2)

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