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3,4-Dihydro-2*H*-benzo[4,5]imidazo[2,1-*b*][1,3]-thiazin-3-ol

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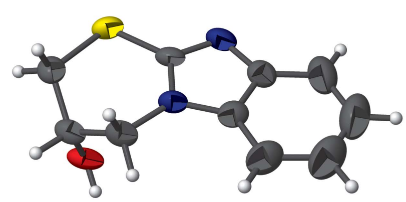
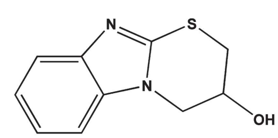
In the title compound, $C_{10}H_{10}N_2OS$, the benzimidazole ring system is almost planar (r.m.s. deviation = 0.007 Å), whereas the heterocyclic six-membered thiazine ring has an envelope conformation, with the hydroxy-substituted C atom as the flap. In the crystal, molecules are linked by O—H⋯N hydrogen bonds to form zigzag chains running along the *b*-axis direction. The chains are linked by C—H⋯O hydrogen bonds and C—H⋯π interactions, forming layers parallel to the *bc* plane.

3D view



Chemical scheme



Structure description

The benzimidazole heterocyclic system is an important pharmacophore and privileged structure in the medicinal chemistry. Its derivatives, and particularly 2-mercaptobenzimidazoles, exert various biological activities such as anticonvulsant (Anandarajagopal *et al.*, 2010; Bansal & Silakari, 2012), antiviral, anticancer (Enumula *et al.*, 2014), anti-ulcer (Gaba *et al.*, 2014), antioxidant, antibacterial (Mavrova *et al.*, 2015), antiprotozoal (Pérez-Villanueva *et al.*, 2013; Walia *et al.*, 2013) and antimicrobial (Yaseen *et al.*, 2010). The 2-mercaptobenzimidazole ring system is present in the structures of many antiparasitic, anthelmintic, antifungal, antiviral and antitumor drugs. In the present work, we have studied the action of epichlorhydrin towards 2-mercaptobenzimidazole in 2-propanol in the presence of a saturated aqueous solution of sodium bicarbonate. This led to the characterized title compound.

The molecular structure of the title compound is shown in Fig. 1. The benzimidazole ring system (N1/N2/C1–C7) is almost planar with an r.m.s. deviation of 0.007 Å. The heterocyclic six-membered thiazine ring (S1/N2/C7–C10) has an envelope conformation

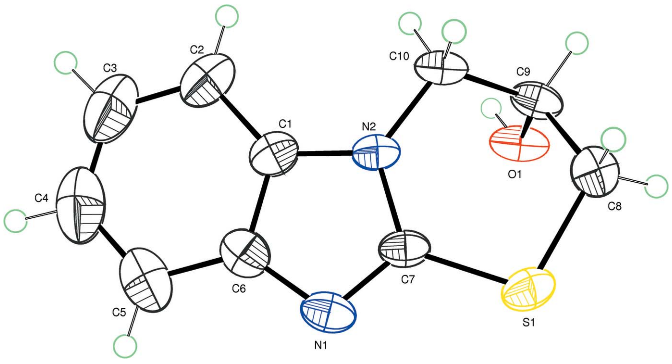


Figure 1
The molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

[puckering parameters: amplitude (Q) = 0.5154 (13) Å, θ = 126.91 (13)°, φ = 63.60 (16)°], with the hydroxy-substituted C atom, C9, as the flap. It deviates from the mean plane through the other 12 atoms of the three-fused ring system by 0.737 (1) Å.

In the crystal, structural cohesion is ensured by O1—H1···N1^{i} hydrogen bonds, which link the molecules into zigzag chains propagating along the b-axis direction. The chains are linked by C8—H8A···O1^{ii} hydrogen bonds to form layers parallel to the bc plane (Fig. 2 and Table 1). Within the layers there are also C—H···π interactions present (Table 1 and Fig. 2)

Synthesis and crystallization

A mixture of 2-mercaptobenzimidazole (1 g, 7 mmol) and epichlorhydrin (0.43 g, 4.7 mmol) in 20 ml of a saturated aqueous solution of sodium bicarbonate and 20 ml of 2-propanol, was heated under reflux for 6 h. After cooling, the product which precipitated was filtered, washed with water and then recrystallized from ethanol solution to afford the title compound as colourless block-like crystals (yield 44%; m.p. 485–487 K).

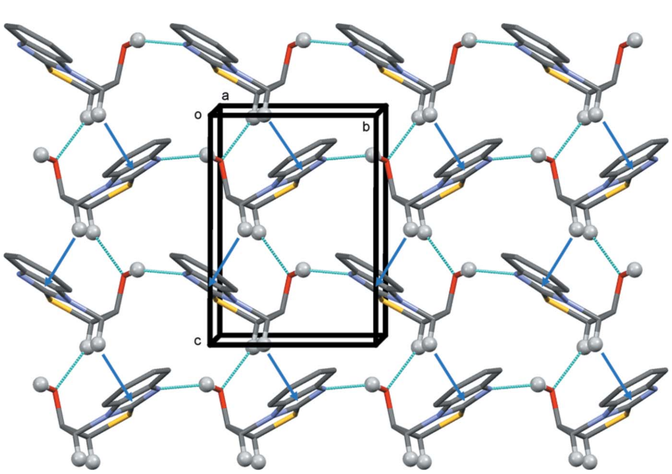


Figure 2
Crystal packing of the title compound, viewed along the a axis, showing molecules linked by hydrogen bonds (dashed lines) and C—H···π interactions (blue arrows); see Table 1 for details. For clarity, only H atoms (grey balls) H1, H8A and H10A have been included.

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

Cg1 is the centroid of the N1/N2/C1/C6/C7 ring.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1^{i}	0.82	2.02	2.7536 (14)	148
C8—H8A···O1^{ii}	0.97	2.38	3.2231 (15)	145
C10—H10A···Cg1^{ii}	0.97	2.62	3.422 (14)	138

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{10}N_2OS$
M_r	206.26
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	17.570 (3), 6.3994 (10), 8.8690 (15)
β (°)	95.641 (8)
V (Å^3)	992.4 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.29
Crystal size (mm)	0.32 × 0.26 × 0.21
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.680, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	32163, 3459, 2928
R_{int}	0.027
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.746
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.126, 1.08
No. of reflections	3459
No. of parameters	127
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.38, −0.18

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXT2014* (Sheldrick, 2015*a*), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008), *SHELXL2014* (Sheldrick, 2015*b*) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements.

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full crystallographic data

IUCrData (2017). **2**, x170429 [https://doi.org/10.1107/S2414314617004291]

3,4-Dihydro-2*H*-benzo[4,5]imidazo[2,1-*b*][1,3]thiazin-3-ol

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3,4-Dihydro-2*H*-benzo[4,5]imidazo[2,1-*b*][1,3]thiazin-3-ol

Crystal data

$C_{10}H_{10}N_2OS$
M_r = 206.26
Monoclinic, $P2_1/c$
a = 17.570 (3) Å
b = 6.3994 (10) Å
c = 8.8690 (15) Å
β = 95.641 (8)°
V = 992.4 (3) Å^3
Z = 4
F(000) = 432
D_x = 1.381 Mg m^{-3}
Melting point: 486 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3459 reflections
θ = 3.4–32.0°
μ = 0.29 mm^{-1}
T = 296 K
Block, colourless
0.32 × 0.26 × 0.21 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.680, T_{max} = 0.747
32163 measured reflections
3459 independent reflections
2928 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 32.0°, θ_{min} = 3.4°
h = −26→26
k = −9→9
l = −10→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.126
S = 1.08
3459 reflections
127 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
$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 0.1833P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.79752 (7)	0.5625 (2)	0.73771 (14)	0.0431 (3)
C2	0.87063 (9)	0.6448 (3)	0.7334 (2)	0.0649 (4)
H2	0.8791	0.7703	0.6848	0.078*
C3	0.93005 (11)	0.5291 (5)	0.8056 (3)	0.0882 (7)
H3	0.9798	0.5781	0.8050	0.106*
C4	0.91779 (13)	0.3425 (5)	0.8789 (3)	0.0896 (7)
H4	0.9594	0.2706	0.9264	0.108*
C5	0.84527 (11)	0.2607 (3)	0.8830 (2)	0.0702 (5)
H5	0.8374	0.1357	0.9327	0.084*
C6	0.78427 (8)	0.3732 (2)	0.80974 (15)	0.0456 (3)
C7	0.67468 (7)	0.49009 (16)	0.71854 (12)	0.0353 (2)
C8	0.56819 (8)	0.7590 (2)	0.57928 (13)	0.0430 (3)
H8A	0.5687	0.7336	0.4716	0.052*
H8B	0.5192	0.8212	0.5948	0.052*
C9	0.63086 (8)	0.91282 (18)	0.62946 (12)	0.0396 (2)
H9	0.6212	1.0424	0.5718	0.048*
C10	0.70875 (8)	0.82999 (19)	0.59837 (13)	0.0415 (3)
H10A	0.7094	0.8063	0.4905	0.050*
H10B	0.7476	0.9332	0.6294	0.050*
N1	0.70658 (7)	0.33100 (15)	0.79621 (12)	0.0437 (2)
N2	0.72630 (6)	0.63499 (15)	0.67993 (11)	0.0360 (2)
O1	0.62792 (7)	0.95827 (14)	0.78494 (9)	0.0477 (2)
H1	0.6619	1.0415	0.8129	0.071*
S1	0.57672 (2)	0.51091 (5)	0.67849 (4)	0.04656 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0444 (6)	0.0453 (6)	0.0404 (6)	−0.0054 (5)	0.0084 (5)	−0.0050 (5)
C2	0.0463 (7)	0.0770 (11)	0.0726 (10)	−0.0131 (7)	0.0112 (7)	−0.0040 (9)
C3	0.0460 (9)	0.120 (2)	0.0986 (17)	−0.0010 (10)	0.0054 (10)	−0.0048 (14)
C4	0.0630 (11)	0.1130 (19)	0.0899 (15)	0.0269 (12)	−0.0080 (10)	−0.0015 (14)
C5	0.0777 (11)	0.0656 (10)	0.0657 (10)	0.0219 (9)	−0.0009 (8)	0.0036 (8)
C6	0.0559 (7)	0.0396 (6)	0.0414 (6)	0.0029 (5)	0.0054 (5)	−0.0042 (5)
C7	0.0461 (6)	0.0282 (4)	0.0321 (5)	−0.0113 (4)	0.0070 (4)	−0.0048 (3)
C8	0.0490 (6)	0.0455 (6)	0.0342 (5)	0.0025 (5)	0.0023 (5)	−0.0068 (4)
C9	0.0614 (7)	0.0310 (5)	0.0274 (4)	−0.0030 (5)	0.0091 (4)	0.0008 (4)
C10	0.0547 (7)	0.0354 (5)	0.0359 (5)	−0.0100 (5)	0.0116 (5)	0.0064 (4)
N1	0.0601 (6)	0.0284 (4)	0.0428 (5)	−0.0071 (4)	0.0065 (4)	−0.0005 (4)
N2	0.0423 (5)	0.0311 (4)	0.0351 (4)	−0.0094 (3)	0.0074 (4)	0.0003 (3)
O1	0.0801 (7)	0.0337 (4)	0.0312 (4)	−0.0166 (4)	0.0157 (4)	−0.0064 (3)
S1	0.04469 (19)	0.04324 (18)	0.0517 (2)	−0.01646 (12)	0.00467 (13)	−0.00391 (12)

Geometric parameters (Å, º)

C1—N2	1.3847 (17)	C7—N2	1.3641 (13)
C1—C2	1.393 (2)	C7—S1	1.7280 (13)
C1—C6	1.3992 (19)	C8—C9	1.5108 (18)
C2—C3	1.385 (3)	C8—S1	1.8145 (14)
C2—H2	0.9300	C8—H8A	0.9700
C3—C4	1.386 (4)	C8—H8B	0.9700
C3—H3	0.9300	C9—O1	1.4152 (13)
C4—C5	1.381 (3)	C9—C10	1.5179 (19)
C4—H4	0.9300	C9—H9	0.9800
C5—C6	1.397 (2)	C10—N2	1.4601 (15)
C5—H5	0.9300	C10—H10A	0.9700
C6—N1	1.3852 (18)	C10—H10B	0.9700
C7—N1	1.3225 (15)	O1—H1	0.8200
N2—C1—C2	131.71 (14)	S1—C8—H8A	108.8
N2—C1—C6	105.92 (11)	C9—C8—H8B	108.8
C2—C1—C6	122.37 (15)	S1—C8—H8B	108.8
C3—C2—C1	116.11 (19)	H8A—C8—H8B	107.7
C3—C2—H2	121.9	O1—C9—C8	109.00 (10)
C1—C2—H2	121.9	O1—C9—C10	111.64 (11)
C2—C3—C4	122.2 (2)	C8—C9—C10	111.29 (10)
C2—C3—H3	118.9	O1—C9—H9	108.3
C4—C3—H3	118.9	C8—C9—H9	108.3
C5—C4—C3	121.69 (19)	C10—C9—H9	108.3
C5—C4—H4	119.2	N2—C10—C9	111.02 (9)
C3—C4—H4	119.2	N2—C10—H10A	109.4
C4—C5—C6	117.34 (19)	C9—C10—H10A	109.4
C4—C5—H5	121.3	N2—C10—H10B	109.4
C6—C5—H5	121.3	C9—C10—H10B	109.4
N1—C6—C5	130.08 (15)	H10A—C10—H10B	108.0
N1—C6—C1	109.63 (11)	C7—N1—C6	104.98 (10)
C5—C6—C1	120.27 (15)	C7—N2—C1	106.15 (10)
N1—C7—N2	113.32 (11)	C7—N2—C10	126.31 (11)
N1—C7—S1	121.93 (9)	C1—N2—C10	127.51 (10)
N2—C7—S1	124.71 (9)	C9—O1—H1	109.5
C9—C8—S1	113.84 (9)	C7—S1—C8	101.55 (6)
C9—C8—H8A	108.8		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the N1/N2/C1/C6/C7 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1^{i}	0.82	2.02	2.7536 (14)	148
C8—H8*A*···O1^{ii}	0.97	2.38	3.2231 (15)	145
C10—H10*A*···*Cg*1^{ii}	0.97	2.62	3.422 (14)	138

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