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Benzo[4,5]imidazo[2,1-*b*]thiazole-2-carbaldehyde

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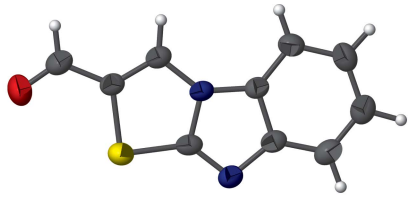
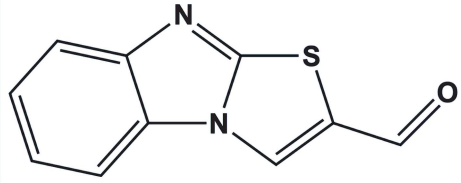
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{10}H_6N_2OS$, is planar, with an r.m.s. deviation of 0.021 Å for the non-H atoms. In the crystal, molecules are linked *via* a pair of C—H···O hydrogen bonds, forming inversion dimers with an $R_2^2(6)$ ring motif. The molecules stack up the *c* axis and are linked by offset π–π interactions [shortest inter-centroid distance = 3.647 (2) Å], forming undulating layers parallel to (100).

3D view



Chemical scheme



Structure description

Substituted and unsubstituted benzimidazole derivatives occupy an important position among medications due to their vast range of biological activities, such as immunomodulator (Fenichel *et al.*, 1980; Dillman *et al.*, 1992) anti-ulcer, anticancer (Abdel-Aziz *et al.*, 2010), antifungal (Pattanaik *et al.*, 1998), antibacterial (Oh *et al.*, 1995), antidiabetic (El-Shorbagi *et al.*, 2001) and fungicidal (Chaudhary *et al.*, 1970). Benzimidazole derivatives are also used as building blocks for the synthesis of nonpeptide antagonists of angiotensin II receptor (Abdel-Aziz *et al.*, 2010; Chaudhary *et al.*, 1970). In view of the current interest in designing new benzimidazole derivatives, we have synthesized benzo[4,5]imidazo[2,1-*b*]thiazole-2-carbaldehyde and report herein its crystal structure.

The title compound (Fig. 1) is planar, with an r.m.s. deviation of 0.021 Å for all the non-H atoms [maximum deviation = 0.032 (4) Å for atom C3]. The bond lengths and angles are close to those observed for the similar compound 1-(6-bromo-3-methyl-1,3-thiazolo[3,2-*a*]benzimidazol-2-yl)ethanone (Abdel-Aziz *et al.*, 2011).

In the crystal of the title compound, molecules are linked by a pair of C—H···O hydrogen bonds, forming inversion dimers with an $R_2^2(6)$ ring motif (Table 1 and Fig. 2). The molecules stack up the *c* axis and are linked by slipped parallel π–π interactions, involving inversion-related molecules, forming undulating layers parallel to the *bc* plane (Fig. 2). The shortest interaction is $Cg1\cdots Cg2^{ii}$ of 3.647 (2) Å, with an interplanar

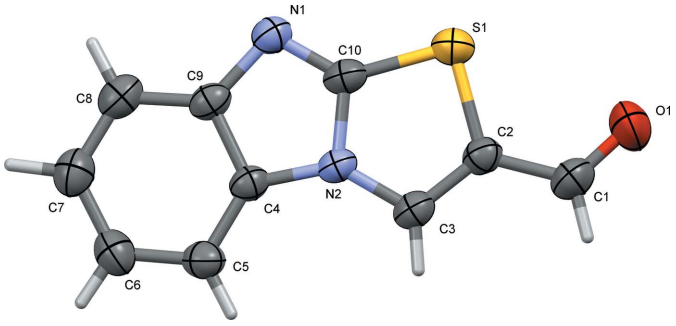


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O1^{i}	0.93	2.55	3.248 (4)	132

Symmetry code: (i) $-x+1, -y+1, -z+1$.

distance of 3.417 (1) Å and a slippage of 1.239 Å [*Cg*1 and *Cg*2 are the centroids of the S1/N2/C2/C3/C10 and N1/N2/C4/C9/C10 rings, respectively; symmetry code: (ii) $-x, -y, -z$].

Synthesis and crystallization

To synthesize the title compound, a number of attempts were made with different bases, like K_2CO_3, NaOH, KOH, triethylamine and ACONa, for the nucleophilic reaction in different solvents, like EtOH, MeOH, CH_3CN and dimethylformamide (DMF). Success was achieved with the following procedure. To a stirred solution of 2-mercaptobenzimidazole (250 mg, 0.0016 mol) in dry acetone, an acetone solution of 2-bromomalonaldehyde (252 mg, 0.0016 mol) was added dropwise with stirring over a period of 60 min. After evaporating the solvent, ice-cold water was added and the mixture neutralized with ammonium hydroxide. The reaction was monitored by thin-layer-chromatography (TLC). The pale-coloured solid which was produced was collected by filtration, washed several times with ice-cold water and petroleum ether, and finally dried *in vacuo*. Colourless block-shaped crystals were obtained by slow evaporation of a solution in water/DMF (1:9 *v*/*v*).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_6N_2OS$
M_r	202.23
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	298
a, b, c (Å)	5.6514 (16), 21.220 (7), 7.381 (2)
β (°)	96.473 (17)
V (Å^3)	879.5 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.33
Crystal size (mm)	0.24 × 0.23 × 0.22
Data collection	
Diffractometer	Bruker SMART APEXII area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.584, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15536, 2357, 935
R_{int}	0.129
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.705
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.058, 0.150, 0.91
No. of reflections	2357
No. of parameters	131
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.35

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008).

Refinement

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

Acknowledgements

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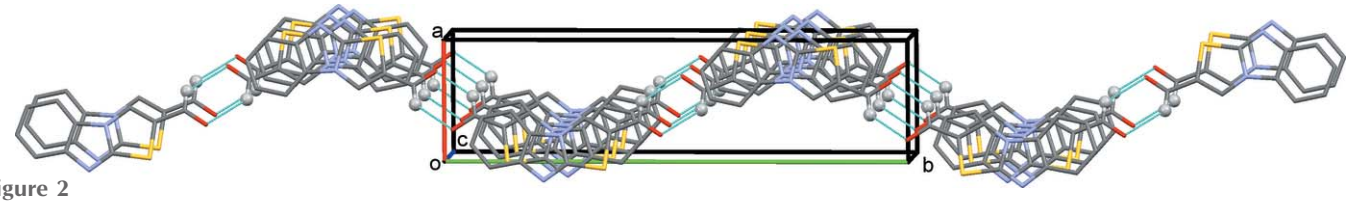

Figure 2
A view along the *c* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1) and, for clarity, only the H atom (grey ball) involved in this interaction has been included.

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full crystallographic data

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Crystal data

$C_{10}H_6N_2OS$
M_r = 202.23
Monoclinic, $P2_1/c$
a = 5.6514 (16) Å
b = 21.220 (7) Å
c = 7.381 (2) Å
β = 96.473 (17)°
V = 879.5 (5) Å^3
Z = 4
$F(000)$ = 416
D_x = 1.527 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 563 reflections
θ = 5–25.0°
μ = 0.33 mm^{-1}
T = 298 K
Block, colourless
0.24 × 0.23 × 0.22 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: microfocus sealed X-ray tube, Incoatec Iμs
Mirror optics monochromator
Detector resolution: 7.9 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
T_{min} = 0.584, T_{max} = 0.746
15536 measured reflections
2357 independent reflections
935 reflections with $I > 2\sigma(I)$
R_{int} = 0.129
θ_{max} = 30.1°, θ_{min} = 1.9°
h = −6→7
k = −29→29
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.150
S = 0.91
2357 reflections
131 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 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}
S1	0.00346 (15)	0.35868 (4)	0.24538 (14)	0.0456 (3)
O1	0.2732 (5)	0.47879 (12)	0.3388 (4)	0.0712 (9)
N1	−0.0906 (5)	0.22884 (13)	0.2066 (4)	0.0405 (7)
N2	0.2655 (4)	0.26299 (12)	0.3445 (4)	0.0344 (7)
C1	0.3851 (6)	0.43192 (18)	0.3864 (5)	0.0524 (10)
H1	0.5378	0.4367	0.4465	0.063*
C2	0.2933 (6)	0.36898 (16)	0.3546 (5)	0.0386 (8)
C3	0.4060 (6)	0.31408 (16)	0.3987 (5)	0.0394 (9)
H3	0.5596	0.3111	0.4583	0.047*
C4	0.2798 (5)	0.19764 (15)	0.3477 (5)	0.0354 (8)
C5	0.4609 (6)	0.15669 (16)	0.4114 (5)	0.0437 (9)
H5	0.6083	0.1711	0.4640	0.052*
C6	0.4101 (6)	0.09323 (17)	0.3922 (5)	0.0513 (10)
H6	0.5260	0.0639	0.4339	0.062*
C7	0.1898 (7)	0.0720 (2)	0.3120 (5)	0.0519 (10)
H7	0.147 (6)	0.0268 (17)	0.294 (5)	0.066 (12)*
C8	0.0123 (6)	0.11321 (17)	0.2454 (5)	0.0468 (9)
H8	−0.1335	0.0984	0.1909	0.056*
C9	0.0572 (5)	0.17760 (16)	0.2619 (4)	0.0370 (8)
C10	0.0414 (5)	0.27784 (16)	0.2589 (5)	0.0402 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0405 (5)	0.0460 (6)	0.0480 (6)	0.0017 (4)	−0.0050 (4)	0.0016 (5)
O1	0.080 (2)	0.0409 (17)	0.087 (2)	0.0059 (14)	−0.0140 (16)	0.0041 (15)
N1	0.0381 (15)	0.0419 (19)	0.0404 (18)	−0.0038 (14)	−0.0014 (13)	−0.0023 (14)
N2	0.0296 (14)	0.0401 (18)	0.0326 (17)	−0.0034 (13)	−0.0003 (12)	−0.0016 (14)
C1	0.049 (2)	0.050 (3)	0.055 (3)	−0.007 (2)	−0.0069 (19)	−0.007 (2)
C2	0.0372 (18)	0.042 (2)	0.035 (2)	−0.0044 (16)	−0.0012 (15)	0.0009 (17)
C3	0.0356 (18)	0.045 (2)	0.037 (2)	−0.0053 (17)	0.0011 (15)	−0.0020 (17)
C4	0.0340 (18)	0.040 (2)	0.032 (2)	−0.0024 (16)	0.0007 (14)	−0.0039 (17)
C5	0.0357 (18)	0.049 (2)	0.046 (2)	0.0009 (16)	0.0013 (16)	0.0043 (18)
C6	0.055 (2)	0.042 (2)	0.057 (3)	0.0080 (18)	0.0087 (19)	0.0030 (19)
C7	0.057 (3)	0.044 (3)	0.055 (3)	−0.005 (2)	0.009 (2)	−0.006 (2)
C8	0.044 (2)	0.053 (2)	0.044 (2)	−0.011 (2)	0.0066 (16)	−0.006 (2)
C9	0.0359 (19)	0.046 (2)	0.029 (2)	−0.0055 (16)	0.0025 (16)	−0.0020 (17)
C10	0.0321 (19)	0.050 (2)	0.037 (2)	−0.0026 (16)	−0.0013 (16)	0.0002 (18)

Geometric parameters (Å, º)

S1—C10	1.730 (3)	C3—H3	0.9300
S1—C2	1.756 (3)	C4—C5	1.384 (4)
O1—C1	1.210 (4)	C4—C9	1.409 (4)
N1—C10	1.312 (4)	C5—C6	1.381 (5)

N1—C9	1.403 (4)	C5—H5	0.9300
N2—C3	1.376 (4)	C6—C7	1.392 (5)
N2—C10	1.386 (4)	C6—H6	0.9300
N2—C4	1.389 (4)	C7—C8	1.379 (5)
C1—C2	1.442 (5)	C7—H7	0.99 (3)
C1—H1	0.9300	C8—C9	1.393 (4)
C2—C3	1.350 (4)	C8—H8	0.9300

C10—S1—C2	89.64 (16)	C6—C5—H5	121.9
C10—N1—C9	103.2 (3)	C4—C5—H5	121.9
C3—N2—C10	114.9 (3)	C5—C6—C7	121.7 (3)
C3—N2—C4	138.6 (3)	C5—C6—H6	119.2
C10—N2—C4	106.5 (2)	C7—C6—H6	119.2
O1—C1—C2	123.2 (3)	C8—C7—C6	121.7 (4)
O1—C1—H1	118.4	C8—C7—H7	114 (2)
C2—C1—H1	118.4	C6—C7—H7	124 (2)
C3—C2—C1	127.5 (3)	C7—C8—C9	118.2 (3)
C3—C2—S1	113.2 (3)	C7—C8—H8	120.9
C1—C2—S1	119.3 (3)	C9—C8—H8	120.9
C2—C3—N2	111.6 (3)	C8—C9—N1	129.6 (3)
C2—C3—H3	124.2	C8—C9—C4	118.7 (3)
N2—C3—H3	124.2	N1—C9—C4	111.6 (3)
C5—C4—N2	132.2 (3)	N1—C10—N2	114.4 (3)
C5—C4—C9	123.5 (3)	N1—C10—S1	134.9 (3)
N2—C4—C9	104.3 (3)	N2—C10—S1	110.7 (2)
C6—C5—C4	116.1 (3)		

O1—C1—C2—C3	178.4 (4)	C7—C8—C9—N1	−179.9 (3)
O1—C1—C2—S1	−1.2 (5)	C7—C8—C9—C4	−0.8 (5)
C10—S1—C2—C3	−0.7 (3)	C10—N1—C9—C8	179.0 (3)
C10—S1—C2—C1	179.0 (3)	C10—N1—C9—C4	−0.1 (3)
C1—C2—C3—N2	−178.8 (3)	C5—C4—C9—C8	2.4 (5)
S1—C2—C3—N2	0.8 (4)	N2—C4—C9—C8	−179.1 (3)
C10—N2—C3—C2	−0.5 (4)	C5—C4—C9—N1	−178.4 (3)
C4—N2—C3—C2	178.8 (3)	N2—C4—C9—N1	0.2 (3)
C3—N2—C4—C5	−1.1 (6)	C9—N1—C10—N2	0.1 (4)
C10—N2—C4—C5	178.3 (3)	C9—N1—C10—S1	179.6 (3)
C3—N2—C4—C9	−179.4 (3)	C3—N2—C10—N1	179.5 (3)
C10—N2—C4—C9	−0.1 (3)	C4—N2—C10—N1	0.0 (4)
N2—C4—C5—C6	179.7 (3)	C3—N2—C10—S1	−0.1 (3)
C9—C4—C5—C6	−2.1 (5)	C4—N2—C10—S1	−179.6 (2)
C4—C5—C6—C7	0.5 (5)	C2—S1—C10—N1	−179.1 (4)
C5—C6—C7—C8	0.9 (5)	C2—S1—C10—N2	0.4 (2)
C6—C7—C8—C9	−0.7 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1^{i}	0.93	2.55	3.248 (4)	132

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.