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(*E*)-4-(Benzo[*d*]thiazol-2-yl)-*N*-(pyridin-3-ylmethylidene)aniline hemihydrate

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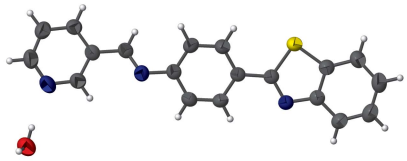
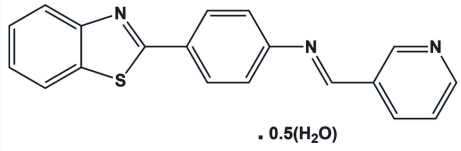
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{19}H_{13}N_3S\cdot0.5H_2O$, is a benzothiazole derivative that crystallized as a hemihydrate, the water O atom being situated on a twofold rotation axis. The dihedral angles between the central benzene ring and the benzothiazole (r.m.s. deviation = 0.012 Å) and pyridine rings are 3.57 (6) and 10.12 (8)°, respectively, indicating that the molecule is nearly planar. The conformation about the N=C bond is *E*. In the crystal, molecules are linked by O_{water}—H···$N_{pyridine}$ hydrogen bonds, forming dimers, which in turn are linked by C—H···O hydrogen bonds into layers parallel to the *ab* plane. The layers are linked by offset π–π interactions, forming a three-dimensional network [shortest intercentroid distance = 3.721 (2) Å].

3D view



Chemical scheme



Structure description

Benzothiazole derivatives are considered to be important because of their wide range of biological activities (Bakthadoss & Selvakumar, 2016), and also because of their high electron affinity and good planarity making them appropriate building blocks in the construction of optical materials (Liu *et al.*, 2013). Herein, we report the synthesis and crystal structure of the title benzothiazole derivative.

The molecular structure is illustrated in Fig. 1. The molecule is relatively planar with dihedral angles between the central benzene ring (C8–C13) and the benzothiazole (r.m.s. deviation = 0.012 Å) and pyridine rings being 3.57 (6) and 10.12 (8)°, respectively.

In the crystal, molecules are linked by O_{water}—H···$N_{pyridine}$ hydrogen bonds, forming dimers, which in turn are linked by C—H···O_{water} hydrogen bonds into layers parallel to the *ab* plane (Table 1 and Fig. 2). The layers are linked by offset π–π interactions forming a three-dimensional network [shortest intercentroid distance $Cg2\cdots Cg3^{i}$ = 3.721 (2) Å;

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N3	0.88 (2)	2.03 (2)	2.894 (2)	170 (2)
C2—H2···O1^{i}	0.93	2.51	3.245 (3)	136

Symmetry code: (i) $x-\frac{1}{2}, y-\frac{1}{2}, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{13}N_3S\cdot0.5H_2O$
M_r	324.39
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	296
a, b, c (Å)	34.026 (5), 10.447 (5), 8.967 (5)
β (°)	97.601 (5)
V (Å^3)	3159 (2)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.21
Crystal size (mm)	0.23 × 0.22 × 0.21
Data collection	
Diffractometer	Bruker SMART CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2002)
T_{min}, T_{max}	0.953, 0.957
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11032, 2794, 2444
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.131, 1.09
No. of reflections	2794
No. of parameters	217
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.23

Computer programs: *SMART* and *SAINT* (Bruker, 2002), *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

*Cg*2 and *Cg*3 are the centroids of rings C1–C6 and N3/C15–C19, respectively; symmetry code: (i) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z$].

Synthesis and crystallization

3-Pyridinecarboxaldehyde (0.29 g, 2.7 mmol) was dissolved in 20 ml of ethanol and added dropwise into 20 ml of an ethanol solution of 4-(benzothiazol-2-yl)aniline (0.61 g, 2.7 mmol). The mixture was stirred at room temperature, and a yellow solid precipitate gradually appeared after 30 min. On completion of the reaction, monitored by TLC, the solid was filtered and recrystallized from ethanol solution to produce block-like yellow crystals (yield 78.04%, 0.66 g).

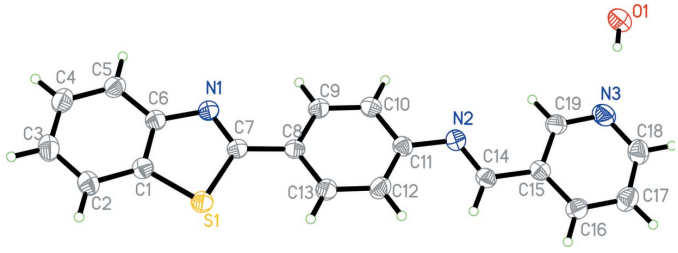


Figure 1
The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

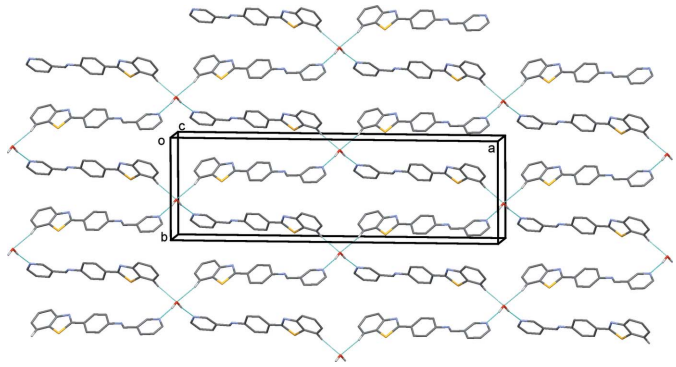


Figure 2
A view along the *c* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1) and for clarity, only H atoms H1 and H2 have been included.

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161608 [https://doi.org/10.1107/S2414314616016084]

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Crystal data

$C_{19}H_{13}N_3S·0.5H_2O$
M_r = 324.39
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 34.026 (5) Å
b = 10.447 (5) Å
c = 8.967 (5) Å
β = 97.601 (5)°
V = 3159 (2) Å^3
Z = 8
F(000) = 1352
D_x = 1.364 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71069 Å
Cell parameters from 5584 reflections
θ = 2.4–26.7°
μ = 0.21 mm^{-1}
T = 296 K
Block, yellow
0.23 × 0.22 × 0.21 mm

Data collection

Bruker SMART CCD area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and *ω* scans
Absorption correction: multi-scan (SADABS; Bruker, 2002)
T_{min} = 0.953, T_{max} = 0.957
11032 measured reflections
2794 independent reflections
2444 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.0°, θ_{min} = 1.2°
h = −37→40
k = −12→12
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.131
S = 1.09
2794 reflections
217 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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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.

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.640666 (11)	0.04206 (4)	0.17319 (4)	0.0537 (2)
N1	0.66886 (3)	0.21158 (11)	0.37023 (13)	0.0455 (3)
O1	1.0000	0.35524 (18)	0.2500	0.0718 (5)
C6	0.63000 (4)	0.18945 (13)	0.39216 (15)	0.0449 (3)
N2	0.83432 (4)	0.14991 (12)	0.10104 (14)	0.0518 (3)
C1	0.60952 (4)	0.09943 (13)	0.29545 (16)	0.0477 (4)
C7	0.67836 (4)	0.14078 (12)	0.26047 (15)	0.0424 (3)
C11	0.79462 (4)	0.14324 (13)	0.13047 (16)	0.0459 (4)
C16	0.89930 (5)	0.06086 (14)	−0.16991 (18)	0.0546 (4)
H16	0.8817	0.0151	−0.2382	0.066*
C15	0.88592 (4)	0.12279 (13)	−0.05054 (15)	0.0455 (4)
C14	0.84443 (4)	0.11634 (14)	−0.02344 (16)	0.0475 (4)
H14	0.8253	0.0870	−0.0993	0.057*
C8	0.71788 (4)	0.14141 (12)	0.21252 (15)	0.0420 (3)
C5	0.61062 (5)	0.24786 (15)	0.50285 (18)	0.0539 (4)
H5	0.6237	0.3079	0.5680	0.065*
C12	0.76579 (5)	0.06122 (15)	0.0601 (2)	0.0570 (4)
H12	0.7720	0.0063	−0.0150	0.068*
C13	0.72815 (5)	0.06029 (15)	0.10019 (18)	0.0546 (4)
H13	0.7092	0.0048	0.0517	0.066*
C10	0.78445 (5)	0.22285 (16)	0.24345 (18)	0.0564 (4)
H10	0.8035	0.2773	0.2932	0.068*
N3	0.95197 (4)	0.19941 (15)	0.02959 (16)	0.0677 (4)
C4	0.57205 (5)	0.21506 (17)	0.51363 (19)	0.0616 (4)
H4	0.5589	0.2539	0.5862	0.074*
C2	0.57032 (5)	0.06641 (16)	0.3070 (2)	0.0598 (4)
H2	0.5568	0.0070	0.2420	0.072*
C9	0.74685 (4)	0.22258 (15)	0.28291 (18)	0.0542 (4)
H9	0.7407	0.2776	0.3580	0.065*
C19	0.91367 (5)	0.19251 (15)	0.04519 (18)	0.0562 (4)
H19	0.9049	0.2368	0.1245	0.067*
C17	0.93888 (5)	0.06715 (17)	−0.1874 (2)	0.0655 (5)
H17	0.9484	0.0255	−0.2670	0.079*
C3	0.55236 (5)	0.12481 (17)	0.4178 (2)	0.0653 (5)
H3	0.5264	0.1032	0.4288	0.078*
C18	0.96381 (5)	0.13569 (17)	−0.0857 (2)	0.0670 (5)
H18	0.9906	0.1382	−0.0972	0.080*
H1	0.9871 (7)	0.300 (2)	0.188 (3)	0.107 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0521 (3)	0.0531 (3)	0.0567 (3)	−0.00853 (15)	0.0102 (2)	−0.01417 (16)
N1	0.0479 (7)	0.0446 (6)	0.0440 (7)	−0.0002 (5)	0.0058 (5)	−0.0019 (5)
O1	0.0611 (11)	0.0734 (12)	0.0771 (13)	0.000	−0.0049 (9)	0.000
C6	0.0478 (8)	0.0437 (7)	0.0428 (8)	0.0019 (6)	0.0043 (6)	0.0062 (6)
N2	0.0454 (7)	0.0584 (8)	0.0514 (8)	0.0014 (5)	0.0060 (6)	−0.0009 (6)
C1	0.0515 (9)	0.0448 (7)	0.0472 (8)	−0.0021 (6)	0.0078 (6)	0.0016 (6)
C7	0.0482 (8)	0.0392 (7)	0.0396 (7)	0.0012 (5)	0.0045 (6)	0.0029 (6)
C11	0.0445 (8)	0.0477 (8)	0.0446 (8)	0.0017 (6)	0.0029 (6)	0.0037 (6)
C16	0.0568 (10)	0.0552 (8)	0.0528 (9)	−0.0105 (7)	0.0105 (7)	−0.0033 (7)
C15	0.0474 (8)	0.0446 (7)	0.0438 (8)	−0.0009 (6)	0.0037 (6)	0.0086 (6)
C14	0.0451 (8)	0.0510 (8)	0.0450 (8)	−0.0007 (6)	0.0005 (6)	0.0045 (6)
C8	0.0459 (8)	0.0408 (7)	0.0385 (7)	0.0007 (5)	0.0027 (6)	0.0037 (5)
C5	0.0565 (9)	0.0563 (9)	0.0496 (9)	0.0048 (7)	0.0096 (7)	−0.0026 (7)
C12	0.0573 (10)	0.0568 (8)	0.0595 (9)	−0.0051 (7)	0.0170 (7)	−0.0154 (7)
C13	0.0513 (9)	0.0559 (8)	0.0576 (9)	−0.0098 (6)	0.0108 (7)	−0.0135 (7)
C10	0.0497 (9)	0.0625 (9)	0.0564 (9)	−0.0081 (7)	0.0043 (7)	−0.0131 (7)
N3	0.0520 (8)	0.0852 (10)	0.0645 (9)	−0.0153 (7)	0.0024 (7)	−0.0035 (8)
C4	0.0596 (10)	0.0702 (10)	0.0577 (10)	0.0102 (8)	0.0175 (8)	0.0050 (8)
C2	0.0514 (9)	0.0627 (9)	0.0658 (10)	−0.0098 (7)	0.0101 (8)	−0.0023 (8)
C9	0.0539 (9)	0.0579 (9)	0.0511 (9)	−0.0044 (7)	0.0084 (7)	−0.0143 (7)
C19	0.0537 (9)	0.0663 (9)	0.0481 (9)	−0.0056 (7)	0.0046 (7)	−0.0020 (7)
C17	0.0654 (11)	0.0642 (10)	0.0715 (12)	−0.0072 (8)	0.0259 (9)	−0.0082 (8)
C3	0.0517 (9)	0.0744 (11)	0.0721 (11)	−0.0022 (8)	0.0164 (8)	0.0085 (9)
C18	0.0498 (9)	0.0773 (11)	0.0761 (12)	−0.0068 (8)	0.0163 (9)	0.0017 (9)

Geometric parameters (Å, º)

S1—C1	1.7297 (16)	C8—C13	1.396 (2)
S1—C7	1.7482 (14)	C5—C4	1.372 (2)
N1—C7	1.3056 (18)	C5—H5	0.9300
N1—C6	1.3818 (19)	C12—C13	1.375 (2)
O1—H1	0.87 (2)	C12—H12	0.9300
C6—C1	1.401 (2)	C13—H13	0.9300
C6—C5	1.402 (2)	C10—C9	1.372 (2)
N2—C14	1.261 (2)	C10—H10	0.9300
N2—C11	1.4118 (18)	N3—C19	1.331 (2)
C1—C2	1.394 (2)	N3—C18	1.336 (2)
C7—C8	1.465 (2)	C4—C3	1.387 (3)
C11—C10	1.389 (2)	C4—H4	0.9300
C11—C12	1.389 (2)	C2—C3	1.377 (2)
C16—C17	1.378 (2)	C2—H2	0.9300
C16—C15	1.378 (2)	C9—H9	0.9300
C16—H16	0.9300	C19—H19	0.9300
C15—C19	1.394 (2)	C17—C18	1.364 (2)
C15—C14	1.465 (2)	C17—H17	0.9300

C14—H14	0.9300	C3—H3	0.9300
C8—C9	1.387 (2)	C18—H18	0.9300

C1—S1—C7	89.23 (7)	C13—C12—H12	119.6
C7—N1—C6	110.47 (12)	C11—C12—H12	119.6
N1—C6—C1	115.55 (12)	C12—C13—C8	120.99 (14)
N1—C6—C5	125.20 (13)	C12—C13—H13	119.5
C1—C6—C5	119.25 (13)	C8—C13—H13	119.5
C14—N2—C11	122.13 (12)	C9—C10—C11	121.16 (14)
C2—C1—C6	121.50 (14)	C9—C10—H10	119.4
C2—C1—S1	129.30 (13)	C11—C10—H10	119.4
C6—C1—S1	109.19 (11)	C19—N3—C18	117.01 (15)
N1—C7—C8	123.18 (12)	C5—C4—C3	120.96 (15)
N1—C7—S1	115.56 (11)	C5—C4—H4	119.5
C8—C7—S1	121.26 (10)	C3—C4—H4	119.5
C10—C11—C12	118.12 (14)	C3—C2—C1	117.67 (16)
C10—C11—N2	116.32 (13)	C3—C2—H2	121.2
C12—C11—N2	125.52 (13)	C1—C2—H2	121.2
C17—C16—C15	119.62 (15)	C10—C9—C8	120.97 (14)
C17—C16—H16	120.2	C10—C9—H9	119.5
C15—C16—H16	120.2	C8—C9—H9	119.5
C16—C15—C19	117.20 (14)	N3—C19—C15	123.79 (15)
C16—C15—C14	122.10 (13)	N3—C19—H19	118.1
C19—C15—C14	120.70 (13)	C15—C19—H19	118.1
N2—C14—C15	121.06 (13)	C18—C17—C16	118.65 (16)
N2—C14—H14	119.5	C18—C17—H17	120.7
C15—C14—H14	119.5	C16—C17—H17	120.7
C9—C8—C13	117.97 (14)	C2—C3—C4	121.61 (15)
C9—C8—C7	119.58 (13)	C2—C3—H3	119.2
C13—C8—C7	122.42 (12)	C4—C3—H3	119.2
C4—C5—C6	119.01 (15)	N3—C18—C17	123.70 (16)
C4—C5—H5	120.5	N3—C18—H18	118.2
C6—C5—H5	120.5	C17—C18—H18	118.2
C13—C12—C11	120.77 (14)		

C7—N1—C6—C1	−0.22 (17)	N1—C6—C5—C4	−178.82 (13)
C7—N1—C6—C5	178.72 (13)	C1—C6—C5—C4	0.1 (2)
N1—C6—C1—C2	178.88 (13)	C10—C11—C12—C13	0.8 (2)
C5—C6—C1—C2	−0.1 (2)	N2—C11—C12—C13	178.31 (14)
N1—C6—C1—S1	−0.18 (16)	C11—C12—C13—C8	−0.1 (3)
C5—C6—C1—S1	−179.19 (11)	C9—C8—C13—C12	−0.3 (2)
C7—S1—C1—C2	−178.59 (16)	C7—C8—C13—C12	−178.52 (14)
C7—S1—C1—C6	0.39 (11)	C12—C11—C10—C9	−1.2 (2)
C6—N1—C7—C8	−178.89 (11)	N2—C11—C10—C9	−178.94 (13)
C6—N1—C7—S1	0.54 (15)	C6—C5—C4—C3	0.5 (2)
C1—S1—C7—N1	−0.56 (11)	C6—C1—C2—C3	−0.5 (2)
C1—S1—C7—C8	178.88 (11)	S1—C1—C2—C3	178.41 (12)
C14—N2—C11—C10	−157.22 (14)	C11—C10—C9—C8	0.9 (2)

C14—N2—C11—C12	25.2 (2)	C13—C8—C9—C10	−0.1 (2)
C17—C16—C15—C19	1.7 (2)	C7—C8—C9—C10	178.17 (14)
C17—C16—C15—C14	−178.10 (14)	C18—N3—C19—C15	0.4 (3)
C11—N2—C14—C15	−179.28 (12)	C16—C15—C19—N3	−1.8 (2)
C16—C15—C14—N2	164.97 (15)	C14—C15—C19—N3	178.03 (14)
C19—C15—C14—N2	−14.8 (2)	C15—C16—C17—C18	−0.4 (3)
N1—C7—C8—C9	−1.6 (2)	C1—C2—C3—C4	1.1 (3)
S1—C7—C8—C9	178.97 (11)	C5—C4—C3—C2	−1.2 (3)
N1—C7—C8—C13	176.59 (13)	C19—N3—C18—C17	1.1 (3)
S1—C7—C8—C13	−2.81 (19)	C16—C17—C18—N3	−1.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3	0.88 (2)	2.03 (2)	2.894 (2)	170 (2)
C2—H2···O1^{i}	0.93	2.51	3.245 (3)	136

Symmetry code: (i) $x-1/2, y-1/2, z$.