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3-Fluoro-12*H*-benzimidazo[2,1-*b*][1,3]-benzothiazin-12-one

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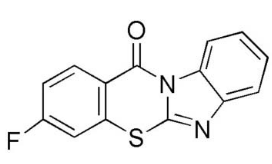
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.106; data-to-parameter ratio = 11.6.

In the title compound, $C_{14}H_7FN_2OS$, prepared by the reaction of 2-bromo-4-fluorobenzoyl choride with 2-mercaptobenzimidazole, the four-membered fused-ring system is essentially planar [maximum deviation from the mean plane = 0.035 (2) Å]. The crystal packing is stabilized by weak intermolecular π–π [minimum ring centroid–centroid separation = 3.509 (7) Å], weak C—F$\cdots\pi$ [F$\cdots$centroid = 3.4464 (17) Å, C—F$\cdots$centroid = 97.72 (11)°] and C—O$\cdots\pi$ [O$\cdots$centroid = 3.5230 (16) and 3.7296 (17) Å, C—O$\cdots$centroid = 86.40 (10) and 86.25 (10)°] interactions and weak intermolecular C—H$\cdots$N hydrogen bonds.

Related literature

For general background to spiranes, see: Dawood & Abdel-Wahab (2010); Dolbier *et al.* (1994); Mavrova *et al.* (2010); Sekar *et al.* (2011).



Experimental

Crystal data

$C_{14}H_7FN_2OS$
M_r = 270.28
Monoclinic, $P2_1/c$
a = 9.5027 (12) Å
b = 7.0759 (9) Å
c = 16.931 (2) Å
β = 94.375 (3)°
V = 1135.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 293 K
0.20 × 0.18 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.944, T_{max} = 0.958
5987 measured reflections
1989 independent reflections
1772 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.106
S = 1.00
1989 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C14—H14$\cdots$N1^{i}	0.93	2.58	3.359 (2)	141

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2160).

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supporting information

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3-Fluoro-12*H*-benzimidazo[2,1-*b*][1,3]benzothiazin-12-one

Zhiming Wang, Bin Yu, Xiuqin Zhang, Yuan Cui and Xiaoqiang Sun

S1. Comment

The chemistry of 2-mercaptoimidazole or 2-mercaptobenzimidazole has attracted much attention of many synthetic chemists owing to the occurrence of these ring systems in various biologically important compounds (Dawood & Abdel-Wahab, 2010; Mavrova *et al.*, 2010). In the past decades, most of these investigations were carried out with imidazole derivatives (Dolbier *et al.*, 1994; Sekar *et al.*, 2011). We herein present the structure of the title compound $C_{14}H_7FN_2OS$, prepared from the reaction of 2-bromo-4-fluorobenzoyl choride with 2-mercaptobenzimidazole.

In the crystal structure, the title compound adopts an essentially planar conformation (Fig. 1), with the maximum atom deviation from the least-squares plane to the four-membered fused-ring system = 0.035 (2) Å. The dihedral angles between the benzimidazole ring (N1–C7) and thiazine ring (S1–C10) = 0.74 (8)°, the benzene ring (C9–C14) and thiazine ring (S1–C10) = 1.00 (4)° and the benzimidazole ring (N1–C7) and benzene ring (C9–C14) = 0.03 (8)°.

The crystal packing is stabilized by weak interactions: (1) intermolecular *π*–*π* interactions: (*a*) imidazole ring N1–C7 (ring 1) and benzene ring C1–C6 (ring 2) of the benzimazole moiety [ring centroid separation = 3.673 (8) Å, symmetry code (i)-*x*+1,-*y*+2, -*z*]; (*b*) between thiazine ring S1–C10 (ring 3) and S1–C10^i = 3.856 (5) Å; (*c*) between ring 3···and ring 2^i = 3.509 (7) Å; (2) C—O···*π* interactions [C(13)—O(1)···*Cg2*, C(13)—O(1)···*Cg3*] and C—F···*π* interactions [C(2)—F(1)···*Cg4*]; (3) intermolecular C—H···N hydrogen bonds [C(6)—H(6)···N(2)].

S2. Experimental

An oven-dried Schlenk tube was charged with a magnetic stirring bar, CuI (0.05 mmol), 1,10-phenanthroline (0.10 mmol), Cs_2CO_3 (0.50 mmol), and 2-mercaptobenzimidazole. The Schlenk tube was capped, and then evacuated and backfilled with N_2 (3 times), then under a positive pressure of N_2, a solution of 2-bromo-4-fluorobenzoyl choride (0.75 mmol) in touene (2 ml, freshly distilled from sodium) was added dropwise *via* syringe, and the mixture was pre-stirred for 1 h at room temperature. The reaction mixture was then stirred at 100 °C. After the reaction was completed, the mixture was cooled to room temperature, passed through Celite and rinsed with 30 ml of CH_2Cl_2. The combined filtrate was concentrated and purified by flash chromatography to give a white solid (93% yield). Single crystals of the title compound suitable for X-ray diffraction were obtained by evaporation of a petroleum ether–chloroform solution.

S3. Refinement

All the H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å, with U_{iso}(H) = 1.2 U_{eq}(C)

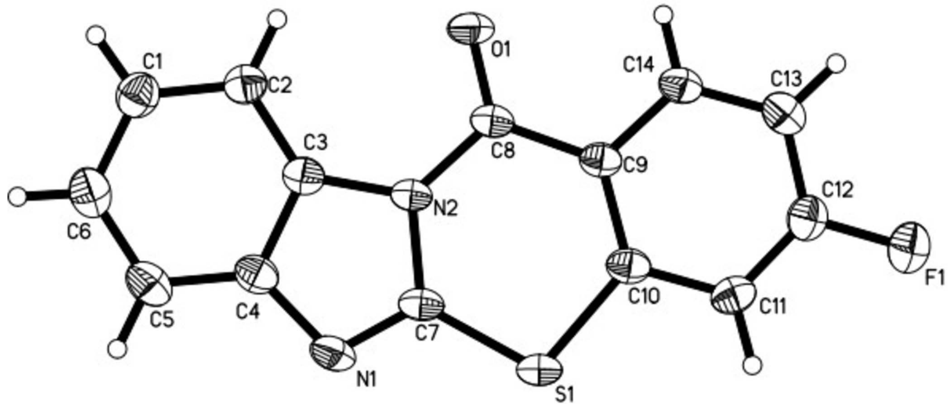

Figure 1

Ellipsoid plot.

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Crystal data

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$a = 9.5027$ (12) Å
$b = 7.0759$ (9) Å
$c = 16.931$ (2) Å
$\beta = 94.375$ (3)°
$V = 1135.1$ (2) Å^3
$Z = 4$
$F(000) = 552$
$D_x = 1.582$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3848 reflections
$\theta = 2.4$–29.8°
$\mu = 0.29$ mm^{-1}
$T = 293$ K
Block, colourless
0.20 × 0.18 × 0.15 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, 2000)
$T_{min} = 0.944$, $T_{max} = 0.958$
5987 measured reflections
1989 independent reflections
1772 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.4°$
$h = -9 \rightarrow 11$
$k = -8 \rightarrow 8$
$l = -20 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.106$
$S = 1.00$
1989 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 0.4006P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	−0.14487 (5)	0.16939 (8)	0.36622 (3)	0.0538 (2)
F1	−0.43167 (13)	0.1082 (2)	0.59933 (8)	0.0691 (4)
O1	0.19345 (15)	0.3338 (2)	0.54945 (8)	0.0550 (4)
N1	0.08266 (18)	0.2319 (2)	0.28889 (9)	0.0512 (4)
N2	0.12490 (15)	0.2778 (2)	0.42060 (8)	0.0386 (3)
C1	0.4856 (2)	0.4053 (3)	0.36667 (13)	0.0549 (5)
H1	0.5742	0.4478	0.3860	0.066*
C2	0.3838 (2)	0.3749 (3)	0.41907 (12)	0.0478 (4)
H2	0.4024	0.3929	0.4733	0.057*
C3	0.2520 (2)	0.3161 (2)	0.38695 (11)	0.0409 (4)
C4	0.2239 (2)	0.2870 (3)	0.30570 (11)	0.0458 (4)
C5	0.3284 (2)	0.3134 (3)	0.25403 (12)	0.0570 (5)
H5	0.3112	0.2914	0.2000	0.068*
C6	0.4596 (2)	0.3741 (3)	0.28586 (13)	0.0600 (5)
H6	0.5314	0.3942	0.2525	0.072*
C7	0.0288 (2)	0.2291 (2)	0.35679 (10)	0.0429 (4)
C8	0.0987 (2)	0.2875 (2)	0.50112 (10)	0.0404 (4)
C9	−0.04393 (18)	0.2395 (2)	0.52280 (10)	0.0387 (4)
C10	−0.1557 (2)	0.1855 (2)	0.46872 (11)	0.0420 (4)
C11	−0.2867 (2)	0.1387 (3)	0.49526 (12)	0.0474 (5)
H11	−0.3609	0.0999	0.4599	0.057*
C12	−0.3033 (2)	0.1512 (3)	0.57463 (12)	0.0496 (5)
C13	−0.1972 (2)	0.2037 (3)	0.62965 (12)	0.0517 (5)
H13	−0.2121	0.2090	0.6833	0.062*
C14	−0.0687 (2)	0.2480 (3)	0.60323 (11)	0.0446 (4)
H14	0.0043	0.2848	0.6397	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0482 (3)	0.0744 (4)	0.0365 (3)	−0.0045 (2)	−0.0113 (2)	−0.0035 (2)
F1	0.0542 (7)	0.0824 (9)	0.0723 (8)	−0.0065 (6)	0.0150 (6)	−0.0017 (7)
O1	0.0544 (8)	0.0695 (9)	0.0387 (7)	−0.0113 (7)	−0.0121 (6)	−0.0058 (6)
N1	0.0585 (10)	0.0611 (10)	0.0325 (8)	0.0036 (8)	−0.0060 (7)	0.0026 (7)
N2	0.0429 (8)	0.0397 (8)	0.0319 (7)	0.0020 (6)	−0.0068 (6)	−0.0009 (6)
C1	0.0490 (11)	0.0546 (11)	0.0610 (12)	0.0014 (9)	0.0031 (9)	0.0037 (9)

C2	0.0474 (10)	0.0477 (10)	0.0475 (10)	0.0006 (8)	−0.0014 (8)	−0.0001 (8)
C3	0.0469 (10)	0.0344 (8)	0.0407 (9)	0.0051 (7)	−0.0021 (8)	0.0031 (7)
C4	0.0537 (11)	0.0430 (9)	0.0399 (10)	0.0074 (8)	−0.0024 (8)	0.0059 (8)
C5	0.0690 (14)	0.0596 (12)	0.0425 (11)	0.0118 (10)	0.0046 (10)	0.0067 (9)
C6	0.0596 (13)	0.0646 (13)	0.0573 (13)	0.0074 (10)	0.0135 (10)	0.0108 (10)
C7	0.0491 (10)	0.0412 (9)	0.0362 (9)	0.0041 (8)	−0.0105 (7)	0.0007 (7)
C8	0.0480 (10)	0.0377 (9)	0.0341 (9)	0.0022 (7)	−0.0070 (7)	−0.0009 (7)
C9	0.0448 (10)	0.0336 (8)	0.0365 (9)	0.0050 (7)	−0.0052 (7)	0.0002 (7)
C10	0.0488 (10)	0.0368 (9)	0.0389 (9)	0.0059 (7)	−0.0066 (8)	0.0012 (7)
C11	0.0431 (10)	0.0449 (10)	0.0525 (11)	0.0021 (8)	−0.0075 (8)	0.0003 (8)
C12	0.0473 (11)	0.0447 (10)	0.0572 (12)	0.0039 (8)	0.0069 (9)	0.0030 (8)
C13	0.0619 (12)	0.0507 (11)	0.0425 (10)	0.0065 (9)	0.0053 (9)	−0.0003 (8)
C14	0.0507 (10)	0.0443 (9)	0.0378 (9)	0.0050 (8)	−0.0046 (8)	−0.0012 (7)

Geometric parameters (Å, º)

S1—C7	1.723 (2)	C4—C5	1.385 (3)
S1—C10	1.7501 (19)	C5—C6	1.388 (3)
F1—C12	1.354 (2)	C5—H5	0.9300
O1—C8	1.214 (2)	C6—H6	0.9300
N1—C7	1.294 (2)	C8—C9	1.471 (3)
N1—C4	1.406 (3)	C9—C10	1.402 (2)
N2—C3	1.401 (2)	C9—C14	1.401 (2)
N2—C7	1.403 (2)	C10—C11	1.395 (3)
N2—C8	1.406 (2)	C11—C12	1.368 (3)
C1—C2	1.378 (3)	C11—H11	0.9300
C1—C6	1.389 (3)	C12—C13	1.371 (3)
C1—H1	0.9300	C13—C14	1.369 (3)
C2—C3	1.390 (3)	C13—H13	0.9300
C2—H2	0.9300	C14—H14	0.9300
C3—C4	1.397 (3)		
C7—S1—C10	101.85 (9)	N1—C7—S1	122.20 (14)
C7—N1—C4	105.11 (16)	N2—C7—S1	124.09 (14)
C3—N2—C7	105.38 (14)	O1—C8—N2	119.32 (17)
C3—N2—C8	127.34 (15)	O1—C8—C9	122.96 (16)
C7—N2—C8	127.28 (15)	N2—C8—C9	117.72 (15)
C2—C1—C6	121.9 (2)	C10—C9—C14	118.16 (17)
C2—C1—H1	119.1	C10—C9—C8	124.53 (16)
C6—C1—H1	119.1	C14—C9—C8	117.31 (16)
C1—C2—C3	116.76 (19)	C11—C10—C9	120.31 (17)
C1—C2—H2	121.6	C11—C10—S1	115.18 (14)
C3—C2—H2	121.6	C9—C10—S1	124.51 (15)
C2—C3—C4	121.90 (18)	C12—C11—C10	118.25 (18)
C2—C3—N2	132.68 (17)	C12—C11—H11	120.9
C4—C3—N2	105.41 (16)	C10—C11—H11	120.9
C5—C4—C3	120.66 (19)	F1—C12—C13	118.95 (18)
C5—C4—N1	128.96 (19)	F1—C12—C11	117.57 (18)

C3—C4—N1	110.38 (17)	C13—C12—C11	123.47 (19)
C4—C5—C6	117.5 (2)	C12—C13—C14	117.93 (19)
C4—C5—H5	121.2	C12—C13—H13	121.0
C6—C5—H5	121.2	C14—C13—H13	121.0
C5—C6—C1	121.2 (2)	C13—C14—C9	121.85 (18)
C5—C6—H6	119.4	C13—C14—H14	119.1
C1—C6—H6	119.4	C9—C14—H14	119.1
N1—C7—N2	113.71 (17)		

C6—C1—C2—C3	−1.7 (3)	C10—S1—C7—N2	−0.75 (17)
C1—C2—C3—C4	0.7 (3)	C3—N2—C8—O1	0.0 (3)
C1—C2—C3—N2	−177.85 (18)	C7—N2—C8—O1	−179.37 (17)
C7—N2—C3—C2	178.39 (19)	C3—N2—C8—C9	−179.66 (15)
C8—N2—C3—C2	−1.1 (3)	C7—N2—C8—C9	0.9 (2)
C7—N2—C3—C4	−0.31 (18)	O1—C8—C9—C10	180.00 (17)
C8—N2—C3—C4	−179.83 (15)	N2—C8—C9—C10	−0.3 (2)
C2—C3—C4—C5	1.0 (3)	O1—C8—C9—C14	−0.2 (3)
N2—C3—C4—C5	179.83 (16)	N2—C8—C9—C14	179.46 (15)
C2—C3—C4—N1	−178.82 (16)	C14—C9—C10—C11	−1.1 (2)
N2—C3—C4—N1	0.06 (19)	C8—C9—C10—C11	178.63 (16)
C7—N1—C4—C5	−179.51 (19)	C14—C9—C10—S1	179.32 (13)
C7—N1—C4—C3	0.2 (2)	C8—C9—C10—S1	−0.9 (2)
C3—C4—C5—C6	−1.6 (3)	C7—S1—C10—C11	−178.27 (13)
N1—C4—C5—C6	178.15 (19)	C7—S1—C10—C9	1.29 (17)
C4—C5—C6—C1	0.6 (3)	C9—C10—C11—C12	1.5 (3)
C2—C1—C6—C5	1.0 (3)	S1—C10—C11—C12	−178.94 (14)
C4—N1—C7—N2	−0.5 (2)	C10—C11—C12—F1	178.98 (16)
C4—N1—C7—S1	179.82 (13)	C10—C11—C12—C13	−1.4 (3)
C3—N2—C7—N1	0.5 (2)	F1—C12—C13—C14	−179.48 (17)
C8—N2—C7—N1	−179.97 (16)	C11—C12—C13—C14	0.9 (3)
C3—N2—C7—S1	−179.78 (13)	C12—C13—C14—C9	−0.5 (3)
C8—N2—C7—S1	−0.3 (2)	C10—C9—C14—C13	0.6 (3)
C10—S1—C7—N1	178.93 (15)	C8—C9—C14—C13	−179.15 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···N1^{i}	0.93	2.58	3.359 (2)	141

Symmetry code: (i) *x*, −*y*+1/2, *z*+1/2.