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2-(3-Oxo-3,4-dihydro-2*H*-1,4-benzothiazin-4-yl)acetohydrazide

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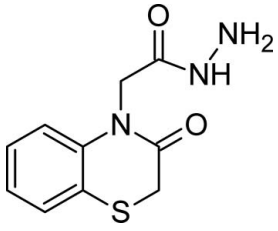
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.027; wR factor = 0.076; data-to-parameter ratio = 13.8.

In the title compound, $C_{10}H_{11}N_3O_2S$, the thiazine ring exists in a conformation intermediate between twist-boat and half-chair. The dihedral angle between the mean plane of the thiazine ring and the hydrazide group is 89.45 (13)°. In the crystal, N—H···O hydrogen bonds link the molecules into (100) sheets and weak C—H···O interactions further consolidate the packing.

Related literature

For the biological and medicinal activity of 1,4-benzothiazine compounds, see: Armenise *et al.* (1991); Gupta *et al.* (1993); Vicente *et al.* (2009); Schiaffella *et al.* (2006); Kaneko *et al.* (2002). For the pharmacological properties of hydrazones and their derivatives, see: Sivasankar *et al.* (1995); Satyanarayana *et al.* (2008). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{10}H_{11}N_3O_2S$
M_r = 237.28
Monoclinic, *Cc*
a = 15.3744 (10) Å
b = 7.5162 (5) Å
c = 9.6256 (7) Å
β = 95.413 (3)°
V = 1107.35 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 296 K
0.46 × 0.23 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.882, T_{max} = 0.946
6103 measured reflections
2168 independent reflections
2077 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.076
S = 1.04
2168 reflections
157 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Absolute structure: Flack (1983), 792 Friedel pairs
Flack parameter: 0.00 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3*N*···O2^{i}	0.96 (3)	2.35 (3)	3.299 (3)	171 (2)
N2—H1*N*···O1^{ii}	0.87 (2)	2.07 (2)	2.935 (2)	175.5 (18)
C3—H3···O2^{iii}	0.93	2.48	3.406 (3)	174
C8—H8*B*···O1^{iv}	0.97	2.57	3.442 (2)	150

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5598).

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2-(3-Oxo-3,4-dihydro-2*H*-1,4-benzothiazin-4-yl)acetohydrazide

Azher Saeed, Zaid Mahmood, Shiyao Yang, Saeed Ahmad and Muhammad Salim

S1. Comment

The 4H-benzo(1,4)thiazine compounds exhibit a broad spectrum of biological activitives, such as tetramic acids substituted benzothiazine derivatives are potent inhibitors of HCV polymerase (Vicente et al., 2009)and the pyrazino subsituted 1,4-benzothiazine derivatives are inhibitors of adhesion molecule-1, (Kaneko et al., 2002).They are also known to have antibacterial (Armenise et al.,1991),anticancer (Gupta et al.,1993), antifungal (Schiaffella et al., 2006) activities. The hydrazone compounds were known for their coordinating capability, pharmacological activity, antibacterial and antifungal properties (Sivasankar et al., 1995) (Satyanarayana, et al., 2008). We paid attention to the preparation of hydrazone derivatives of 2-(3-oxo-2,3-dihydro-2H-1,4-benzothiazin-3-one and we report here the structure of the title compound.

The dihedral angle between the aromatic benzene ring C1–C6 and thiazine ring C1/C6/N1/C7/C8/S1 is 16.77(0.10)° while the hydrazide group C9/C10/N2/N3 is oriented at dihedral angle of 89.45(0.13)° with respect to the thiazine ring. The symmetry related intermolecular N—H···O and C—H···O interaction form the dimer along the b axis which results in a ring motif $R_2^2(9)$ (Bernstein et al., 1995). The crystal structure is further stabilized through the N—H···O and week C—H···O interaction to form three dimentional network.

S2. Experimental

Ethyl 2-(3-oxo-2,3-dihydrobenzo[b][1,4]thiazin-4-yl)acetate (1.26 g, 5mmol) was refluxed in 50 ml ethanol with 2.0 ml of hydrazine for 5 hours. On completion the solution was evaporated under reduce pressure and solid obtained was purified from ethanol. Colourless needles of (I) were obtained by slow evaporation from methanol (m.p. 430 K).

S3. Refinement

The C-H H-atoms were positioned with idealized geometry with C—H = 0.93 Å and were refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(C). The N-H H atoms were located in difference map with N—H= 0.76 (4)—0.83 (4) Å, U_{iso}(H) = 1.2 for N atoms.

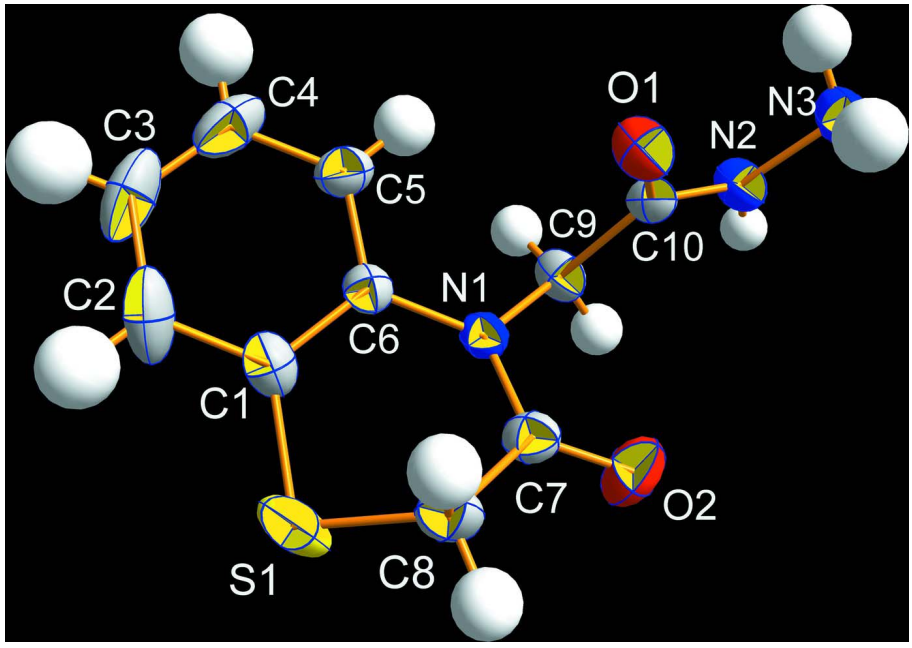

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

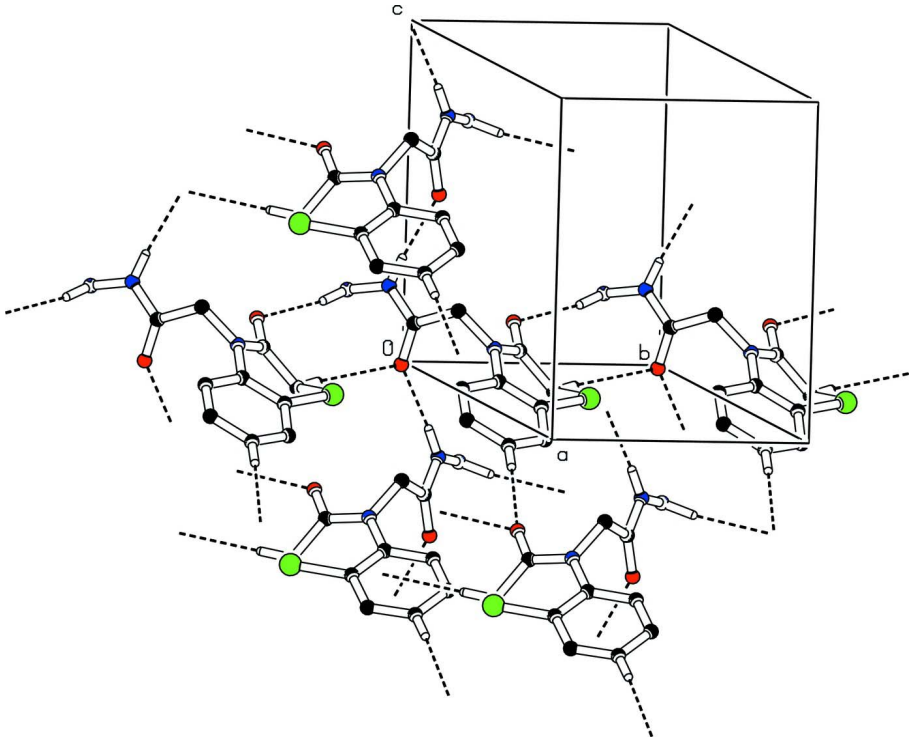

Figure 2

The crystal packing of the title compound and Intermolecular hydrogen bonds are shown by dashed lines.

2-(3-Oxo-3,4-dihydro-2*H*-1,4-benzothiazin-4-yl)acetohydrazide

Crystal data

$C_{10}H_{11}N_3O_2S$
$M_r = 237.28$
Monoclinic, *Cc*
Hall symbol: C -2yc
$a = 15.3744\ (10)$ Å
$b = 7.5162\ (5)$ Å
$c = 9.6256\ (7)$ Å
$\beta = 95.413\ (3)°$
$V = 1107.35\ (13)$ Å^3
$Z = 4$
$F(000) = 496$
$D_x = 1.423$ Mg m^{-3}
Melting point: 430 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4206 reflections
$\theta = 3.0–31.2°$
$\mu = 0.28$ mm^{-1}
$T = 296$ K
Needle, colorless
0.46 × 0.23 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min} = 0.882$, $T_{max} = 0.946$
6103 measured reflections
2168 independent reflections
2077 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 28.4°$, $\theta_{min} = 2.7°$
$h = -12\rightarrow20$
$k = -8\rightarrow10$
$l = -12\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.076$
$S = 1.04$
2168 reflections
157 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.2667P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Absolute structure: Flack (1983), 792 Friedel pairs
Absolute structure parameter: 0.00 (7)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.81283 (13)	0.1402 (3)	0.4407 (2)	0.0444 (4)
C2	0.88393 (15)	0.1642 (4)	0.3624 (3)	0.0684 (7)

H2	0.9061	0.0674	0.3170	0.082*
C3	0.92138 (15)	0.3276 (5)	0.3514 (3)	0.0727 (7)
H3	0.9691	0.3412	0.2998	0.087*
C4	0.88855 (14)	0.4717 (4)	0.4166 (3)	0.0621 (6)
H4	0.9145	0.5827	0.4099	0.074*
C5	0.81688 (12)	0.4524 (2)	0.4924 (2)	0.0451 (4)
H5	0.7944	0.5514	0.5347	0.054*
C6	0.77806 (10)	0.2868 (2)	0.50596 (15)	0.0325 (3)
C7	0.64582 (13)	0.1320 (2)	0.56774 (18)	0.0393 (4)
C8	0.65696 (13)	−0.0005 (2)	0.4532 (2)	0.0473 (4)
H8A	0.6398	0.0537	0.3633	0.057*
H8B	0.6194	−0.1024	0.4638	0.057*
C9	0.68864 (12)	0.3985 (2)	0.69169 (16)	0.0371 (4)
H9A	0.7419	0.4638	0.7190	0.044*
H9B	0.6710	0.3388	0.7738	0.044*
C10	0.61760 (11)	0.52828 (19)	0.63748 (15)	0.0311 (3)
N1	0.70588 (9)	0.26545 (17)	0.58681 (13)	0.0328 (3)
N2	0.57596 (11)	0.60513 (19)	0.73773 (16)	0.0391 (3)
N3	0.50598 (12)	0.7263 (3)	0.70912 (19)	0.0491 (4)
O1	0.60169 (9)	0.56431 (16)	0.51324 (12)	0.0441 (3)
O2	0.58443 (11)	0.1213 (2)	0.63853 (16)	0.0602 (4)
S1	0.76839 (4)	−0.07275 (6)	0.45907 (6)	0.06435 (19)
H1N	0.5831 (14)	0.561 (3)	0.822 (3)	0.036 (5)*
H2N	0.4722 (19)	0.678 (4)	0.641 (3)	0.072 (9)*
H3N	0.5321 (18)	0.834 (4)	0.681 (3)	0.065 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0391 (9)	0.0492 (9)	0.0446 (10)	0.0071 (7)	0.0020 (8)	−0.0098 (8)
C2	0.0384 (11)	0.108 (2)	0.0598 (13)	0.0113 (12)	0.0099 (10)	−0.0256 (14)
C3	0.0360 (10)	0.126 (2)	0.0580 (14)	−0.0063 (14)	0.0133 (9)	0.0076 (15)
C4	0.0409 (10)	0.0773 (15)	0.0673 (14)	−0.0139 (10)	0.0019 (10)	0.0272 (12)
C5	0.0411 (10)	0.0407 (9)	0.0531 (12)	−0.0021 (8)	0.0022 (8)	0.0107 (8)
C6	0.0334 (8)	0.0347 (7)	0.0288 (8)	0.0042 (6)	0.0004 (6)	0.0033 (6)
C7	0.0462 (10)	0.0359 (8)	0.0360 (9)	−0.0013 (7)	0.0057 (7)	0.0027 (7)
C8	0.0532 (11)	0.0358 (9)	0.0529 (11)	−0.0081 (7)	0.0049 (8)	−0.0080 (7)
C9	0.0463 (9)	0.0382 (8)	0.0266 (8)	0.0068 (7)	0.0026 (7)	−0.0034 (6)
C10	0.0391 (8)	0.0292 (7)	0.0253 (7)	−0.0012 (6)	0.0052 (6)	−0.0015 (5)
N1	0.0393 (7)	0.0293 (6)	0.0304 (7)	0.0025 (5)	0.0068 (6)	−0.0012 (5)
N2	0.0516 (9)	0.0405 (7)	0.0261 (7)	0.0102 (6)	0.0081 (6)	0.0003 (6)
N3	0.0518 (10)	0.0517 (10)	0.0455 (9)	0.0147 (8)	0.0134 (8)	0.0016 (8)
O1	0.0585 (8)	0.0504 (7)	0.0239 (6)	0.0137 (6)	0.0059 (5)	0.0007 (5)
O2	0.0559 (9)	0.0728 (9)	0.0553 (9)	−0.0161 (7)	0.0223 (7)	−0.0008 (8)
S1	0.0661 (3)	0.0354 (2)	0.0898 (4)	0.0116 (2)	−0.0018 (3)	−0.0191 (3)

Geometric parameters (Å, º)

C1—C2	1.397 (3)	C7—C8	1.508 (3)
C1—C6	1.400 (2)	C8—S1	1.793 (2)
C1—S1	1.756 (2)	C8—H8A	0.9700
C2—C3	1.364 (4)	C8—H8B	0.9700
C2—H2	0.9300	C9—N1	1.463 (2)
C3—C4	1.372 (4)	C9—C10	1.520 (2)
C3—H3	0.9300	C9—H9A	0.9700
C4—C5	1.385 (3)	C9—H9B	0.9700
C4—H4	0.9300	C10—O1	1.228 (2)
C5—C6	1.392 (2)	C10—N2	1.338 (2)
C5—H5	0.9300	N2—N3	1.417 (2)
C6—N1	1.423 (2)	N2—H1N	0.87 (2)
C7—O2	1.218 (2)	N3—H2N	0.87 (3)
C7—N1	1.364 (2)	N3—H3N	0.96 (3)
C2—C1—C6	119.5 (2)	S1—C8—H8A	109.5
C2—C1—S1	120.28 (17)	C7—C8—H8B	109.5
C6—C1—S1	120.24 (15)	S1—C8—H8B	109.5
C3—C2—C1	121.0 (2)	H8A—C8—H8B	108.1
C3—C2—H2	119.5	N1—C9—C10	111.90 (13)
C1—C2—H2	119.5	N1—C9—H9A	109.2
C2—C3—C4	119.9 (2)	C10—C9—H9A	109.2
C2—C3—H3	120.0	N1—C9—H9B	109.2
C4—C3—H3	120.0	C10—C9—H9B	109.2
C3—C4—C5	120.3 (2)	H9A—C9—H9B	107.9
C3—C4—H4	119.9	O1—C10—N2	122.83 (15)
C5—C4—H4	119.9	O1—C10—C9	123.14 (14)
C4—C5—C6	120.8 (2)	N2—C10—C9	113.99 (13)
C4—C5—H5	119.6	C7—N1—C6	124.21 (13)
C6—C5—H5	119.6	C7—N1—C9	115.53 (14)
C5—C6—C1	118.46 (16)	C6—N1—C9	120.12 (13)
C5—C6—N1	121.03 (15)	C10—N2—N3	122.91 (15)
C1—C6—N1	120.49 (15)	C10—N2—H1N	118.4 (13)
O2—C7—N1	121.60 (17)	N3—N2—H1N	117.0 (14)
O2—C7—C8	120.86 (17)	N2—N3—H2N	105.3 (19)
N1—C7—C8	117.53 (15)	N2—N3—H3N	105.6 (16)
C7—C8—S1	110.54 (14)	H2N—N3—H3N	112 (2)
C7—C8—H8A	109.5	C1—S1—C8	95.78 (9)
C6—C1—C2—C3	1.6 (3)	O2—C7—N1—C6	−178.88 (18)
S1—C1—C2—C3	−177.5 (2)	C8—C7—N1—C6	−0.2 (2)
C1—C2—C3—C4	−0.8 (4)	O2—C7—N1—C9	−3.3 (2)
C2—C3—C4—C5	−0.7 (4)	C8—C7—N1—C9	175.45 (16)
C3—C4—C5—C6	1.2 (3)	C5—C6—N1—C7	155.73 (17)
C4—C5—C6—C1	−0.3 (3)	C1—C6—N1—C7	−25.7 (2)
C4—C5—C6—N1	178.29 (17)	C5—C6—N1—C9	−19.7 (2)

C2—C1—C6—C5	−1.1 (3)	C1—C6—N1—C9	158.89 (15)
S1—C1—C6—C5	178.02 (14)	C10—C9—N1—C7	−76.87 (18)
C2—C1—C6—N1	−179.71 (19)	C10—C9—N1—C6	98.95 (17)
S1—C1—C6—N1	−0.6 (2)	O1—C10—N2—N3	4.2 (3)
O2—C7—C8—S1	−135.26 (18)	C9—C10—N2—N3	−178.15 (17)
N1—C7—C8—S1	46.0 (2)	C2—C1—S1—C8	−143.10 (19)
N1—C9—C10—O1	−26.9 (2)	C6—C1—S1—C8	37.80 (16)
N1—C9—C10—N2	155.47 (14)	C7—C8—S1—C1	−58.15 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O2^{i}	0.96 (3)	2.35 (3)	3.299 (3)	171 (2)
N2—H1*N*···O1^{ii}	0.87 (2)	2.07 (2)	2.935 (2)	175.5 (18)
C3—H3···O2^{iii}	0.93	2.48	3.406 (3)	174
C8—H8*B*···O1^{iv}	0.97	2.57	3.442 (2)	150

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*, −*y*+1, *z*+1/2; (iii) *x*+1/2, −*y*+1/2, *z*−1/2; (iv) *x*, *y*−1, *z*.