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3-(1-Benzofuran-2-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione monohydrate

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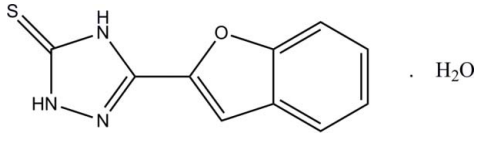
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.108; data-to-parameter ratio = 27.2.

In the title hydrate, $C_{10}H_7N_3OS\cdot H_2O$, the essentially planar benzofuran [maximum deviation = 0.006 (1) Å] and 4,5-dihydro-1*H*-1,2,4-triazole [maximum deviation = 0.007 (1) Å] rings form a dihedral angle of 11.67 (6)°. In the crystal, O—H···N, O—H···S, N—H···O and N—H···S hydrogen bonds link the molecules into sheets lying parallel to the *bc* plane. Aromatic π–π stacking interactions [centroid–centroid distances = 3.5078 (8)–3.6113 (8) Å] are also observed.

Related literature

For background to 1,2,4-triazoles, see: Shujuan *et al.* (2004); Clemons *et al.* (2004); Johnston (2002); Wei *et al.* (2007). For related structures, see: Jing *et al.* (2012); Fun *et al.* (2011); Abdel-Aziz *et al.* (2011). For stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



. H_2O

Experimental

Crystal data

$C_{10}H_7N_3OS\cdot H_2O$
M_r = 235.26
Monoclinic, $P2_1/c$
a = 7.1446 (1) Å
b = 8.8075 (1) Å
c = 17.3274 (2) Å
β = 111.942 (1)°
V = 1011.36 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 100 K
0.39 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.891, T_{max} = 0.955
19917 measured reflections
4162 independent reflections
3347 reflections with $> I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.108
S = 1.07
4162 reflections
153 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1*W*—H1*OW*···N2^{i}	0.90	2.05	2.9135 (14)	160
O1*W*—H2*OW*···S1^{ii}	0.82	2.46	3.2674 (11)	167
N1—H1*N1*···O1*W*^{iii}	0.90 (2)	1.81 (2)	2.7100 (14)	172.6 (19)
N3—H1*N3*···S1^{iv}	0.846 (18)	2.498 (18)	3.3242 (10)	165.7 (16)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6837).

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supplementary materials

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3-(1-Benzofuran-2-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione monohydrate

Hoong-Kun Fun, Suhana Arshad, Nithinchandra, Balakrishna Kalluraya and Shobhitha Shetty

Comment

The 1,2,4-triazole nucleus has been incorporated into a wide variety of therapeutically interesting compounds. Several compounds containing 1,2,4-triazole rings are well known as drugs. For example, fluconazole is used as an antimicrobial drug (Shujuan *et al.*, 2004), whereas vorozole, letrozole and anastrozole are non-steroidal drugs used for the treatment of cancer (Clemons *et al.*, 2004) and loreclezole is used as an anticonvulsant (Johnston, 2002). Similarly substituted derivatives of triazole possess comprehensive bioactivities such as antimicrobial, anti-inflammatory, analgesic, antihypertensive, anticonvulsant and antiviral activities (Wei *et al.*, 2007). We now report the synthesis and crystal structure of the title compound.

The asymmetric unit of the title compound, (Fig. 1), consists of one 5-(1-Benzofuran-2-yl)-2,4-dihydro-3*H*-1,2,4-triazole-3-thione molecule and one water molecule. The benzofuran ring (O1/C3–C10) and the 4,5-dihydro -1*H*-1,2,4-triazole ring (N1–N3/C1/C2) are essentially planar with maximum deviations of 0.006 (1) Å at atom O1 and 0.007 (1) Å at atom N3, respectively. The dihedral angle between the benzofuran and 4,5-dihydro- 1*H*-1,2,4-triazole rings is 11.67 (6)°. Bond lengths and angles are within normal ranges and comparable to the related structures (Jing *et al.*, 2012; Fun *et al.*, 2011; Abdel-Aziz *et al.*, 2011).

The crystal packing is shown in Fig. 2. The molecules are linked *via* O1W—H1OW···N2, O1W—H2OW···S1, N1—H1N1···O1W and N3—H1N3···S1 hydrogen bonds (Table 1) into two-dimensional networks parallel to *bc*-plane. π–π interactions of *Cg*1···*Cg*1 = 3.6113 (8) Å (symmetry code: 1 - *x*, -*y*, 1 - *z*), *Cg*1···*Cg*2 = 3.5078 (8) Å (symmetry code: 2 - *x*, -*y*, 1 - *z*), *Cg*2···*Cg*3 = 3.5881 (8) Å (symmetry code: 1 - *x*, -*y*, 1 - *z*) and *Cg*3···*Cg*2 = 3.6056 (8) Å (symmetry code: 2 - *x*, -*y*, 1 - *z*) further stabilized the crystal structure [*Cg*1, *Cg*2 and *Cg*3 are the centroids of the O1/C3–C5/C10, N1–N3/C1/C2 and C5–C10 rings, respectively].

Experimental

A mixture of 2-(1-benzofuran-2-ylcarbonyl)hydrazinecarbothioamide (0.01 mol) and 10% KOH (10 ml) was refluxed for 3 h. The mixture was cooled to room temperature and then neutralized by the gradual addition of glacial acetic acid. The solid product obtained was collected by filtration, washed with ethanol and dried. It was then recrystallized using ethanol. Yellow blocks of the title compound were obtained by slow evaporation of the ethanolic solution.

Refinement

O– and N-bound H atoms were located from a difference Fourier map. O-bound H atoms were fixed at their found positions (O–H = 0.8961 and 0.8208 Å), with U_{iso}(H) = 1.5 U_{eq}(O), whereas N-bound H atoms was refined freely [N–H = 0.844 (18) and 0.90 (2) Å]. The remaining H atoms were positioned geometrically [C–H = 0.93 Å] and refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(C). In the final refinement, one outlier (1 1 1) was omitted.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

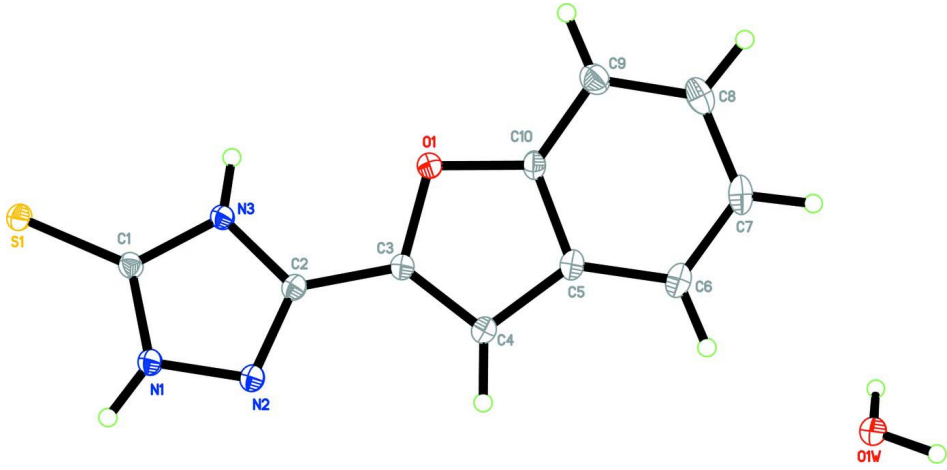

Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

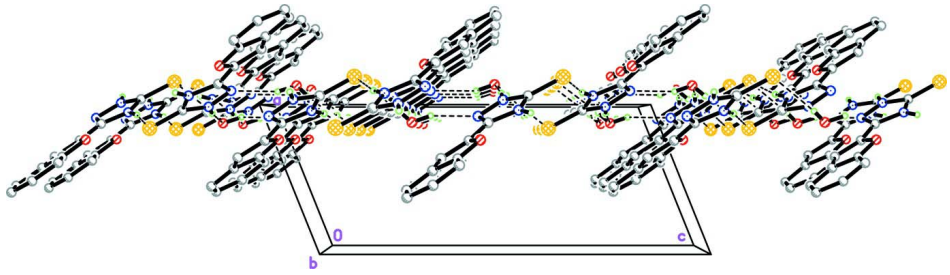

Figure 2

The crystal packing of the title compound, viewed down the *b* axis. The H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

3-(1-Benzofuran-2-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione monohydrate

Crystal data

$C_{10}H_7N_3OS·H_2O$
$M_r = 235.26$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.1446$ (1) Å
$b = 8.8075$ (1) Å
$c = 17.3274$ (2) Å
$\beta = 111.942$ (1)°
$V = 1011.36$ (2) Å^3
$Z = 4$
$F(000) = 488$
$D_x = 1.545$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6273 reflections
$\theta = 2.5$–33.4°
$\mu = 0.31$ mm^{-1}
$T = 100$ K
Block, yellow
0.39 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.891, T_{max} = 0.955

19917 measured reflections
4162 independent reflections
3347 reflections with > I > 2σ(I)
R_{int} = 0.039
θ_{max} = 34.2°, θ_{min} = 2.5°
h = −11→11
k = −13→13
l = −26→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.108
S = 1.07
4162 reflections
153 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
$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.4347P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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	1.16013 (5)	−0.27662 (3)	0.789110 (18)	0.01410 (8)
O1	0.76589 (13)	0.12211 (10)	0.53014 (5)	0.01383 (17)
N1	1.01801 (16)	−0.33637 (12)	0.62307 (6)	0.01333 (19)
N2	0.91410 (16)	−0.27263 (11)	0.54676 (6)	0.01371 (19)
N3	0.95013 (15)	−0.11026 (11)	0.64872 (6)	0.01228 (18)
C1	1.04179 (18)	−0.24126 (13)	0.68643 (7)	0.0121 (2)
C2	0.87285 (17)	−0.13492 (13)	0.56444 (7)	0.0120 (2)
C3	0.76153 (17)	−0.02501 (13)	0.50237 (7)	0.0122 (2)
C4	0.65217 (18)	−0.03918 (13)	0.41967 (7)	0.0139 (2)
H4A	0.6284	−0.1277	0.3882	0.017*
C5	0.58061 (17)	0.11092 (14)	0.39077 (7)	0.0134 (2)
C6	0.46309 (19)	0.17499 (15)	0.31360 (8)	0.0169 (2)
H6A	0.4126	0.1157	0.2658	0.020*
C7	0.42492 (19)	0.32943 (16)	0.31110 (8)	0.0185 (2)

H7A	0.3474	0.3741	0.2606	0.022*
C8	0.5000 (2)	0.42034 (15)	0.38265 (9)	0.0193 (2)
H8A	0.4710	0.5236	0.3784	0.023*
C9	0.6168 (2)	0.35953 (14)	0.45977 (8)	0.0175 (2)
H9A	0.6675	0.4189	0.5075	0.021*
C10	0.65281 (18)	0.20498 (13)	0.46092 (7)	0.0128 (2)
O1W	0.12342 (14)	0.13102 (10)	0.11923 (6)	0.01697 (18)
H1OW	0.0827	0.1677	0.0673	0.025*
H2OW	0.0538	0.1810	0.1383	0.025*
H1N1	1.062 (3)	−0.433 (2)	0.6261 (12)	0.032 (5)*
H1N3	0.933 (3)	−0.032 (2)	0.6736 (11)	0.016 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01807 (14)	0.01305 (13)	0.00963 (13)	0.00052 (9)	0.00341 (10)	0.00123 (9)
O1	0.0168 (4)	0.0119 (4)	0.0113 (4)	0.0021 (3)	0.0035 (3)	−0.0001 (3)
N1	0.0172 (5)	0.0122 (4)	0.0099 (4)	0.0022 (3)	0.0043 (3)	0.0014 (3)
N2	0.0167 (5)	0.0130 (4)	0.0103 (4)	0.0017 (3)	0.0038 (3)	0.0009 (3)
N3	0.0158 (4)	0.0110 (4)	0.0098 (4)	0.0021 (3)	0.0044 (3)	0.0005 (3)
C1	0.0137 (5)	0.0112 (4)	0.0118 (5)	0.0005 (4)	0.0051 (4)	0.0013 (4)
C2	0.0132 (5)	0.0126 (5)	0.0100 (5)	0.0005 (4)	0.0040 (4)	0.0004 (4)
C3	0.0130 (5)	0.0121 (5)	0.0108 (5)	0.0008 (4)	0.0036 (4)	0.0007 (4)
C4	0.0152 (5)	0.0132 (5)	0.0115 (5)	0.0006 (4)	0.0027 (4)	−0.0001 (4)
C5	0.0123 (5)	0.0159 (5)	0.0113 (5)	0.0007 (4)	0.0038 (4)	0.0024 (4)
C6	0.0156 (5)	0.0214 (6)	0.0120 (5)	0.0006 (4)	0.0030 (4)	0.0029 (4)
C7	0.0153 (5)	0.0224 (6)	0.0168 (6)	0.0041 (4)	0.0049 (4)	0.0085 (5)
C8	0.0187 (5)	0.0166 (5)	0.0236 (6)	0.0048 (4)	0.0090 (5)	0.0061 (5)
C9	0.0200 (6)	0.0147 (5)	0.0182 (6)	0.0029 (4)	0.0077 (4)	0.0010 (4)
C10	0.0134 (5)	0.0135 (5)	0.0108 (5)	0.0018 (4)	0.0037 (4)	0.0026 (4)
O1W	0.0225 (4)	0.0144 (4)	0.0136 (4)	0.0006 (3)	0.0064 (3)	0.0009 (3)

Geometric parameters (Å, º)

S1—C1	1.6892 (12)	C4—H4A	0.9300
O1—C10	1.3784 (14)	C5—C10	1.4003 (17)
O1—C3	1.3785 (14)	C5—C6	1.4045 (17)
N1—C1	1.3403 (16)	C6—C7	1.3848 (19)
N1—N2	1.3715 (14)	C6—H6A	0.9300
N1—H1N1	0.90 (2)	C7—C8	1.403 (2)
N2—C2	1.3112 (15)	C7—H7A	0.9300
N3—C1	1.3664 (15)	C8—C9	1.3914 (18)
N3—C2	1.3718 (15)	C8—H8A	0.9300
N3—H1N3	0.844 (18)	C9—C10	1.3840 (17)
C2—C3	1.4448 (16)	C9—H9A	0.9300
C3—C4	1.3575 (16)	O1W—H1OW	0.8961
C4—C5	1.4382 (16)	O1W—H2OW	0.8208
C10—O1—C3	105.34 (9)	C10—C5—C6	118.94 (11)
C1—N1—N2	113.03 (10)	C10—C5—C4	105.90 (10)

C1—N1—H1N1	127.4 (13)	C6—C5—C4	135.16 (12)
N2—N1—H1N1	119.6 (13)	C7—C6—C5	117.72 (12)
C2—N2—N1	103.96 (10)	C7—C6—H6A	121.1
C1—N3—C2	107.80 (10)	C5—C6—H6A	121.1
C1—N3—H1N3	125.3 (12)	C6—C7—C8	121.81 (12)
C2—N3—H1N3	126.5 (12)	C6—C7—H7A	119.1
N1—C1—N3	104.16 (10)	C8—C7—H7A	119.1
N1—C1—S1	127.44 (9)	C9—C8—C7	121.54 (12)
N3—C1—S1	128.40 (9)	C9—C8—H8A	119.2
N2—C2—N3	111.04 (10)	C7—C8—H8A	119.2
N2—C2—C3	123.72 (11)	C10—C9—C8	115.71 (12)
N3—C2—C3	125.24 (10)	C10—C9—H9A	122.1
C4—C3—O1	112.58 (10)	C8—C9—H9A	122.1
C4—C3—C2	131.59 (11)	O1—C10—C9	125.35 (11)
O1—C3—C2	115.82 (10)	O1—C10—C5	110.37 (10)
C3—C4—C5	105.81 (10)	C9—C10—C5	124.28 (11)
C3—C4—H4A	127.1	H1OW—O1W—H2OW	101.1
C5—C4—H4A	127.1		

C1—N1—N2—C2	−0.29 (14)	C2—C3—C4—C5	−178.79 (12)
N2—N1—C1—N3	−0.49 (13)	C3—C4—C5—C10	−0.85 (13)
N2—N1—C1—S1	179.94 (9)	C3—C4—C5—C6	179.54 (14)
C2—N3—C1—N1	1.06 (13)	C10—C5—C6—C7	0.09 (18)
C2—N3—C1—S1	−179.38 (9)	C4—C5—C6—C7	179.66 (13)
N1—N2—C2—N3	0.98 (13)	C5—C6—C7—C8	0.00 (19)
N1—N2—C2—C3	−179.39 (11)	C6—C7—C8—C9	0.0 (2)
C1—N3—C2—N2	−1.34 (14)	C7—C8—C9—C10	−0.17 (19)
C1—N3—C2—C3	179.04 (11)	C3—O1—C10—C9	−179.75 (12)
C10—O1—C3—C4	0.01 (13)	C3—O1—C10—C5	−0.58 (13)
C10—O1—C3—C2	179.45 (10)	C8—C9—C10—O1	179.33 (11)
N2—C2—C3—C4	11.6 (2)	C8—C9—C10—C5	0.28 (19)
N3—C2—C3—C4	−168.79 (13)	C6—C5—C10—O1	−179.42 (10)
N2—C2—C3—O1	−167.68 (11)	C4—C5—C10—O1	0.90 (13)
N3—C2—C3—O1	11.90 (17)	C6—C5—C10—C9	−0.24 (19)
O1—C3—C4—C5	0.54 (14)	C4—C5—C10—C9	−179.92 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*OW*···N2^{i}	0.90	2.05	2.9135 (14)	160
O1*W*—H2*OW*···S1^{ii}	0.82	2.46	3.2674 (11)	167
N1—H1*N*1···O1*W*^{iii}	0.90 (2)	1.81 (2)	2.7100 (14)	172.6 (19)
N3—H1*N*3···S1^{iv}	0.846 (18)	2.498 (18)	3.3242 (10)	165.7 (16)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) −*x*+1, −*y*, −*z*+1; (iii) *x*+1, −*y*−1/2, *z*+1/2; (iv) −*x*+2, *y*+1/2, −*z*+3/2.