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# 2-(1-Benzothiophen-2-yl)-4*H*-1,3,4-oxadiazin-5(6*H*)-one

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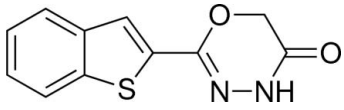
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.064; $wR$ factor = 0.192; data-to-parameter ratio = 12.7.

In the title compound, $C_{11}H_8N_2O_2S$, the oxadiazinone ring is nearly planar [maximum deviation = 0.016 (4) Å], and is approximately coplanar with the benzothiophene ring system [dihedral angle = 3.1 (5)°]. In the crystal, molecules are linked by N—H···O hydrogen bonds, forming chains running along the *b*-axis direction.

## Related literature

For applications of oxadiazin derivatives, see: De Sarro *et al.* (2005); Shigeki *et al.* (2012).



## Experimental

### Crystal data

$C_{11}H_8N_2O_2S$
$M_r$ = 232.25
Monoclinic, $P2_1/n$
$a$ = 7.4950 (15) Å
$b$ = 6.0350 (12) Å
$c$ = 22.412 (5) Å
$\beta$ = 93.08 (3)°
$V$ = 1012.3 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.30 mm$^{-1}$
$T$ = 293 K
0.30 × 0.20 × 0.10 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.915, $T_{max}$ = 0.970
1999 measured reflections
1848 independent reflections
1167 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.064
3 standard reflections every 200 reflections
intensity decay: 1%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.192
$S$ = 1.00
1848 reflections
145 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$A$···O2$^{i}$ | 0.86 | 2.03 | 2.848 (5) | 158 |

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{5}{2}$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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*.

The work was supported by Nanjing College of Chemical Technology, China (NHKY-2013–02).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5757).

## References

De Sarro, G., Gitto, R., Russo, E., Ibbadu, G. F., Barreca, M. L., De Luca, L. & Chimirri, A. (2005). *Curr. Top. Med. Chem.* **5**, 31–42.
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# supporting information

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## 2-(1-Benzothiophen-2-yl)-4*H*-1,3,4-oxadiazin-5(6*H*)-one

**Hong-Shun Sun, Yu-Long Li, Ning Xu and Lin-Jiang Shen**

### S1. Comment

Noncompetitive *α*-amino-3-hydroxy-5-methyl-4-isoxazolepropanoic acid (AMPA) receptor antagonists have actively been explored, driven by the belief that such drugs would exercise effectiveness independent of glutamatelevels and the synaptic membrane polarization state, with minor influence on normal glutamatergic activity compared with competitive antagonists (De Sarro *et al.*, 2005). Commercially available 2,4-diphenyl-4*H*-1,3,4-oxadiazin-5-one was selected as a suitable starting compound with a novel structure *versus* other HTS (high throughput screening) hit compounds and known noncompetitive AMPA antagonists (Shigeki *et al.*, 2012). The title compound is a new oxadiazin compound with a similar structure to it. We synthesized it and report here its crystal structure.

The molecular structure of the title compound is shown in Fig. 1. The benzothiophene ring makes a dihedral angle of 3.1 (5)° to oxadiazin ring.

As shown in Figure 2, the molecules are linked by N—H···O and weak S···S (Table 1) bonds into a three-dimensional network, which consolidate the crystal packing.

### S2. Experimental

*N*′-(2-chloroacetyl)benzo[*b*]thiophene-2-carbohydrazide (0.27 g, 1 mmol) was dissolved in 10 ml acetonitrile, after which potassium carbonate (0.28 g, 2.0 mmol) was added and the mixture was refluxed for 3 h under heating. The reaction solution was left and cooled to room temperature, evaporated, diluted with EtOAc, washed with water and brine, and then dried over $MgSO_4$. After the drying agent was filtered off, the product was evaporated and the resulting crystalline residues were recrystallized from n-hexane and EtOAc to give the compound (0.19 g, 0.8 mmol, 80.0%) as a yellow crystal suitable for X-ray analysis.

### S3. Refinement

H atoms were positioned geometrically, with N— H = 0.86 Å and C—H = 0.93, and 0.97 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms, $U_{iso}(H) = 1.2U_{eq}(C,N)$.

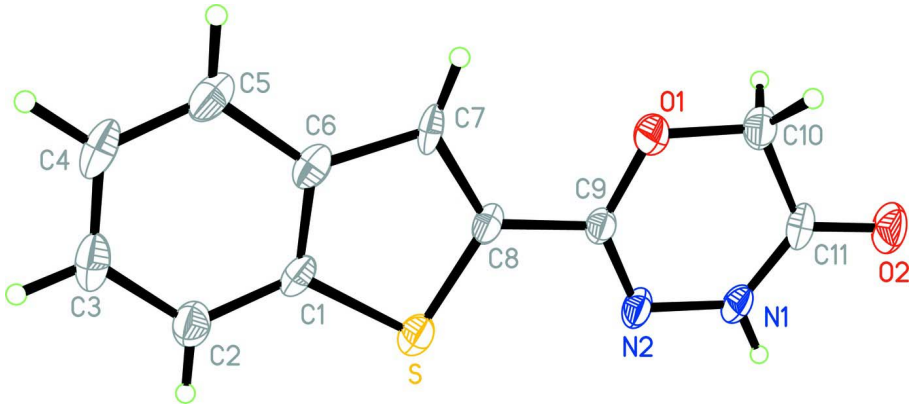


**Figure 1**

The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level.

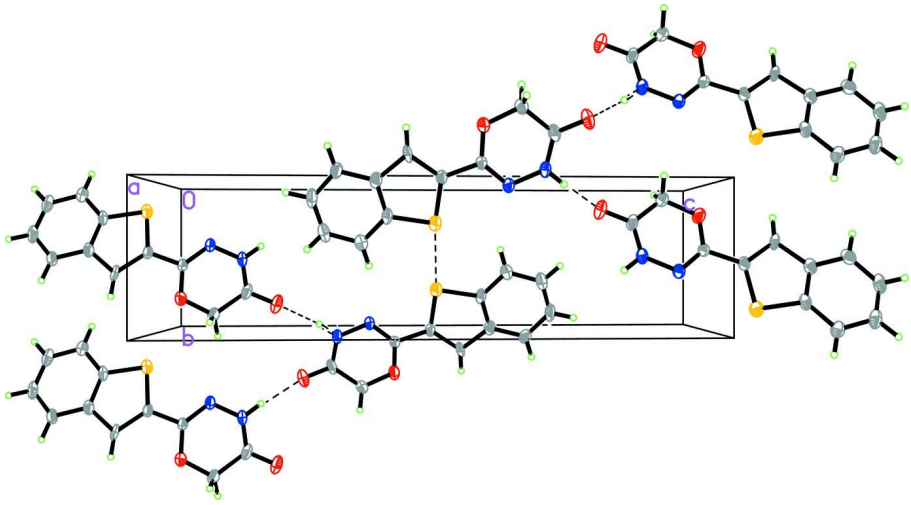


**Figure 2**

A packing diagram of (I). Intermolecular hydrogen bonds are shown as dashed lines.

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*Crystal data*

$C_{11}H_8N_2O_2S$
$M_r = 232.25$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.4950\ (15)$ Å
$b = 6.0350\ (12)$ Å
$c = 22.412\ (5)$ Å
$\beta = 93.08\ (3)°$
$V = 1012.3\ (4)$ Å$^3$
$Z = 4$

$F(000) = 480$
$D_x = 1.524$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–12°$
$\mu = 0.30$ mm$^{-1}$
$T = 293$ K
Block, colorless
0.30 × 0.20 × 0.10 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.915, $T_{max}$ = 0.970
1999 measured reflections
1848 independent reflections
1167 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.064
$\theta_{max}$ = 25.4°, $\theta_{min}$ = 1.8°
$h$ = 0→9
$k$ = 0→7
$l$ = −27→27
3 standard reflections every 200 reflections
intensity decay: 1%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.192
$S$ = 1.00
1848 reflections
145 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.650P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 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)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S | 0.66885 (16) | 0.2027 (2) | 0.99910 (5) | 0.0507 (4) |
| O2 | 0.8714 (5) | 0.7881 (6) | 1.23337 (14) | 0.0632 (10) |
| O1 | 0.8545 (5) | 0.7527 (5) | 1.07326 (14) | 0.0606 (10) |
| C1 | 0.6558 (5) | 0.2443 (8) | 0.92291 (17) | 0.0391 (10) |
| N1 | 0.7891 (5) | 0.5094 (6) | 1.17093 (15) | 0.0442 (9) |
| H1A | 0.7685 | 0.4264 | 1.2010 | 0.053* |
| C2 | 0.5909 (6) | 0.0894 (8) | 0.8802 (2) | 0.0491 (12) |
| H2B | 0.5496 | −0.0488 | 0.8917 | 0.059* |
| N2 | 0.7558 (5) | 0.4203 (6) | 1.11441 (15) | 0.0430 (9) |
| C3 | 0.5905 (6) | 0.1488 (9) | 0.8209 (2) | 0.0540 (13) |
| H3B | 0.5454 | 0.0508 | 0.7918 | 0.065* |
| C4 | 0.6552 (6) | 0.3490 (9) | 0.8041 (2) | 0.0555 (13) |
| H4A | 0.6566 | 0.3817 | 0.7636 | 0.067* |
| C5 | 0.7186 (6) | 0.5054 (8) | 0.84494 (19) | 0.0514 (12) |
| H5A | 0.7590 | 0.6427 | 0.8325 | 0.062* |

| | | | | |
|---|---|---|---|---|
| C6 | 0.7203 (6) | 0.4501 (8) | 0.90646 (18) | 0.0429 (11) |
| C7 | 0.7809 (5) | 0.5961 (8) | 0.95690 (16) | 0.0371 (10) |
| H7A | 0.8256 | 0.7395 | 0.9550 | 0.044* |
| C8 | 0.7548 (5) | 0.4656 (7) | 1.00939 (17) | 0.0379 (10) |
| C9 | 0.7923 (5) | 0.5458 (7) | 1.07104 (17) | 0.0342 (9) |
| C10 | 0.8941 (6) | 0.8522 (7) | 1.13060 (19) | 0.0461 (11) |
| H10A | 0.8284 | 0.9903 | 1.1326 | 0.055* |
| H10B | 1.0204 | 0.8878 | 1.1341 | 0.055* |
| C11 | 0.8500 (6) | 0.7115 (8) | 1.18252 (19) | 0.0446 (11) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S | 0.0665 (8) | 0.0529 (7) | 0.0334 (7) | −0.0047 (6) | 0.0096 (5) | 0.0009 (6) |
| O2 | 0.076 (2) | 0.082 (3) | 0.0322 (18) | −0.002 (2) | 0.0036 (15) | −0.0179 (18) |
| O1 | 0.104 (3) | 0.049 (2) | 0.0293 (18) | −0.0176 (18) | 0.0092 (17) | −0.0028 (14) |
| C1 | 0.038 (2) | 0.056 (3) | 0.024 (2) | 0.0058 (19) | 0.0081 (17) | 0.0047 (19) |
| N1 | 0.059 (2) | 0.052 (2) | 0.0225 (18) | −0.0017 (19) | 0.0104 (16) | 0.0012 (16) |
| C2 | 0.054 (3) | 0.057 (3) | 0.037 (3) | 0.001 (2) | 0.005 (2) | −0.006 (2) |
| N2 | 0.062 (2) | 0.043 (2) | 0.0248 (18) | 0.0046 (18) | 0.0097 (16) | −0.0007 (16) |
| C3 | 0.052 (3) | 0.078 (4) | 0.031 (2) | 0.012 (3) | −0.002 (2) | −0.013 (3) |
| C4 | 0.064 (3) | 0.079 (4) | 0.024 (2) | 0.019 (3) | 0.007 (2) | 0.003 (2) |
| C5 | 0.071 (3) | 0.051 (3) | 0.034 (3) | 0.013 (2) | 0.016 (2) | 0.008 (2) |
| C6 | 0.046 (2) | 0.053 (3) | 0.031 (2) | 0.009 (2) | 0.0105 (18) | 0.002 (2) |
| C7 | 0.0324 (19) | 0.060 (3) | 0.0186 (19) | 0.013 (2) | 0.0016 (15) | −0.0124 (19) |
| C8 | 0.049 (2) | 0.041 (2) | 0.024 (2) | 0.007 (2) | 0.0071 (17) | −0.0004 (18) |
| C9 | 0.042 (2) | 0.036 (2) | 0.026 (2) | 0.0013 (18) | 0.0036 (17) | −0.0014 (18) |
| C10 | 0.057 (3) | 0.046 (3) | 0.036 (2) | 0.005 (2) | 0.005 (2) | −0.007 (2) |
| C11 | 0.047 (2) | 0.058 (3) | 0.029 (2) | 0.012 (2) | 0.0056 (19) | −0.011 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S—C8 | 1.723 (5) | C3—C4 | 1.363 (7) |
| S—C1 | 1.723 (4) | C3—H3B | 0.9300 |
| O2—C11 | 1.232 (5) | C4—C5 | 1.382 (7) |
| O1—C9 | 1.333 (5) | C4—H4A | 0.9300 |
| O1—C10 | 1.435 (5) | C5—C6 | 1.418 (6) |
| C1—C6 | 1.390 (6) | C5—H5A | 0.9300 |
| C1—C2 | 1.406 (6) | C6—C7 | 1.485 (6) |
| N1—C11 | 1.323 (6) | C7—C8 | 1.438 (6) |
| N1—N2 | 1.387 (5) | C7—H7A | 0.9300 |
| N1—H1A | 0.8600 | C8—C9 | 1.477 (5) |
| C2—C3 | 1.377 (6) | C10—C11 | 1.492 (6) |
| C2—H2B | 0.9300 | C10—H10A | 0.9700 |
| N2—C9 | 1.274 (5) | C10—H10B | 0.9700 |
| | | | |
| C8—S—C1 | 90.0 (2) | C1—C6—C5 | 118.9 (4) |
| C9—O1—C10 | 118.7 (3) | C1—C6—C7 | 115.1 (4) |

| | | | |
|---|---|---|---|
| C6—C1—C2 | 121.8 (4) | C5—C6—C7 | 125.9 (4) |
| C6—C1—S | 113.0 (3) | C8—C7—C6 | 104.4 (4) |
| C2—C1—S | 125.2 (4) | C8—C7—H7A | 127.8 |
| C11—N1—N2 | 125.5 (4) | C6—C7—H7A | 127.8 |
| C11—N1—H1A | 117.3 | C7—C8—C9 | 124.0 (4) |
| N2—N1—H1A | 117.3 | C7—C8—S | 117.5 (3) |
| C3—C2—C1 | 117.8 (5) | C9—C8—S | 118.6 (3) |
| C3—C2—H2B | 121.1 | N2—C9—O1 | 128.1 (4) |
| C1—C2—H2B | 121.1 | N2—C9—C8 | 118.8 (4) |
| C9—N2—N1 | 115.5 (4) | O1—C9—C8 | 113.0 (3) |
| C4—C3—C2 | 121.1 (5) | O1—C10—C11 | 114.6 (4) |
| C4—C3—H3B | 119.5 | O1—C10—H10A | 108.6 |
| C2—C3—H3B | 119.5 | C11—C10—H10A | 108.6 |
| C3—C4—C5 | 122.5 (4) | O1—C10—H10B | 108.6 |
| C3—C4—H4A | 118.8 | C11—C10—H10B | 108.6 |
| C5—C4—H4A | 118.8 | H10A—C10—H10B | 107.6 |
| C4—C5—C6 | 117.9 (5) | O2—C11—N1 | 123.6 (4) |
| C4—C5—H5A | 121.0 | O2—C11—C10 | 119.0 (4) |
| C6—C5—H5A | 121.0 | N1—C11—C10 | 117.4 (4) |

| | | | |
|---|---|---|---|
| C8—S—C1—C6 | 1.8 (3) | C6—C7—C8—S | 0.0 (4) |
| C8—S—C1—C2 | −179.4 (4) | C1—S—C8—C7 | −1.0 (3) |
| C6—C1—C2—C3 | −0.9 (6) | C1—S—C8—C9 | 177.3 (3) |
| S—C1—C2—C3 | −179.7 (3) | N1—N2—C9—O1 | −1.6 (6) |
| C11—N1—N2—C9 | 1.8 (6) | N1—N2—C9—C8 | −178.0 (3) |
| C1—C2—C3—C4 | 1.7 (7) | C10—O1—C9—N2 | 2.6 (7) |
| C2—C3—C4—C5 | −2.2 (7) | C10—O1—C9—C8 | 179.1 (4) |
| C3—C4—C5—C6 | 1.9 (7) | C7—C8—C9—N2 | 176.5 (4) |
| C2—C1—C6—C5 | 0.7 (6) | S—C8—C9—N2 | −1.7 (5) |
| S—C1—C6—C5 | 179.5 (3) | C7—C8—C9—O1 | −0.4 (6) |
| C2—C1—C6—C7 | 178.9 (4) | S—C8—C9—O1 | −178.6 (3) |
| S—C1—C6—C7 | −2.2 (4) | C9—O1—C10—C11 | −3.3 (6) |
| C4—C5—C6—C1 | −1.1 (6) | N2—N1—C11—O2 | 177.0 (4) |
| C4—C5—C6—C7 | −179.1 (4) | N2—N1—C11—C10 | −2.8 (6) |
| C1—C6—C7—C8 | 1.4 (4) | O1—C10—C11—O2 | −176.5 (4) |
| C5—C6—C7—C8 | 179.5 (4) | O1—C10—C11—N1 | 3.4 (6) |
| C6—C7—C8—C9 | −178.2 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···O2$^{i}$ | 0.86 | 2.03 | 2.848 (5) | 158 |

Symmetry code: (i) −*x*+3/2, *y*−1/2, −*z*+5/2.