Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 3-(6-Bromo-4-oxo-4*H*-chromen-3-yl)-3,4-dihydro-2*H*-1,2,4-benzothiadiazine-1,1-dione

**Mariya al-Rashida,[a]‡ Saeed Ahmad Nagra,[a] Islam Ullah Khan,[b] George Kostakis[c] and Ghulam Abbas[a]***

[a]Institute of Chemistry, University of the Punjab, Lahore, Pakistan, [b]Department of Chemistry, Government College University, Lahore, Pakistan, and [c]Institute of Inorganic Chemistry, Karlsruhe Institute of Technology, D-76133 Karlsruhe, Germany
Correspondence e-mail: abbas191@gmail.com

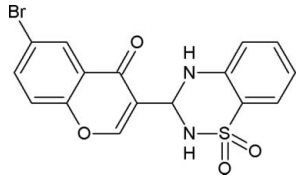
Received 15 October 2010; accepted 1 November 2010

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.046; $wR$ factor = 0.103; data-to-parameter ratio = 17.4.

The molecular structure of the title compound, $C_{16}H_{11}BrN_2O_4S$, is very similar to that of the previously reported fluoro analogue [al-Rashida *et al.* (2010). *Acta Cryst.* E**66**, o2707]. The mean planes of the bicyclic chromone system and the benzene ring of the benzothiadiazine derivative make a dihedral angle of 58.23 (8)°. An intramolecular N—H···O hydrogen bond occurs. In the crystal, molecules are linked into layers by N—H···O and C—H···O hydrogen bonds, generating an infinite two-dimensional network.

## Related literature

For background to the importance of the 1,2,4-benzothiadiazine-1,1-dioxide ring system in pharmaceutical and medicinal chemistry, see: Zhu *et al.* (2005); Kamal *et al.* (2007*a*). For a survey on the antimicrobial activity of benzothiadiazine derivatives, see: Di Bella *et al.* (1983); Kamal *et al.* (2007*a*,*b*). The sulfonamide group is an active pharmacophore, see: Weisman & Brown (1964). For related structures, see: al-Rashida *et al.* (2009, 2010).




## Experimental

### Crystal data

$C_{16}H_{11}BrN_2O_4S$ $M_r$ = 407.24
Monoclinic, $P2_1/n$
$a$ = 7.0778 (4) Å
$b$ = 8.6070 (6) Å
$c$ = 25.6290 (16) Å
$\beta$ = 94.607 (3)°
$V$ = 1556.24 (17) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 2.80 mm$^{-1}$
$T$ = 296 K
0.28 × 0.28 × 0.22 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min}$ = 0.475, $T_{max}$ = 0.540
17309 measured reflections
3873 independent reflections
1969 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.058

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.103
$S$ = 0.98
3873 reflections
223 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.50 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.51 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N4—H4*A*···O4 | 0.79 (3) | 2.59 (3) | 2.987 (3) | 113 (3) |
| N4—H4*A*···O3$^{i}$ | 0.79 (3) | 2.23 (3) | 2.999 (3) | 164 (3) |
| C13—H13···O3$^{i}$ | 0.93 | 2.54 (1) | 3.314 (4) | 141 (1) |
| N2—H2*A*···O2 | 0.84 (2) | 2.67 (3) | 3.222 (4) | 124 (2) |
| C2—H2···O2 | 0.93 | 2.41 (1) | 3.330 (4) | 169 (1) |
| N2—H2*A*···O4 | 0.84 (2) | 2.12 (3) | 2.903 (4) | 154 (3) |

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

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 for Windows* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors acknowledge the Higher Education Commission (HEC), Islamabad, Pakistan, for financial and GCU, Lahore, for technical suppport.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2069).

## References

al-Rashida, M., Nagra, S. A., Khan, I. U., Kostakis, G. & Abbas, G. (2010). *Acta Cryst.* E**66**, o2707.
al-Rashida, M., Tahir, M. N., Nagra, S. A., Imran, M. & Iqbal, J. (2009). *Acta Cryst.* E**65**, o1818–o1819.
Bruker (2007). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Di Bella, M., Monzani, A., Andrisano, M. G., Fabio, U. & Quaglio, G. P. (1983). *Farmaco*, **38**, 466–472.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Kamal, A., Ahmed, S. K., Reddy, K. S., Khan, M. N. A., Shetty, R. V. C. R. N. C., Siddhardha, B., Murty, U. S. N., China, A. & Nagaraja, V. (2007*a*). *Lett. Drug Des. Discov.* **4**, 550–556.

‡ Additional corresponding author, e-mail: maria_al_rashida@hotmail.com.

Kamal, A., Khan, M. N. A., Reddy, K. S., Rohini, K., Sastry, G. N., Sateesh, B. & Sridhar, B. (2007*b*). *Bioorg. Med. Chem. Lett.* **17**, 5400–5405.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Weisman, R. A. & Brown, G. M. (1964). *J. Biol. Chem.* **239**, 326–331.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Zhu, Z., Zhu, S., Liu, D., Cao, T., Wang, L. & Tepel, M. (2005). *Hypertension*, **45**, 233–239.

# supporting information

*Acta Cryst.* (2010). E**66**, o3081–o3082 [https://doi.org/10.1107/S1600536810044648]

## 3-(6-Bromo-4-oxo-4*H*-chromen-3-yl)-3,4-dihydro-2*H*-1,2,4-benzothiadiazine-1,1-dione

**Mariya al-Rashida, Saeed Ahmad Nagra, Islam Ullah Khan, George Kostakis and Ghulam Abbas**

### S1. Comment

The 1,2,4-benzothiadiazine-1,1-dioxide ring system is of considerable importance in medicinal and pharmaceutical chemistry (Zhu *et al.*, 2005; Kamal *et al.*, 2007*a*). Novel products from reactions of 4- and 2-aminobenzenesulfonamide with 6-(un)substituted-4-oxo-4*H*-1-benzopyran-3-carboxaldehyde have already been reported by us (Mariya-al-Rashida *et al.*, 2009, 2010). In continuation of our project, the crystal structure of the title compound is reported here (Fig. 1).

In the crystal structure, the two rings of the chromone system (Br1, O1, O4, C2—C10) are coplanar making a dihedral angle of 1.0 (2)°. The carbon atom C11 deviates from the mean plane of the chromone ring by 0.016 (4) Å. The phenyl ring (C12—C17) of the benzothiadiazine moiety and the atoms S1, N4 and C11 are almost planar as well (rms deviation = 0.007) and make a dihedral angle of 58.23 (8)° with the mean plane of the bicyclic chromone system. The crystal structure is stabilized by intra- and intermolecular N—H···O and C—H···O hydrogen bonds which link the molecules into an infinite two-dimensional network (Fig. 2).

### S2. Experimental

A solution of 2-aminobenzenesulfonamide (1.0 mmol) in 10 ml e thanol was slowly added to the stirred solution of 6-bromo-4-oxo-4*H*-1-benzopyran-3-carboxaldehyde (1.0 mmol) containing catalytic amount of *p*-toluene sulfonic acid (*p*-TsOH) and refluxed for 3 hrs. The resulting product was isolated by filtration, washed with ethanol, dried and recrystallized from hot ethanol and acetone (1:1) (yield 77%, m.p. 496 K).

### S3. Refinement

The H atoms attached to N were located in a difference Fourier map and their coordinates were refined, with $U_{iso}$(H) = 1.2$U_{eq}$(N). The remaining H atoms were positioned geometrically with C-H = 0.93 and 0.98 Å for aromatic and methine H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}$(H) = 1.2$U_{eq}$(C).

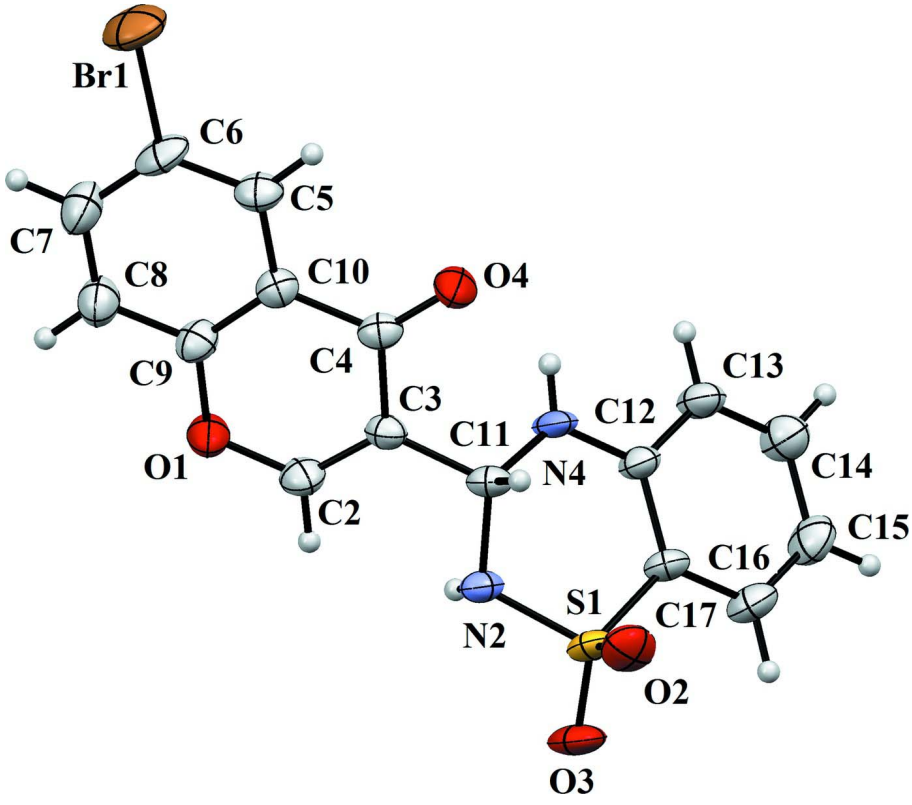



**Figure 1**

The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

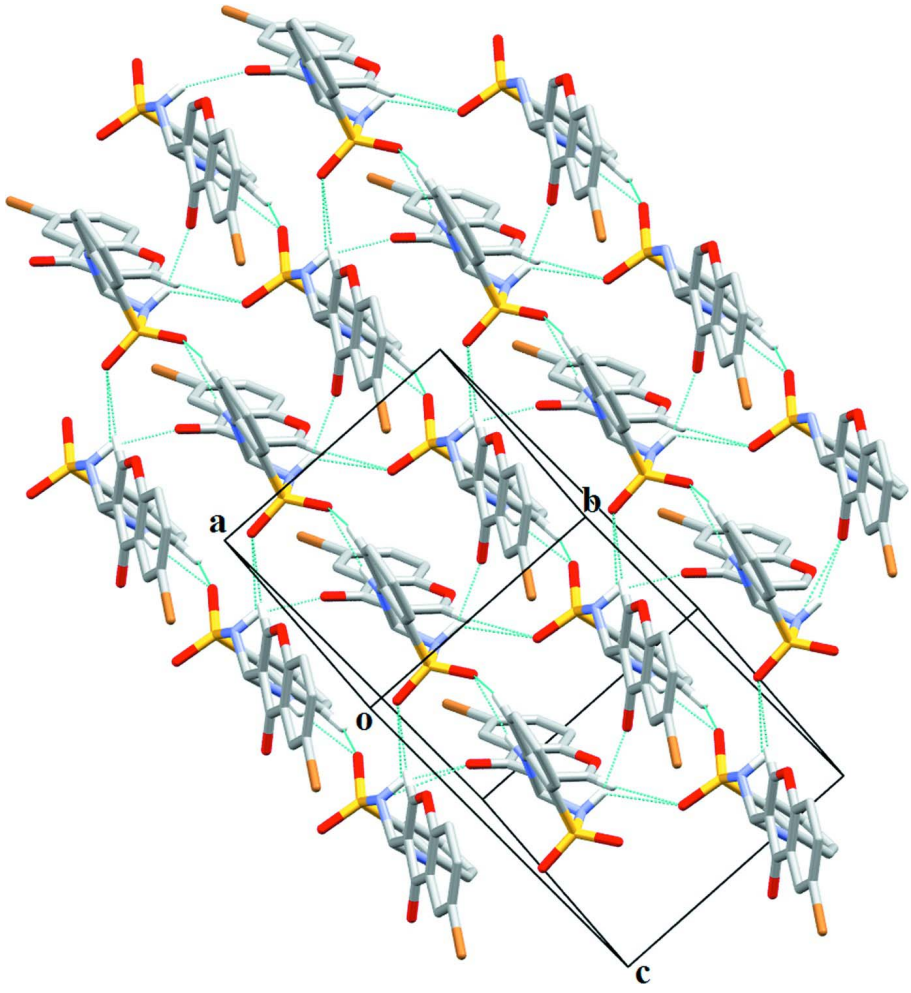



**Figure 2**

A packing diagram of the title compound showing hydrogen bonds as dashed lines.

**3-(6-Bromo-4-oxo-4*H*-chromen-3-yl)-3,4-dihydro-2*H*-1,2,4- benzothiadiazine-1,1-dione**

*Crystal data*

$C_{16}H_{11}BrN_2O_4S$
$M_r = 407.24$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.0778$ (4) Å
$b = 8.6070$ (6) Å
$c = 25.6290$ (16) Å
$\beta = 94.607$ (3)°
$V = 1556.24$ (17) Å$^3$
$Z = 4$
$F(000) = 816$
$D_x = 1.738$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2932 reflections
$\theta = 3.1$–$22.1$°
$\mu = 2.80$ mm$^{-1}$
$T = 296$ K
Needle, white
0.28 × 0.28 × 0.22 mm

*Data collection*

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min} = 0.475$, $T_{max} = 0.540$

17309 measured reflections
3873 independent reflections
1969 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 28.4°$, $\theta_{min} = 1.6°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 8$
$l = -34 \rightarrow 34$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.103$
$S = 0.98$
3873 reflections
223 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
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.1442P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.50$ e Å$^{-3}$
$\Delta\rho_{min} = -0.51$ 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 | 1.22103 (10) | 0.73566 (10) | 0.31055 (3) | 0.0337 (2) |
| O2 | 1.2394 (3) | 0.5717 (3) | 0.30708 (9) | 0.0463 (6) |
| O3 | 1.3885 (3) | 0.8294 (3) | 0.31425 (9) | 0.0465 (6) |
| N2 | 1.0884 (3) | 0.7953 (3) | 0.25943 (10) | 0.0287 (6) |
| H2A | 1.085 (4) | 0.893 (3) | 0.2592 (12) | 0.034* |
| N4 | 0.8018 (3) | 0.7790 (4) | 0.30189 (10) | 0.0371 (7) |
| H4A | 0.690 (4) | 0.780 (4) | 0.3005 (13) | 0.045* |
| Br1 | −0.00741 (5) | 0.67254 (5) | 0.060030 (16) | 0.06277 (19) |
| C5 | 0.3194 (4) | 0.7029 (4) | 0.13029 (13) | 0.0350 (8) |
| H5 | 0.2622 | 0.6278 | 0.1498 | 0.042* |
| C6 | 0.2305 (5) | 0.7577 (4) | 0.08503 (13) | 0.0403 (9) |
| C7 | 0.3086 (5) | 0.8725 (4) | 0.05590 (14) | 0.0455 (9) |
| H7 | 0.2429 | 0.9099 | 0.0256 | 0.055* |
| C8 | 0.4824 (5) | 0.9304 (4) | 0.07182 (13) | 0.0424 (9) |
| H8 | 0.5374 | 1.0068 | 0.0523 | 0.051* |
| C9 | 0.5766 (4) | 0.8743 (4) | 0.11749 (12) | 0.0324 (8) |
| O1 | 0.7509 (3) | 0.9378 (3) | 0.13110 (8) | 0.0398 (6) |
| C10 | 0.4984 (4) | 0.7614 (4) | 0.14695 (12) | 0.0287 (7) |
| C4 | 0.6000 (4) | 0.7086 (4) | 0.19593 (12) | 0.0286 (8) |
| O4 | 0.5369 (3) | 0.6116 (3) | 0.22456 (9) | 0.0395 (6) |

| | | | | |
|---|---|---|---|---|
| C3 | 0.7845 (4) | 0.7819 (4) | 0.20758 (12) | 0.0272 (7) |
| C2 | 0.8469 (4) | 0.8871 (4) | 0.17524 (12) | 0.0347 (8) |
| H2 | 0.9665 | 0.9291 | 0.1838 | 0.042* |
| C11 | 0.8977 (4) | 0.7310 (4) | 0.25680 (12) | 0.0289 (7) |
| H11 | 0.9066 | 0.6174 | 0.2568 | 0.035* |
| C12 | 0.8888 (4) | 0.7966 (4) | 0.35104 (12) | 0.0295 (8) |
| C13 | 0.7836 (4) | 0.8366 (4) | 0.39290 (13) | 0.0372 (8) |
| H13 | 0.6531 | 0.8494 | 0.3871 | 0.045* |
| C14 | 0.8682 (5) | 0.8573 (4) | 0.44190 (14) | 0.0431 (9) |
| H14 | 0.7942 | 0.8825 | 0.4691 | 0.052* |
| C16 | 1.1691 (5) | 0.8056 (4) | 0.41215 (13) | 0.0402 (9) |
| H16 | 1.2999 | 0.7964 | 0.4183 | 0.048* |
| C15 | 1.0631 (5) | 0.8417 (4) | 0.45229 (14) | 0.0445 (9) |
| H15 | 1.1196 | 0.8557 | 0.4860 | 0.053* |
| C17 | 1.0843 (4) | 0.7823 (4) | 0.36172 (12) | 0.0291 (8) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0158 (4) | 0.0421 (6) | 0.0427 (5) | 0.0059 (4) | −0.0002 (4) | −0.0004 (4) |
| O2 | 0.0393 (14) | 0.0381 (15) | 0.0622 (17) | 0.0148 (11) | 0.0081 (12) | 0.0055 (12) |
| O3 | 0.0160 (11) | 0.0624 (17) | 0.0608 (16) | −0.0034 (11) | 0.0001 (11) | −0.0038 (13) |
| N2 | 0.0172 (13) | 0.0291 (15) | 0.0393 (16) | −0.0005 (12) | 0.0005 (12) | 0.0016 (14) |
| N4 | 0.0121 (12) | 0.070 (2) | 0.0293 (16) | 0.0004 (14) | 0.0002 (13) | −0.0041 (14) |
| Br1 | 0.0358 (2) | 0.0836 (4) | 0.0653 (3) | −0.0100 (2) | −0.01830 (19) | −0.0107 (2) |
| C5 | 0.0287 (17) | 0.035 (2) | 0.040 (2) | −0.0072 (15) | −0.0010 (16) | −0.0056 (17) |
| C6 | 0.0290 (19) | 0.049 (2) | 0.040 (2) | 0.0009 (17) | −0.0140 (16) | −0.0148 (18) |
| C7 | 0.051 (2) | 0.046 (2) | 0.037 (2) | −0.0006 (19) | −0.0116 (18) | 0.0050 (18) |
| C8 | 0.047 (2) | 0.042 (2) | 0.036 (2) | −0.0048 (18) | −0.0029 (18) | 0.0077 (17) |
| C9 | 0.0332 (18) | 0.032 (2) | 0.0313 (19) | −0.0038 (15) | −0.0044 (15) | −0.0040 (16) |
| O1 | 0.0367 (13) | 0.0447 (15) | 0.0367 (14) | −0.0182 (11) | −0.0056 (11) | 0.0071 (11) |
| C10 | 0.0283 (17) | 0.0265 (19) | 0.0309 (18) | −0.0002 (14) | 0.0009 (15) | −0.0027 (15) |
| C4 | 0.0250 (17) | 0.0269 (19) | 0.0338 (19) | −0.0007 (14) | 0.0014 (15) | −0.0058 (16) |
| O4 | 0.0347 (13) | 0.0426 (14) | 0.0406 (14) | −0.0153 (11) | −0.0015 (11) | 0.0104 (12) |
| C3 | 0.0219 (16) | 0.0315 (19) | 0.0286 (17) | −0.0038 (14) | 0.0034 (14) | −0.0041 (15) |
| C2 | 0.0283 (18) | 0.040 (2) | 0.035 (2) | −0.0090 (16) | −0.0022 (16) | −0.0028 (17) |
| C11 | 0.0171 (15) | 0.0334 (19) | 0.0361 (19) | −0.0019 (14) | 0.0027 (14) | −0.0035 (15) |
| C12 | 0.0190 (15) | 0.038 (2) | 0.0303 (18) | 0.0018 (14) | −0.0024 (14) | −0.0023 (15) |
| C13 | 0.0213 (16) | 0.052 (2) | 0.038 (2) | 0.0039 (16) | 0.0010 (15) | −0.0052 (18) |
| C14 | 0.039 (2) | 0.051 (2) | 0.039 (2) | 0.0055 (17) | 0.0017 (17) | −0.0085 (18) |
| C16 | 0.0257 (18) | 0.048 (2) | 0.044 (2) | 0.0029 (16) | −0.0094 (17) | −0.0032 (18) |
| C15 | 0.044 (2) | 0.054 (3) | 0.033 (2) | 0.0006 (18) | −0.0092 (17) | −0.0068 (18) |
| C17 | 0.0167 (15) | 0.0338 (19) | 0.0363 (19) | 0.0029 (13) | −0.0010 (14) | −0.0017 (15) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O2 | 1.421 (2) | C9—C10 | 1.375 (4) |
| S1—O3 | 1.431 (2) | O1—C2 | 1.345 (4) |

| | | | |
|---|---|---|---|
| S1—N2 | 1.632 (3) | C10—C4 | 1.468 (4) |
| S1—C17 | 1.738 (3) | C4—O4 | 1.219 (3) |
| N2—C11 | 1.456 (4) | C4—C3 | 1.459 (4) |
| N2—H2A | 0.84 (2) | C3—C2 | 1.327 (4) |
| N4—C12 | 1.365 (4) | C3—C11 | 1.504 (4) |
| N4—C11 | 1.446 (4) | C2—H2 | 0.9300 |
| N4—H4A | 0.79 (3) | C11—H11 | 0.9800 |
| Br1—C6 | 1.900 (3) | C12—C13 | 1.398 (4) |
| C5—C6 | 1.359 (4) | C12—C17 | 1.394 (4) |
| C5—C10 | 1.398 (4) | C13—C14 | 1.359 (4) |
| C5—H5 | 0.9300 | C13—H13 | 0.9300 |
| C6—C7 | 1.380 (5) | C14—C15 | 1.391 (5) |
| C7—C8 | 1.359 (5) | C14—H14 | 0.9300 |
| C7—H7 | 0.9300 | C16—C15 | 1.357 (5) |
| C8—C9 | 1.387 (4) | C16—C17 | 1.395 (4) |
| C8—H8 | 0.9300 | C16—H16 | 0.9300 |
| C9—O1 | 1.368 (4) | C15—H15 | 0.9300 |

| | | | |
|---|---|---|---|
| O2—S1—O3 | 119.01 (14) | O4—C4—C10 | 123.3 (3) |
| O2—S1—N2 | 108.16 (14) | C3—C4—C10 | 114.2 (3) |
| O3—S1—N2 | 107.20 (14) | C2—C3—C4 | 120.3 (3) |
| O2—S1—C17 | 109.63 (15) | C2—C3—C11 | 122.8 (3) |
| O3—S1—C17 | 109.16 (14) | C4—C3—C11 | 117.0 (3) |
| N2—S1—C17 | 102.36 (14) | C3—C2—O1 | 125.2 (3) |
| C11—N2—S1 | 112.9 (2) | C3—C2—H2 | 117.4 |
| C11—N2—H2A | 111 (2) | O1—C2—H2 | 117.4 |
| S1—N2—H2A | 109 (2) | N4—C11—N2 | 110.3 (2) |
| C12—N4—C11 | 124.3 (2) | N4—C11—C3 | 109.6 (2) |
| C12—N4—H4A | 115 (3) | N2—C11—C3 | 111.0 (3) |
| C11—N4—H4A | 120 (3) | N4—C11—H11 | 108.6 |
| C6—C5—C10 | 118.8 (3) | N2—C11—H11 | 108.6 |
| C6—C5—H5 | 120.6 | C3—C11—H11 | 108.6 |
| C10—C5—H5 | 120.6 | N4—C12—C13 | 120.5 (3) |
| C5—C6—C7 | 122.2 (3) | N4—C12—C17 | 122.6 (3) |
| C5—C6—Br1 | 119.4 (3) | C13—C12—C17 | 116.9 (3) |
| C7—C6—Br1 | 118.5 (2) | C14—C13—C12 | 121.3 (3) |
| C8—C7—C6 | 119.5 (3) | C14—C13—H13 | 119.4 |
| C8—C7—H7 | 120.3 | C12—C13—H13 | 119.4 |
| C6—C7—H7 | 120.3 | C13—C14—C15 | 121.4 (3) |
| C7—C8—C9 | 119.2 (3) | C13—C14—H14 | 119.3 |
| C7—C8—H8 | 120.4 | C15—C14—H14 | 119.3 |
| C9—C8—H8 | 120.4 | C15—C16—C17 | 120.8 (3) |
| O1—C9—C10 | 122.5 (3) | C15—C16—H16 | 119.6 |
| O1—C9—C8 | 116.0 (3) | C17—C16—H16 | 119.6 |
| C10—C9—C8 | 121.5 (3) | C16—C15—C14 | 118.5 (3) |
| C2—O1—C9 | 118.0 (2) | C16—C15—H15 | 120.7 |
| C9—C10—C5 | 118.8 (3) | C14—C15—H15 | 120.7 |
| C9—C10—C4 | 119.8 (3) | C16—C17—C12 | 121.0 (3) |

| | | | |
|---|---|---|---|
| C5—C10—C4 | 121.3 (3) | C16—C17—S1 | 120.5 (2) |
| O4—C4—C3 | 122.5 (3) | C12—C17—S1 | 118.4 (2) |
| | | | |
| O2—S1—N2—C11 | 61.9 (2) | C9—O1—C2—C3 | −1.5 (5) |
| O3—S1—N2—C11 | −168.6 (2) | C12—N4—C11—N2 | −35.8 (4) |
| C17—S1—N2—C11 | −53.8 (2) | C12—N4—C11—C3 | −158.3 (3) |
| C10—C5—C6—C7 | −2.0 (5) | S1—N2—C11—N4 | 61.6 (3) |
| C10—C5—C6—Br1 | 177.0 (2) | S1—N2—C11—C3 | −176.8 (2) |
| C5—C6—C7—C8 | 1.9 (5) | C2—C3—C11—N4 | 114.2 (3) |
| Br1—C6—C7—C8 | −177.2 (3) | C4—C3—C11—N4 | −66.5 (3) |
| C6—C7—C8—C9 | −0.9 (5) | C2—C3—C11—N2 | −7.9 (4) |
| C7—C8—C9—O1 | 179.9 (3) | C4—C3—C11—N2 | 171.4 (2) |
| C7—C8—C9—C10 | 0.1 (5) | C11—N4—C12—C13 | −177.1 (3) |
| C10—C9—O1—C2 | −0.4 (4) | C11—N4—C12—C17 | 5.6 (5) |
| C8—C9—O1—C2 | 179.7 (3) | N4—C12—C13—C14 | −178.8 (3) |
| O1—C9—C10—C5 | 179.9 (3) | C17—C12—C13—C14 | −1.4 (5) |
| C8—C9—C10—C5 | −0.2 (5) | C12—C13—C14—C15 | 0.9 (5) |
| O1—C9—C10—C4 | 2.1 (5) | C17—C16—C15—C14 | −1.0 (5) |
| C8—C9—C10—C4 | −178.0 (3) | C13—C14—C15—C16 | 0.3 (5) |
| C6—C5—C10—C9 | 1.2 (5) | C15—C16—C17—C12 | 0.5 (5) |
| C6—C5—C10—C4 | 179.0 (3) | C15—C16—C17—S1 | −179.9 (3) |
| C9—C10—C4—O4 | 178.2 (3) | N4—C12—C17—C16 | 178.0 (3) |
| C5—C10—C4—O4 | 0.5 (5) | C13—C12—C17—C16 | 0.6 (5) |
| C9—C10—C4—C3 | −2.0 (4) | N4—C12—C17—S1 | −1.6 (4) |
| C5—C10—C4—C3 | −179.7 (3) | C13—C12—C17—S1 | −179.0 (2) |
| O4—C4—C3—C2 | −179.9 (3) | O2—S1—C17—C16 | 89.7 (3) |
| C10—C4—C3—C2 | 0.3 (4) | O3—S1—C17—C16 | −42.3 (3) |
| O4—C4—C3—C11 | 0.8 (4) | N2—S1—C17—C16 | −155.7 (3) |
| C10—C4—C3—C11 | −179.0 (3) | O2—S1—C17—C12 | −90.7 (3) |
| C4—C3—C2—O1 | 1.4 (5) | O3—S1—C17—C12 | 137.3 (3) |
| C11—C3—C2—O1 | −179.3 (3) | N2—S1—C17—C12 | 24.0 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N4—H4*A*···O4 | 0.79 (3) | 2.59 (3) | 2.987 (3) | 113 (3) |
| N4—H4*A*···O3$^{i}$ | 0.79 (3) | 2.23 (3) | 2.999 (3) | 164 (3) |
| C13—H13···O3$^{i}$ | 0.93 | 2.54 (1) | 3.314 (4) | 141 (1) |
| N2—H2*A*···O2 | 0.84 (2) | 2.67 (3) | 3.222 (4) | 124 (2) |
| C2—H2···O2 | 0.93 | 2.41 (1) | 3.330 (4) | 169 (1) |
| N2—H2*A*···O4 | 0.84 (2) | 2.12 (3) | 2.903 (4) | 154 (3) |

Symmetry code: (i) *x*−1, *y*, *z*.