

N-(2,3-Dimethylphenyl)-4-hydroxy-2-methyl-2H-1,2-benzothiazine-3-carboxamide 1,1-dioxide

Waseeq Ahmad Siddiqui,^{a*} Iftikhar Hussain Bukahari,^a Muhammad Zia-ur-Rehman,^b Islam Ullah Khan^c and Graham John Tizzard^d

^aDepartment of Chemistry, University of Sargodha, Sargodha, Pakistan, ^bApplied Chemistry Research Centre, PCSIR Laboratories Complex, Ferozpur Road, Lahore 54600, Pakistan, ^cDepartment of Chemistry, Government College University, Lahore 54000, Pakistan, and ^dSchool of Chemistry, University of Southampton, England
Correspondence e-mail: waseeqsiddiqui@gmail.com

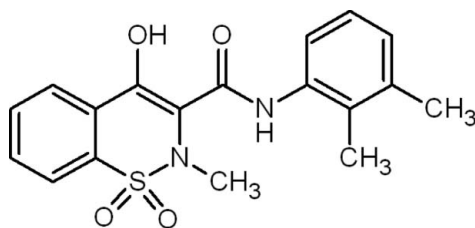
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.045; wR factor = 0.114; data-to-parameter ratio = 16.4.

In the crystal structure of the title compound, $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_4\text{S}$, the thiazine ring adopts a distorted half-chair conformation. 1,2-Benzothiazines of this kind have a wide range of biological activities and are mainly used as medicines in the treatment of inflammation and rheumatoid arthritis. The enolic H atom is involved in an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond, forming a six-membered ring. The molecules arrange themselves into centrosymmetric dimers by means of intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. A weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interaction is also present.

Related literature

For the synthesis of related molecules, see: Siddiqui, Ahmad, Khan *et al.* (2007); Zia-ur-Rehman *et al.* (2006); For the biological activity of 1,2-benzothiazine-1,1-dioxides, see: Zia-ur-Rehman *et al.* (2009). For related structures, see: Siddiqui *et al.* (2008); Siddiqui, Ahmad, Siddiqui *et al.* (2007). For the pharmacological background to 1,2-benzothiazine-3-carboxamide 1,1-dioxide derivatives, see Gennari *et al.* (1994); Lombardino & Wiseman (1972).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_4\text{S}$
 $M_r = 358.40$
 Monoclinic, $P2_1/n$
 $a = 10.2461$ (3) Å
 $b = 8.5421$ (2) Å
 $c = 19.8944$ (5) Å
 $\beta = 104.832$ (1)°
 $V = 1683.20$ (8) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.22$ mm⁻¹
 $T = 120$ K
 $0.30 \times 0.10 \times 0.10$ mm

Data collection

Bruker–Nonius CCD camera on κ -goniostat diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2007)
 $T_{\min} = 0.855$, $T_{\max} = 0.978$
 18371 measured reflections
 3828 independent reflections
 3000 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.055$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.114$
 $S = 1.06$
 3828 reflections
 234 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.27$ e Å⁻³
 $\Delta\rho_{\min} = -0.57$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|----------|-------------|-------------|---------------|
| $\text{O1}-\text{H1O}\cdots\text{O2}$ | 0.84 | 1.79 | 2.5320 (18) | 147 |
| $\text{N2}-\text{H1N}\cdots\text{O3}^i$ | 0.85 (2) | 2.26 (2) | 2.972 (2) | 141 (2) |
| $\text{C3}-\text{H3}\cdots\text{O4}^{ii}$ | 0.95 | 2.49 | 3.352 (3) | 150 |

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

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL and local programs.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2887).

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supplementary materials

Acta Cryst. (2009). E65, o644-o645 [doi:10.1107/S1600536809006837]

N-(2,3-Dimethylphenyl)-4-hydroxy-2-methyl-2*H*-1,2-benzothiazine-3-carboxamide 1,1-dioxide

W. A. Siddiqui, I. H. Bukahari, M. Zia-ur-Rehman, I. U. Khan and G. J. Tizzard

Comment

1,2-Benzothiazine-3-carboxamide 1,1-dioxide derivatives belonging to oxicams, a class of non-steroidal anti-inflammatory drugs (NSAIDs), are well known as analgesic and anti-inflammatory agents since the introduction of Piroxicam (Lombardino & Wiseman, 1972) in the United States in 1982 where it gained immediate acceptance and remained among the top fifty prescription drugs for several years. Besides having anti-inflammatory activity, these have also been found to be used for the treatment of rheumatoid arthritis, ankylosing spondylitis, osteoarthritis and other inflammatory rheumatic and non-rheumatic processes, including onsets and traumatologic lesions (Gennari *et al.*, 1994).

In continuation of our work on the synthesis (Siddiqui, Ahmad, Khan *et al.*, 2007, Zia-ur-Rehman *et al.*, 2006, biological activity (Zia-ur-Rehman *et al.*, 2009) and crystal structures (Siddiqui, Ahmad, Siddiqui *et al.*, 2007, Siddiqui *et al.*, 2008) of various 1,2-benzothiazine-1,1-dioxides, we herein report the synthesis and crystal structure of the title compound (**I**) (Scheme and figure 1). The thiazine ring, involving two double bonds, exhibits a distorted half-chair conformation. The enolic hydrogen on O1 is involved in intramolecular hydrogen bonding giving rise to a six-membered hydrogen bond ring (Table 1). The molecules form centrosymmetric dimers through intermolecular N—H \cdots O hydrogen bonds. In addition, the crystal packing is stabilized by weak C—H \cdots O contacts.

Experimental

A mixture of methyl 4-hydroxy-2-methyl-2*H*-1,2-benzothiazine-3-carboxylate-1,1-dioxide (2.693 g; 10.0 mmoles), 2,3-dimethyl aniline (1.818 g; 15.0 mmoles) and xylene (25.0 ml) was refluxed under nitrogen atmosphere in a Soxhlet apparatus having Linde type 4Å molecular sieves. Three fourth of the xylene was then distilled off and the remaining contents were allowed to stand overnight at room temperature. Settled solids were filtered off, washed with diethyl ether and crystallized from ethanol. Yield: 78%.

Refinement

All hydrogen atoms were identified in the difference map. Those bonded to O and C were fixed in ideal positions and treated as riding on their parent atoms. In the case of the methyl and hydroxyl H atoms the torsion angles were refined. The following distances were used: Methyl C—H 0.98 Å. ° Aromatic C—H 0.95 Å. ° Hydroxyl O—H 0.84 Å. U(H) was set to 1.2Ueq of the parent atoms or 1.5Ueq for methyl groups. The H atom bonded to N was freely refined.

Figures

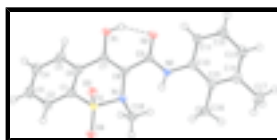


Fig. 1. The molecular structure of the title compound with displacement ellipsoids at the 50% probability level.

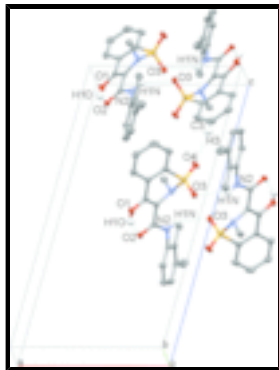


Fig. 2. Perspective view of the three-dimensional crystal packing showing hydrogen-bonded interactions (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity.

***N*-(2,3-Dimethylphenyl)-4-hydroxy-2-methyl-2*H*-1,2-benzothiazine-3-carboxamide 1,1-dioxide**

Crystal data

$C_{18}H_{18}N_2O_4S$

$M_r = 358.40$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2yn$

$a = 10.2461\ (3)\ \text{\AA}$

$b = 8.5421\ (2)\ \text{\AA}$

$c = 19.8944\ (5)\ \text{\AA}$

$\beta = 104.8320\ (10)^\circ$

$V = 1683.20\ (8)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 752$

$D_x = 1.414\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 10340 reflections

$\theta = 2.9\text{--}27.5^\circ$

$\mu = 0.22\ \text{mm}^{-1}$

$T = 120\ \text{K}$

Block, colourless

$0.30 \times 0.10 \times 0.10\ \text{mm}$

Data collection

Bruker–Nonius CCD camera on κ -goniostat diffractometer

3828 independent reflections

Radiation source: Bruker Nonius FR591 Rotating Anode

3000 reflections with $I > 2\sigma(I)$

Monochromator: graphite

$R_{\text{int}} = 0.055$

Detector resolution: $9.091\ \text{pixels mm}^{-1}$

$\theta_{\text{max}} = 27.5^\circ$

$T = 120\ \text{K}$

$\theta_{\text{min}} = 3.2^\circ$

φ and ω scans to fill the asymmetric unit

$h = -13 \rightarrow 11$

Absorption correction: multi-scan (SADABS; Sheldrick, 2007)

$k = -11 \rightarrow 9$

$T_{\text{min}} = 0.855$, $T_{\text{max}} = 0.978$

$l = -25 \rightarrow 24$

18371 measured reflections

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

$$R[F^2 > 2\sigma(F^2)] = 0.045$$

$$wR(F^2) = 0.114$$

$$S = 1.06$$

3828 reflections

234 parameters

Primary atom site location: structure-invariant direct methods

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.8901P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.57 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Special details

Experimental. *SADABS* was used to perform the Absorption correction Parameter refinement on 11612 reflections reduced $R(\text{int})$ from 0.0681 to 0.0328 Ratio of minimum to maximum apparent transmission: 0.867291 The given Tmin and Tmax were generated using the *SHELX SIZE* command

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|--------------|----------------------------------|
| S1 | 0.65769 (5) | 0.38059 (5) | 0.08727 (2) | 0.01787 (14) |
| O2 | 0.85778 (13) | 0.51574 (15) | -0.10128 (6) | 0.0190 (3) |
| O1 | 0.93907 (13) | 0.27205 (16) | -0.03046 (7) | 0.0208 (3) |
| H1O | 0.9310 | 0.3360 | -0.0633 | 0.031* |
| N2 | 0.70234 (16) | 0.67387 (18) | -0.06965 (8) | 0.0162 (3) |
| N1 | 0.73387 (15) | 0.51959 (17) | 0.05471 (8) | 0.0155 (3) |
| O3 | 0.55860 (13) | 0.31248 (15) | 0.03050 (7) | 0.0207 (3) |
| O4 | 0.61656 (14) | 0.44317 (17) | 0.14534 (7) | 0.0255 (3) |
| C6 | 0.88260 (18) | 0.2273 (2) | 0.07631 (9) | 0.0173 (4) |
| C9 | 0.78894 (18) | 0.5530 (2) | -0.05975 (9) | 0.0154 (4) |
| C10 | 0.67298 (17) | 0.7818 (2) | -0.12596 (9) | 0.0166 (4) |
| C7 | 0.87323 (18) | 0.3265 (2) | 0.01508 (9) | 0.0162 (4) |
| C1 | 0.78842 (19) | 0.2430 (2) | 0.11619 (9) | 0.0187 (4) |
| C18 | 0.8113 (2) | 0.6356 (2) | 0.10495 (10) | 0.0226 (4) |
| H18A | 0.8911 | 0.5851 | 0.1348 | 0.034* |
| H18B | 0.7542 | 0.6764 | 0.1336 | 0.034* |
| H18C | 0.8397 | 0.7220 | 0.0795 | 0.034* |
| C5 | 0.98373 (19) | 0.1138 (2) | 0.09600 (10) | 0.0216 (4) |
| H5 | 1.0480 | 0.1000 | 0.0695 | 0.026* |
| C8 | 0.80079 (18) | 0.4615 (2) | 0.00417 (9) | 0.0151 (4) |

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| | | | | |
|------|--------------|------------|---------------|------------|
| C14 | 0.61045 (19) | 1.0447 (2) | -0.16562 (11) | 0.0226 (4) |
| C16 | 0.6568 (2) | 0.9831 (2) | -0.03662 (10) | 0.0233 (4) |
| H16A | 0.5896 | 0.9260 | -0.0190 | 0.035* |
| H16B | 0.6410 | 1.0959 | -0.0344 | 0.035* |
| H16C | 0.7475 | 0.9578 | -0.0082 | 0.035* |
| C4 | 0.9901 (2) | 0.0215 (2) | 0.15404 (11) | 0.0266 (5) |
| H4 | 1.0594 | -0.0549 | 0.1672 | 0.032* |
| C15 | 0.64525 (18) | 0.9363 (2) | -0.11082 (10) | 0.0181 (4) |
| C12 | 0.6398 (2) | 0.8437 (2) | -0.24637 (10) | 0.0246 (4) |
| H12 | 0.6402 | 0.8140 | -0.2923 | 0.030* |
| C11 | 0.67017 (19) | 0.7340 (2) | -0.19320 (9) | 0.0203 (4) |
| H11 | 0.6887 | 0.6283 | -0.2025 | 0.024* |
| C3 | 0.8966 (2) | 0.0393 (2) | 0.19319 (11) | 0.0294 (5) |
| H3 | 0.9024 | -0.0246 | 0.2329 | 0.035* |
| C13 | 0.6091 (2) | 0.9960 (2) | -0.23262 (10) | 0.0248 (4) |
| H13 | 0.5865 | 1.0692 | -0.2697 | 0.030* |
| C2 | 0.7944 (2) | 0.1507 (2) | 0.17439 (10) | 0.0258 (4) |
| H2 | 0.7299 | 0.1633 | 0.2009 | 0.031* |
| C17 | 0.5769 (2) | 1.2125 (3) | -0.15299 (12) | 0.0350 (5) |
| H17A | 0.5529 | 1.2691 | -0.1973 | 0.052* |
| H17B | 0.6555 | 1.2624 | -0.1217 | 0.052* |
| H17C | 0.5006 | 1.2151 | -0.1317 | 0.052* |
| H1N | 0.657 (2) | 0.683 (3) | -0.0397 (12) | 0.026 (6)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|---------------|--------------|---------------|
| S1 | 0.0187 (2) | 0.0193 (3) | 0.0181 (2) | -0.00080 (18) | 0.00918 (18) | -0.00048 (18) |
| O2 | 0.0210 (7) | 0.0221 (7) | 0.0160 (6) | 0.0015 (5) | 0.0085 (5) | 0.0000 (5) |
| O1 | 0.0218 (7) | 0.0229 (7) | 0.0208 (7) | 0.0045 (6) | 0.0112 (6) | 0.0014 (5) |
| N2 | 0.0159 (8) | 0.0182 (8) | 0.0155 (8) | 0.0018 (6) | 0.0062 (6) | 0.0012 (6) |
| N1 | 0.0162 (8) | 0.0170 (8) | 0.0151 (7) | -0.0005 (6) | 0.0073 (6) | -0.0023 (6) |
| O3 | 0.0156 (7) | 0.0230 (7) | 0.0245 (7) | -0.0022 (5) | 0.0072 (5) | -0.0032 (6) |
| O4 | 0.0325 (8) | 0.0269 (8) | 0.0232 (7) | 0.0000 (6) | 0.0183 (6) | -0.0027 (6) |
| C6 | 0.0168 (9) | 0.0173 (9) | 0.0163 (9) | -0.0034 (7) | 0.0018 (7) | -0.0016 (7) |
| C9 | 0.0157 (9) | 0.0163 (9) | 0.0136 (8) | -0.0038 (7) | 0.0028 (7) | -0.0017 (7) |
| C10 | 0.0121 (8) | 0.0192 (9) | 0.0170 (9) | -0.0021 (7) | 0.0013 (7) | 0.0026 (7) |
| C7 | 0.0130 (8) | 0.0187 (9) | 0.0171 (9) | -0.0023 (7) | 0.0041 (7) | -0.0018 (7) |
| C1 | 0.0223 (9) | 0.0172 (9) | 0.0161 (9) | -0.0019 (7) | 0.0037 (7) | -0.0020 (7) |
| C18 | 0.0263 (10) | 0.0227 (10) | 0.0197 (10) | -0.0050 (8) | 0.0075 (8) | -0.0057 (8) |
| C5 | 0.0192 (10) | 0.0194 (10) | 0.0243 (10) | 0.0010 (7) | 0.0020 (8) | -0.0008 (8) |
| C8 | 0.0147 (8) | 0.0179 (9) | 0.0134 (9) | -0.0015 (7) | 0.0049 (7) | -0.0016 (7) |
| C14 | 0.0180 (9) | 0.0190 (10) | 0.0273 (10) | -0.0014 (8) | -0.0003 (8) | 0.0024 (8) |
| C16 | 0.0267 (11) | 0.0192 (10) | 0.0250 (10) | 0.0004 (8) | 0.0083 (9) | -0.0035 (8) |
| C4 | 0.0292 (11) | 0.0210 (10) | 0.0246 (10) | 0.0028 (8) | -0.0023 (9) | 0.0033 (8) |
| C15 | 0.0140 (9) | 0.0193 (9) | 0.0199 (9) | -0.0028 (7) | 0.0025 (7) | -0.0007 (7) |
| C12 | 0.0275 (11) | 0.0277 (11) | 0.0163 (9) | -0.0036 (8) | 0.0015 (8) | 0.0010 (8) |
| C11 | 0.0235 (10) | 0.0196 (10) | 0.0168 (9) | -0.0011 (8) | 0.0034 (8) | -0.0005 (7) |

| | | | | | | |
|-----|-------------|-------------|-------------|-------------|-------------|------------|
| C3 | 0.0429 (13) | 0.0229 (11) | 0.0187 (10) | -0.0008 (9) | 0.0013 (9) | 0.0054 (8) |
| C13 | 0.0250 (11) | 0.0249 (11) | 0.0211 (10) | -0.0024 (8) | -0.0005 (8) | 0.0073 (8) |
| C2 | 0.0353 (12) | 0.0248 (10) | 0.0190 (10) | -0.0030 (9) | 0.0101 (9) | 0.0017 (8) |
| C17 | 0.0428 (14) | 0.0220 (11) | 0.0361 (13) | 0.0039 (9) | 0.0029 (11) | 0.0019 (9) |

Geometric parameters (Å, °)

| | | | |
|------------|-------------|---------------|-------------|
| S1—O4 | 1.4308 (13) | C5—C4 | 1.386 (3) |
| S1—O3 | 1.4345 (14) | C5—H5 | 0.9500 |
| S1—N1 | 1.6424 (15) | C14—C13 | 1.393 (3) |
| S1—C1 | 1.7639 (19) | C14—C15 | 1.405 (3) |
| O2—C9 | 1.257 (2) | C14—C17 | 1.510 (3) |
| O1—C7 | 1.344 (2) | C16—C15 | 1.504 (3) |
| O1—H1O | 0.8400 | C16—H16A | 0.9800 |
| N2—C9 | 1.343 (2) | C16—H16B | 0.9800 |
| N2—C10 | 1.422 (2) | C16—H16C | 0.9800 |
| N2—H1N | 0.85 (2) | C4—C3 | 1.389 (3) |
| N1—C8 | 1.442 (2) | C4—H4 | 0.9500 |
| N1—C18 | 1.485 (2) | C12—C13 | 1.383 (3) |
| C6—C5 | 1.400 (3) | C12—C11 | 1.388 (3) |
| C6—C1 | 1.404 (3) | C12—H12 | 0.9500 |
| C6—C7 | 1.467 (3) | C11—H11 | 0.9500 |
| C9—C8 | 1.471 (2) | C3—C2 | 1.394 (3) |
| C10—C11 | 1.392 (3) | C3—H3 | 0.9500 |
| C10—C15 | 1.399 (3) | C13—H13 | 0.9500 |
| C7—C8 | 1.359 (3) | C2—H2 | 0.9500 |
| C1—C2 | 1.389 (3) | C17—H17A | 0.9800 |
| C18—H18A | 0.9800 | C17—H17B | 0.9800 |
| C18—H18B | 0.9800 | C17—H17C | 0.9800 |
| C18—H18C | 0.9800 | | |
| O4—S1—O3 | 119.46 (8) | C7—C8—C9 | 120.66 (16) |
| O4—S1—N1 | 108.44 (8) | N1—C8—C9 | 118.12 (15) |
| O3—S1—N1 | 107.14 (8) | C13—C14—C15 | 118.96 (18) |
| O4—S1—C1 | 109.98 (9) | C13—C14—C17 | 119.71 (18) |
| O3—S1—C1 | 108.17 (8) | C15—C14—C17 | 121.33 (18) |
| N1—S1—C1 | 102.26 (8) | C15—C16—H16A | 109.5 |
| C7—O1—H1O | 109.5 | C15—C16—H16B | 109.5 |
| C9—N2—C10 | 127.78 (16) | H16A—C16—H16B | 109.5 |
| C9—N2—H1N | 115.6 (16) | C15—C16—H16C | 109.5 |
| C10—N2—H1N | 116.6 (16) | H16A—C16—H16C | 109.5 |
| C8—N1—C18 | 115.56 (14) | H16B—C16—H16C | 109.5 |
| C8—N1—S1 | 112.65 (12) | C5—C4—C3 | 120.83 (19) |
| C18—N1—S1 | 116.30 (12) | C5—C4—H4 | 119.6 |
| C5—C6—C1 | 118.36 (17) | C3—C4—H4 | 119.6 |
| C5—C6—C7 | 121.28 (17) | C10—C15—C14 | 118.67 (17) |
| C1—C6—C7 | 120.36 (17) | C10—C15—C16 | 119.51 (17) |
| O2—C9—N2 | 123.99 (16) | C14—C15—C16 | 121.80 (17) |
| O2—C9—C8 | 119.87 (16) | C13—C12—C11 | 120.17 (18) |
| N2—C9—C8 | 116.14 (15) | C13—C12—H12 | 119.9 |

supplementary materials

| | | | |
|---------------|--------------|-----------------|--------------|
| C11—C10—C15 | 121.91 (17) | C11—C12—H12 | 119.9 |
| C11—C10—N2 | 120.98 (17) | C12—C11—C10 | 118.68 (18) |
| C15—C10—N2 | 117.10 (16) | C12—C11—H11 | 120.7 |
| O1—C7—C8 | 122.29 (16) | C10—C11—H11 | 120.7 |
| O1—C7—C6 | 114.99 (16) | C4—C3—C2 | 120.22 (19) |
| C8—C7—C6 | 122.71 (16) | C4—C3—H3 | 119.9 |
| C2—C1—C6 | 121.79 (18) | C2—C3—H3 | 119.9 |
| C2—C1—S1 | 121.16 (15) | C12—C13—C14 | 121.55 (18) |
| C6—C1—S1 | 117.01 (14) | C12—C13—H13 | 119.2 |
| N1—C18—H18A | 109.5 | C14—C13—H13 | 119.2 |
| N1—C18—H18B | 109.5 | C1—C2—C3 | 118.73 (19) |
| H18A—C18—H18B | 109.5 | C1—C2—H2 | 120.6 |
| N1—C18—H18C | 109.5 | C3—C2—H2 | 120.6 |
| H18A—C18—H18C | 109.5 | C14—C17—H17A | 109.5 |
| H18B—C18—H18C | 109.5 | C14—C17—H17B | 109.5 |
| C4—C5—C6 | 120.06 (18) | H17A—C17—H17B | 109.5 |
| C4—C5—H5 | 120.0 | C14—C17—H17C | 109.5 |
| C6—C5—H5 | 120.0 | H17A—C17—H17C | 109.5 |
| C7—C8—N1 | 121.22 (16) | H17B—C17—H17C | 109.5 |
| O4—S1—N1—C8 | 169.35 (12) | C6—C7—C8—C9 | -176.92 (16) |
| O3—S1—N1—C8 | -60.45 (14) | C18—N1—C8—C7 | 94.4 (2) |
| C1—S1—N1—C8 | 53.19 (14) | S1—N1—C8—C7 | -42.6 (2) |
| O4—S1—N1—C18 | 32.66 (15) | C18—N1—C8—C9 | -85.4 (2) |
| O3—S1—N1—C18 | 162.85 (13) | S1—N1—C8—C9 | 137.60 (14) |
| C1—S1—N1—C18 | -83.51 (14) | O2—C9—C8—C7 | -7.7 (3) |
| C10—N2—C9—O2 | -1.0 (3) | N2—C9—C8—C7 | 172.23 (16) |
| C10—N2—C9—C8 | 179.07 (16) | O2—C9—C8—N1 | 172.09 (16) |
| C9—N2—C10—C11 | 35.7 (3) | N2—C9—C8—N1 | -8.0 (2) |
| C9—N2—C10—C15 | -145.22 (18) | C6—C5—C4—C3 | 0.3 (3) |
| C5—C6—C7—O1 | 18.7 (2) | C11—C10—C15—C14 | 1.6 (3) |
| C1—C6—C7—O1 | -160.50 (16) | N2—C10—C15—C14 | -177.46 (16) |
| C5—C6—C7—C8 | -162.85 (18) | C11—C10—C15—C16 | -176.85 (17) |
| C1—C6—C7—C8 | 18.0 (3) | N2—C10—C15—C16 | 4.1 (2) |
| C5—C6—C1—C2 | 0.7 (3) | C13—C14—C15—C10 | -2.1 (3) |
| C7—C6—C1—C2 | 179.90 (17) | C17—C14—C15—C10 | 178.91 (18) |
| C5—C6—C1—S1 | -176.97 (14) | C13—C14—C15—C16 | 176.29 (18) |
| C7—C6—C1—S1 | 2.2 (2) | C17—C14—C15—C16 | -2.7 (3) |
| O4—S1—C1—C2 | 32.47 (18) | C13—C12—C11—C10 | -2.0 (3) |
| O3—S1—C1—C2 | -99.61 (17) | C15—C10—C11—C12 | 0.5 (3) |
| N1—S1—C1—C2 | 147.51 (16) | N2—C10—C11—C12 | 179.45 (17) |
| O4—S1—C1—C6 | -149.86 (14) | C5—C4—C3—C2 | 0.1 (3) |
| O3—S1—C1—C6 | 78.06 (16) | C11—C12—C13—C14 | 1.4 (3) |
| N1—S1—C1—C6 | -34.82 (16) | C15—C14—C13—C12 | 0.6 (3) |
| C1—C6—C5—C4 | -0.7 (3) | C17—C14—C13—C12 | 179.7 (2) |
| C7—C6—C5—C4 | -179.94 (17) | C6—C1—C2—C3 | -0.2 (3) |
| O1—C7—C8—N1 | -178.38 (15) | S1—C1—C2—C3 | 177.33 (16) |
| C6—C7—C8—N1 | 3.3 (3) | C4—C3—C2—C1 | -0.2 (3) |
| O1—C7—C8—C9 | 1.4 (3) | | |

Hydrogen-bond geometry (Å, °)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------|----------|-------------|-------------|---------------|
| O1—H1O \cdots O2 | 0.84 | 1.79 | 2.5320 (18) | 147 |
| N2—H1N \cdots O3 ⁱ | 0.85 (2) | 2.26 (2) | 2.972 (2) | 141 (2) |
| C3—H3 \cdots O4 ⁱⁱ | 0.95 | 2.49 | 3.352 (3) | 150 |

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+3/2, y-1/2, -z+1/2$.

Fig. 1

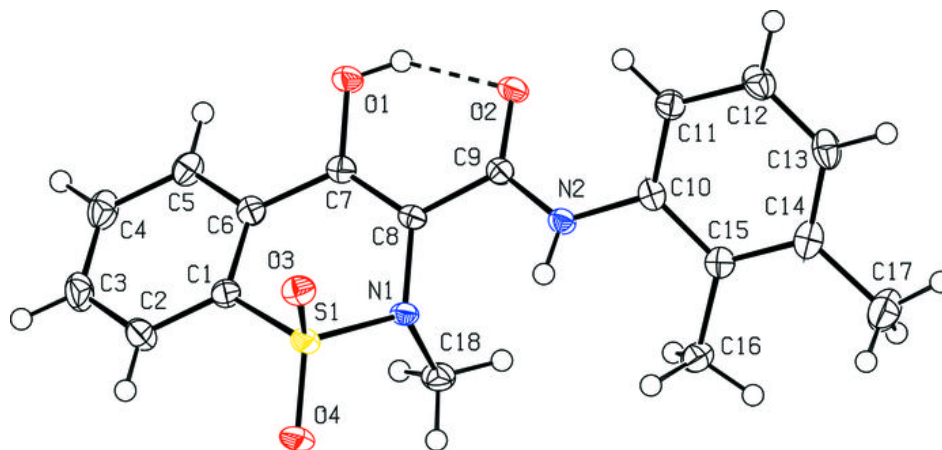


Fig. 2

