

# N-[2-(Aminocarbonyl)phenyl]-4-hydroxy-2H-1,2-benzothiazine-3-carboxamide 1,1-dioxide dimethyl sulfoxide solvate

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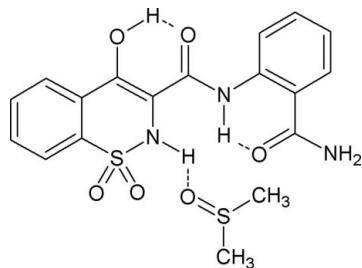
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Key indicators: single-crystal X-ray study;  $T = 150$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å; R factor = 0.030; wR factor = 0.085; data-to-parameter ratio = 16.9.

The title compound,  $\text{C}_{16}\text{H}_{13}\text{N}_3\text{O}_5\text{S}\cdot\text{C}_2\text{H}_6\text{OS}$ , is of interest as a precursor to biologically active (benzothiazin-3-yl)quinazolones and its structure is reported here as the dimethyl sulfoxide (DMSO) solvate. The structure displays intramolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonding, giving rise to six-membered hydrogen-bonded rings, with head-to-tail intermolecular pairs of  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds linking pairs of neighbouring molecules and two independent  $\text{N}-\text{H}\cdots\text{O}=\text{S}$  intermolecular hydrogen bonds to the DMSO molecule resulting in the pairs of molecules being linked into stepped chains parallel to  $a$ .

## Related literature

For related literature, see: Bihovsky *et al.* (2004); Fabiola *et al.* (1998); Fringuelli *et al.* (2005); Kojić-Prodić & Ružić-Toroš (1982); Rehman *et al.* (2005, 2006); Turck *et al.* (1996); Weast *et al.* (1984).



## Experimental

### Crystal data

$\text{C}_{16}\text{H}_{13}\text{N}_3\text{O}_5\text{S}\cdot\text{C}_2\text{H}_6\text{OS}$   
 $M_r = 437.48$   
 Monoclinic,  $P2_1/n$   
 $a = 8.8314$  (5) Å  
 $b = 9.1405$  (5) Å  
 $c = 24.5041$  (14) Å  
 $\beta = 96.499$  (2)°

$V = 1965.34$  (19) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.31$  mm<sup>-1</sup>  
 $T = 150$  (2) K  
 $0.90 \times 0.45 \times 0.45$  mm

### Data collection

Bruker SMART 1000 CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)  
 $T_{\min} = 0.766$ ,  $T_{\max} = 0.872$

16737 measured reflections  
 4713 independent reflections  
 4123 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.016$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.085$   
 $S = 1.01$   
 4713 reflections  
 279 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.34$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.37$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O6}$	0.803 (18)	1.985 (18)	2.7837 (15)	173.5 (17)
$\text{O3}-\text{H3}\cdots\text{O4}$	0.90 (2)	1.69 (2)	2.5255 (14)	153.7 (17)
$\text{N2}-\text{H2}\cdots\text{O5}$	0.849 (17)	1.864 (17)	2.5846 (15)	141.8 (16)
$\text{N2}-\text{H2}\cdots\text{N1}$	0.849 (17)	2.307 (17)	2.7322 (15)	111.3 (13)
$\text{N3}-\text{H3A}\cdots\text{O6}^i$	0.86 (2)	2.07 (2)	2.9139 (16)	168.3 (18)
$\text{N3}-\text{H3B}\cdots\text{O2}^{ii}$	0.85 (2)	2.19 (2)	3.0235 (16)	169.4 (18)

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

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; 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: SK3158).

## References

- Bihovsky, R., Tao, M., Mallamo, J. P. & Wells, G. J. (2004). *Bioorg. Med. Chem. Lett.* **14**, 1035–1038.  
 Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Bruker (2001). *SMART* (Version 5.611) and *S SAINT* (Version 6.02a). Bruker AXS Inc., Madison, Wisconsin, USA.  
 Fabiola, G. F., Pattabhi, V., Manjunatha, S. G., Rao, G. V. & Nagarajan, K. (1998). *Acta Cryst.* **C54**, 2001–2003.  
 Fringuelli, R., Milanese, L. & Schiaffella, F. (2005). *Mini Rev. Med. Chem.* **5**, 1061–1073.

- Kojić-Prodić, B. & Ružić-Toroš, Ž. (1982). *Acta Cryst.* **B38**, 2948–2951.
- Rehman, M. Z., Choudary, J. A., Ahmad, S. & Siddiqui, H. L. (2006). *Chem. Pharm. Bull.* **54**, 1175–1178.
- Rehman, M. Z., Choudary, J. A. & Ahmad, S. (2005). *Bull. Korean Chem. Soc.* **26**, 1771–1775.
- Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.
- Turck, D., Busch, U., Heinzel, G., Narjes, H. & Nehmiz, G. (1996). *J. Clin. Pharmacol.* **36**, 79–84.
- Weast, R. C., Astle, M. J. & Beyer, W. H. (1984). *Handbook of Chemistry and Physics*. 65th ed. Boca Raton, Florida: CRC Press.

**supplementary materials**

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***N*-[2-(Aminocarbonyl)phenyl]-4-hydroxy-2*H*-1,2-benzothiazine-3-carboxamide 1,1-dioxide dimethyl sulfoxide solvate**

**M. Zia-ur-Rehman, J. A. Choudary, M. R. J. Elsegood, H. L. Siddiqui and G. W. Weaver**

**Comment**

Benzothiazines represent an important class of organic heterocyclic compounds characterized by highly pronounced biological properties. 1,2-benzothiazine-1,1-dioxides are known for their anti-rheumatic and anti-inflammatory activities (Turck *et al.*, 1996; Bihovsky *et al.*, 2004) while 1,4-benzothiazine-1,1-dioxides are known for their anti-microbial, anti-allergic and enzyme inhibition activities (Fringuelli *et al.*, 2005). As part of a research program synthesizing benzothiazinic bioactive compounds, *N*-[2-(aminocarbonyl) phenyl]-4-hydroxy-2*H*-1,2-benzothiazine-3-carboxamide 1,1-dioxide was prepared from methyl 4-hydroxy-2*H*-1,2-benzothiazine-3-carboxylate 1,1-dioxide as an intermediate of 2-(4-hydroxy-1,1-dioxido-2*H*-1,2-benzothiazin-3-yl)quinazolin-4(3*H*)-ones (Rehman *et al.*, 2005; Rehman *et al.*, 2006).

In this paper, the structure of the title compound, (I), co-crystallized with dimethyl sulfoxide, (C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>O<sub>6</sub>S<sub>2</sub>) is reported (Scheme and Fig. 1). The thiazine ring, involving two double bonds, exhibits a half-chair conformation; with S1—C1—C6—C7 relatively planar and N1 showing significant departure from plane due to its pyramidal geometry (sum of angles at N1 = 344.4 °). The torsion angles in the ring also support this conformation (Table 2). The S1—N1—C8 bond angle is the largest [115.33 (9) Å] among those of polymorphs of Piroxicam [113.0 (2) Å; Kojić-Prodić & Ružić-Toroš, 1982] or Meloxicam [112.8 (1) Å; Fabiola *et al.*, 1998] due to the absence of the methyl group at N1.

The molecule is not far from being planar since the the enolic hydrogen on O3 is involved in intramolecular hydrogen bonding [O3—H3···O4] with the amido oxygen at C4 giving rise to a six-membered hydrogen bonded ring. Relevant torsion angles are presented in Table 2. The C1—S1 bond (Table 2) is shorter than the normal C—S single bond (1.81–2.55 Å) (Weast *et al.*, 1984) due to partial double bond character and is very close to the values observed in hetrocyclic systems [1.57–1.75 (6) Å] described for common sulfonamides (Weast *et al.*, 1984). The C7—C8 bond length (Table 2) is also shortened due to partial double bond character indicating the stability of the enol form of the molecule.

The intramolecular [N2—H2···O5] interaction forms a second six-membered hydrogen-bonded ring. Each molecule (I) is hydrogen bonded to its neighbour *via* a centrosymmetric head-to-tail interaction with anthranilamide hydrogen H3B hydrogen bonded to the sulfone oxygen [N3—H3B···O2<sup>ii</sup>] (Table 1 and Fig. 2). These pairs of molecules are then linked into chains *via* two independent hydrogen bonds to the DMSO molecule which acts as a bridge; anthranilamide hydrogen, H3A, is linked to DMSO oxygen, O6, which is further bonded to amino hydrogen H1<sup>i</sup> of the adjacent benzothiazine molecule to form an infinite stepped chain parallel to a (see Fig. 2 which also gives symmetry operators).

**Experimental**

The title compound was synthesized according to our method (Rehman *et al.*, 2005). The compound was co-crystallized with DMSO by dissolving it in a mixture of methanol & DMSO (80:20 *v/v*) at room temperature and the crystals were obtained by slow evaporation (followed by drying under high vacuum).

## Refinement

H atoms bound to C were placed in geometric positions (C—H distance = 0.95 Å for aryl H; 1.00 Å for methyl H) using a riding model. H atoms on N and O had coordinates freely refined.  $U_{iso}$  values were set to  $1.2U_{eq}$  ( $1.5U_{eq}$  for methyl H and OH).

## Figures

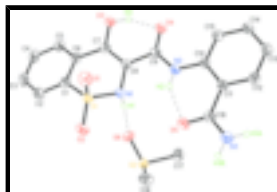


Fig. 1. Asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level, H atoms are represented by circles of arbitrary radius and hydrogen bonds are shown as dashed lines.

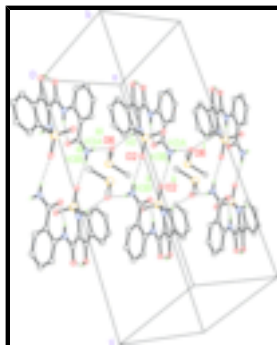


Fig. 2. Packing plot of (I) showing head-to-tail hydrogen bonding between pairs of molecules and hydrogen bonding with dimethyl sulfoxide to form an infinite chains. Symmetry operators: i =  $x + 1, y, z$ ; ii =  $-x + 1, -y + 1, -z + 1$ ; iii =  $x - 1, y, z$ .

## *N*-[2-(Aminocarbonyl)phenyl]-4-hydroxy-2*H*-1,2-benzothiazine- 3-carboxamide 1,1-dioxide dimethyl sulfoxide solvate

### Crystal data

$C_{16}H_{13}N_3O_5S \cdot C_2H_6OS$

$M_r = 437.48$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P 2_1n$

$a = 8.8314 (5) \text{ \AA}$

$b = 9.1405 (5) \text{ \AA}$

$c = 24.5041 (14) \text{ \AA}$

$\beta = 96.499 (2)^\circ$

$V = 1965.34 (19) \text{ \AA}^3$

$Z = 4$

$F_{000} = 912$

$D_x = 1.479 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

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

Cell parameters from 10921 reflections

$\theta = 2.3\text{--}28.8^\circ$

$\mu = 0.31 \text{ mm}^{-1}$

$T = 150 (2) \text{ K}$

Block, colourless

$0.90 \times 0.45 \times 0.45 \text{ mm}$

### Data collection

Bruker SMART 1000 CCD  
diffractometer

Radiation source: sealed tube

4713 independent reflections

4123 reflections with  $I > 2\sigma(I)$

Monochromator: graphite  $R_{\text{int}} = 0.016$   
 $T = 150(2)$  K  $\theta_{\text{max}} = 28.9^\circ$   
 $\omega$  rotation with narrow frames scans  $\theta_{\text{min}} = 1.7^\circ$   
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)  $h = -11 \rightarrow 11$   
 $T_{\text{min}} = 0.766$ ,  $T_{\text{max}} = 0.872$   $k = -12 \rightarrow 12$   
 16737 measured reflections  $l = -32 \rightarrow 32$

### Refinement

Refinement on  $F^2$  Secondary atom site location: structure-invariant direct methods  
 Least-squares matrix: full Hydrogen site location: geom except NH & OH coords freely refined  
 $R[F^2 > 2\sigma(F^2)] = 0.030$  H atoms treated by a mixture of independent and constrained refinement  
 $wR(F^2) = 0.085$   $w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.9251P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $S = 1.02$   $(\Delta/\sigma)_{\text{max}} = 0.001$   
 4713 reflections  $\Delta\rho_{\text{max}} = 0.34 \text{ e } \text{\AA}^{-3}$   
 279 parameters  $\Delta\rho_{\text{min}} = -0.36 \text{ e } \text{\AA}^{-3}$   
 Primary atom site location: structure-invariant direct methods Extinction correction: none

### 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.28875 (13)	0.71759 (13)	0.39205 (4)	0.0229 (2)
H1	0.2612 (19)	0.634 (2)	0.3911 (7)	0.027*
S1	0.23801 (4)	0.80523 (4)	0.444708 (13)	0.02510 (9)
O1	0.33285 (12)	0.93249 (12)	0.45368 (4)	0.0330 (2)
O2	0.23237 (13)	0.70279 (12)	0.48863 (4)	0.0353 (2)
C1	0.05307 (15)	0.86379 (14)	0.42033 (5)	0.0244 (3)
C2	-0.06252 (17)	0.87069 (16)	0.45421 (6)	0.0307 (3)
H2A	-0.0458	0.8371	0.4911	0.037*
C3	-0.20305 (17)	0.92747 (17)	0.43331 (6)	0.0339 (3)

## supplementary materials

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H3C	-0.2837	0.9319	0.4559	0.041*
C4	-0.22597 (17)	0.97760 (17)	0.37973 (7)	0.0339 (3)
H4	-0.3216	1.0189	0.3662	0.041*
C5	-0.11114 (16)	0.96846 (15)	0.34543 (6)	0.0281 (3)
H5	-0.1288	1.0024	0.3086	0.034*
C6	0.03031 (15)	0.90915 (14)	0.36529 (5)	0.0232 (3)
C7	0.14923 (14)	0.88905 (14)	0.32880 (5)	0.0220 (2)
O3	0.13057 (11)	0.96755 (11)	0.28223 (4)	0.0274 (2)
H3	0.209 (2)	0.940 (2)	0.2644 (7)	0.041*
C8	0.26952 (14)	0.79629 (13)	0.34112 (5)	0.0210 (2)
C9	0.38850 (14)	0.78367 (14)	0.30398 (5)	0.0217 (2)
O4	0.37241 (11)	0.84771 (11)	0.25857 (4)	0.0283 (2)
N2	0.51089 (12)	0.70322 (12)	0.32316 (5)	0.0231 (2)
H2	0.5093 (19)	0.6621 (19)	0.3542 (7)	0.028*
C10	0.64929 (14)	0.68352 (14)	0.30069 (5)	0.0218 (2)
C11	0.76511 (14)	0.59886 (14)	0.33032 (5)	0.0216 (2)
C12	0.90429 (15)	0.58378 (14)	0.30863 (5)	0.0240 (3)
H12	0.9827	0.5264	0.3279	0.029*
C13	0.93062 (15)	0.65033 (15)	0.25981 (5)	0.0261 (3)
H13	1.0261	0.6389	0.2459	0.031*
C14	0.81693 (16)	0.73342 (15)	0.23146 (5)	0.0272 (3)
H14	0.8351	0.7801	0.1982	0.033*
C15	0.67642 (16)	0.74937 (15)	0.25111 (5)	0.0264 (3)
H15	0.5985	0.8051	0.2309	0.032*
C16	0.73975 (15)	0.52543 (15)	0.38339 (5)	0.0250 (3)
O5	0.61267 (11)	0.52392 (13)	0.40027 (4)	0.0360 (3)
N3	0.85668 (15)	0.45913 (16)	0.41225 (6)	0.0366 (3)
H3A	0.947 (2)	0.459 (2)	0.4028 (8)	0.044*
H3B	0.840 (2)	0.421 (2)	0.4425 (8)	0.044*
S2	0.23625 (4)	0.28521 (3)	0.414879 (13)	0.02426 (9)
O6	0.17641 (11)	0.43343 (11)	0.39463 (4)	0.0314 (2)
C17	0.35062 (19)	0.22342 (18)	0.36405 (7)	0.0367 (3)
H17A	0.2866	0.2107	0.3290	0.055*
H17B	0.3980	0.1298	0.3755	0.055*
H17C	0.4302	0.2958	0.3596	0.055*
C18	0.38444 (18)	0.31915 (18)	0.46878 (6)	0.0350 (3)
H18A	0.4597	0.3854	0.4556	0.053*
H18B	0.4339	0.2266	0.4805	0.053*
H18C	0.3414	0.3641	0.4999	0.053*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0264 (5)	0.0209 (5)	0.0220 (5)	0.0013 (4)	0.0050 (4)	0.0042 (4)
S1	0.02709 (17)	0.02870 (18)	0.01969 (15)	0.00122 (12)	0.00346 (12)	0.00252 (12)
O1	0.0334 (5)	0.0366 (6)	0.0280 (5)	-0.0042 (4)	-0.0001 (4)	-0.0039 (4)
O2	0.0394 (6)	0.0438 (6)	0.0239 (5)	0.0069 (5)	0.0084 (4)	0.0112 (4)
C1	0.0264 (6)	0.0215 (6)	0.0256 (6)	0.0007 (5)	0.0041 (5)	-0.0015 (5)

C2	0.0353 (7)	0.0304 (7)	0.0280 (7)	-0.0008 (6)	0.0099 (6)	-0.0021 (5)
C3	0.0317 (7)	0.0340 (8)	0.0386 (8)	0.0008 (6)	0.0145 (6)	-0.0059 (6)
C4	0.0272 (7)	0.0335 (8)	0.0417 (8)	0.0066 (6)	0.0065 (6)	-0.0041 (6)
C5	0.0268 (7)	0.0267 (7)	0.0310 (7)	0.0044 (5)	0.0041 (5)	-0.0005 (5)
C6	0.0241 (6)	0.0195 (6)	0.0264 (6)	0.0003 (5)	0.0041 (5)	-0.0009 (5)
C7	0.0238 (6)	0.0202 (6)	0.0220 (6)	-0.0015 (5)	0.0020 (5)	0.0017 (4)
O3	0.0279 (5)	0.0291 (5)	0.0256 (5)	0.0051 (4)	0.0044 (4)	0.0081 (4)
C8	0.0227 (6)	0.0211 (6)	0.0196 (6)	-0.0008 (5)	0.0033 (4)	0.0023 (4)
C9	0.0217 (6)	0.0218 (6)	0.0216 (6)	-0.0019 (5)	0.0022 (5)	0.0003 (4)
O4	0.0269 (5)	0.0346 (5)	0.0238 (5)	0.0037 (4)	0.0053 (4)	0.0083 (4)
N2	0.0228 (5)	0.0264 (6)	0.0207 (5)	0.0019 (4)	0.0054 (4)	0.0052 (4)
C10	0.0223 (6)	0.0211 (6)	0.0225 (6)	-0.0011 (5)	0.0044 (5)	-0.0013 (5)
C11	0.0234 (6)	0.0198 (6)	0.0220 (6)	-0.0020 (5)	0.0044 (5)	-0.0002 (4)
C12	0.0236 (6)	0.0220 (6)	0.0268 (6)	0.0002 (5)	0.0042 (5)	-0.0004 (5)
C13	0.0256 (6)	0.0261 (6)	0.0282 (6)	-0.0015 (5)	0.0098 (5)	-0.0025 (5)
C14	0.0321 (7)	0.0271 (6)	0.0239 (6)	-0.0014 (5)	0.0094 (5)	0.0027 (5)
C15	0.0286 (7)	0.0269 (6)	0.0240 (6)	0.0027 (5)	0.0054 (5)	0.0037 (5)
C16	0.0244 (6)	0.0243 (6)	0.0269 (6)	0.0005 (5)	0.0050 (5)	0.0041 (5)
O5	0.0242 (5)	0.0497 (6)	0.0357 (5)	0.0062 (4)	0.0099 (4)	0.0200 (5)
N3	0.0259 (6)	0.0486 (8)	0.0367 (7)	0.0088 (6)	0.0092 (5)	0.0214 (6)
S2	0.02129 (16)	0.02251 (16)	0.02931 (17)	0.00014 (11)	0.00425 (12)	0.00387 (12)
O6	0.0265 (5)	0.0257 (5)	0.0420 (6)	0.0046 (4)	0.0040 (4)	0.0074 (4)
C17	0.0420 (8)	0.0333 (8)	0.0367 (8)	0.0036 (6)	0.0120 (7)	-0.0045 (6)
C18	0.0346 (8)	0.0407 (8)	0.0284 (7)	-0.0038 (6)	-0.0023 (6)	0.0053 (6)

*Geometric parameters (Å, °)*

N1—C8	1.4337 (16)	C10—C15	1.4004 (18)
N1—S1	1.6244 (11)	C10—C11	1.4157 (18)
N1—H1	0.803 (18)	C11—C12	1.4001 (17)
S1—O2	1.4316 (10)	C11—C16	1.5025 (17)
S1—O1	1.4357 (11)	C12—C13	1.3850 (18)
S1—C1	1.7579 (14)	C12—H12	0.9500
C1—C2	1.3881 (19)	C13—C14	1.382 (2)
C1—C6	1.4035 (18)	C13—H13	0.9500
C2—C3	1.389 (2)	C14—C15	1.3885 (19)
C2—H2A	0.9500	C14—H14	0.9500
C3—C4	1.384 (2)	C15—H15	0.9500
C3—H3C	0.9500	C16—O5	1.2394 (16)
C4—C5	1.391 (2)	C16—N3	1.3298 (18)
C4—H4	0.9500	N3—H3A	0.86 (2)
C5—C6	1.3977 (18)	N3—H3B	0.85 (2)
C5—H5	0.9500	S2—O6	1.5171 (10)
C6—C7	1.4658 (18)	S2—C18	1.7782 (15)
C7—O3	1.3424 (15)	S2—C17	1.7819 (15)
C7—C8	1.3660 (18)	C17—H17A	0.9800
O3—H3	0.90 (2)	C17—H17B	0.9800
C8—C9	1.4705 (17)	C17—H17C	0.9800
C9—O4	1.2511 (15)	C18—H18A	0.9800



## supplementary materials

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C9—N2	1.3472 (17)	C18—H18B	0.9800
N2—C10	1.4078 (16)	C18—H18C	0.9800
N2—H2	0.849 (17)		
C8—N1—S1	115.33 (9)	C15—C10—N2	121.71 (12)
C8—N1—H1	116.6 (12)	C15—C10—C11	119.68 (12)
S1—N1—H1	112.5 (12)	N2—C10—C11	118.56 (11)
O2—S1—O1	118.96 (7)	C12—C11—C10	118.26 (11)
O2—S1—N1	108.11 (6)	C12—C11—C16	120.52 (11)
O1—S1—N1	108.21 (6)	C10—C11—C16	121.22 (11)
O2—S1—C1	110.16 (7)	C13—C12—C11	121.68 (12)
O1—S1—C1	107.99 (6)	C13—C12—H12	119.2
N1—S1—C1	102.09 (6)	C11—C12—H12	119.2
C2—C1—C6	121.75 (13)	C14—C13—C12	119.48 (12)
C2—C1—S1	121.90 (11)	C14—C13—H13	120.3
C6—C1—S1	116.29 (10)	C12—C13—H13	120.3
C1—C2—C3	118.92 (13)	C13—C14—C15	120.69 (12)
C1—C2—H2A	120.5	C13—C14—H14	119.7
C3—C2—H2A	120.5	C15—C14—H14	119.7
C4—C3—C2	120.15 (13)	C14—C15—C10	120.20 (12)
C4—C3—H3C	119.9	C14—C15—H15	119.9
C2—C3—H3C	119.9	C10—C15—H15	119.9
C3—C4—C5	121.01 (14)	O5—C16—N3	119.70 (12)
C3—C4—H4	119.5	O5—C16—C11	121.55 (12)
C5—C4—H4	119.5	N3—C16—C11	118.75 (12)
C4—C5—C6	119.79 (13)	C16—N3—H3A	123.4 (13)
C4—C5—H5	120.1	C16—N3—H3B	116.6 (13)
C6—C5—H5	120.1	H3A—N3—H3B	119.8 (18)
C5—C6—C1	118.31 (12)	O6—S2—C18	106.68 (7)
C5—C6—C7	120.66 (12)	O6—S2—C17	104.98 (7)
C1—C6—C7	120.97 (12)	C18—S2—C17	98.44 (8)
O3—C7—C8	122.49 (12)	S2—C17—H17A	109.5
O3—C7—C6	115.09 (11)	S2—C17—H17B	109.5
C8—C7—C6	122.41 (11)	H17A—C17—H17B	109.5
C7—O3—H3	103.6 (12)	S2—C17—H17C	109.5
C7—C8—N1	121.08 (11)	H17A—C17—H17C	109.5
C7—C8—C9	120.29 (11)	H17B—C17—H17C	109.5
N1—C8—C9	118.48 (11)	S2—C18—H18A	109.5
O4—C9—N2	124.86 (12)	S2—C18—H18B	109.5
O4—C9—C8	119.77 (11)	H18A—C18—H18B	109.5
N2—C9—C8	115.35 (11)	S2—C18—H18C	109.5
C9—N2—C10	129.14 (11)	H18A—C18—H18C	109.5
C9—N2—H2	117.4 (11)	H18B—C18—H18C	109.5
C10—N2—H2	113.4 (11)		
C8—N1—S1—O2	167.49 (9)	C6—C7—C8—C9	-177.98 (11)
C8—N1—S1—O1	-62.43 (11)	S1—N1—C8—C7	-35.64 (16)
C8—N1—S1—C1	51.33 (11)	S1—N1—C8—C9	139.83 (10)
O2—S1—C1—C2	29.67 (14)	C7—C8—C9—O4	-6.60 (19)
O1—S1—C1—C2	-101.74 (12)	N1—C8—C9—O4	177.89 (12)

N1—S1—C1—C2	144.33 (12)	C7—C8—C9—N2	172.19 (12)
O2—S1—C1—C6	-152.95 (10)	N1—C8—C9—N2	-3.32 (17)
O1—S1—C1—C6	75.65 (11)	O4—C9—N2—C10	6.4 (2)
N1—S1—C1—C6	-38.28 (11)	C8—C9—N2—C10	-172.37 (12)
C6—C1—C2—C3	-1.6 (2)	C9—N2—C10—C15	-1.1 (2)
S1—C1—C2—C3	175.59 (11)	C9—N2—C10—C11	176.67 (12)
C1—C2—C3—C4	-0.7 (2)	C15—C10—C11—C12	-0.07 (18)
C2—C3—C4—C5	1.8 (2)	N2—C10—C11—C12	-177.90 (11)
C3—C4—C5—C6	-0.6 (2)	C15—C10—C11—C16	-179.44 (12)
C4—C5—C6—C1	-1.7 (2)	N2—C10—C11—C16	2.74 (18)
C4—C5—C6—C7	175.70 (13)	C10—C11—C12—C13	0.64 (19)
C2—C1—C6—C5	2.8 (2)	C16—C11—C12—C13	-179.98 (12)
S1—C1—C6—C5	-174.57 (10)	C11—C12—C13—C14	-0.3 (2)
C2—C1—C6—C7	-174.52 (12)	C12—C13—C14—C15	-0.7 (2)
S1—C1—C6—C7	8.09 (17)	C13—C14—C15—C10	1.3 (2)
C5—C6—C7—O3	18.55 (18)	N2—C10—C15—C14	176.87 (13)
C1—C6—C7—O3	-164.17 (12)	C11—C10—C15—C14	-0.9 (2)
C5—C6—C7—C8	-160.94 (13)	C12—C11—C16—O5	-172.01 (13)
C1—C6—C7—C8	16.34 (19)	C10—C11—C16—O5	7.3 (2)
O3—C7—C8—N1	177.95 (11)	C12—C11—C16—N3	7.1 (2)
C6—C7—C8—N1	-2.60 (19)	C10—C11—C16—N3	-173.56 (13)
O3—C7—C8—C9	2.56 (19)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N1—H1 $\cdots$ O6	0.803 (18)	1.985 (18)	2.7837 (15)	173.5 (17)
O3—H3 $\cdots$ O4	0.90 (2)	1.69 (2)	2.5255 (14)	153.7 (17)
N2—H2 $\cdots$ O5	0.849 (17)	1.864 (17)	2.5846 (15)	141.8 (16)
N2—H2 $\cdots$ N1	0.849 (17)	2.307 (17)	2.7322 (15)	111.3 (13)
N3—H3A $\cdots$ O6 <sup>i</sup>	0.86 (2)	2.07 (2)	2.9139 (16)	168.3 (18)
N3—H3B $\cdots$ O2 <sup>ii</sup>	0.85 (2)	2.19 (2)	3.0235 (16)	169.4 (18)

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

Fig. 1

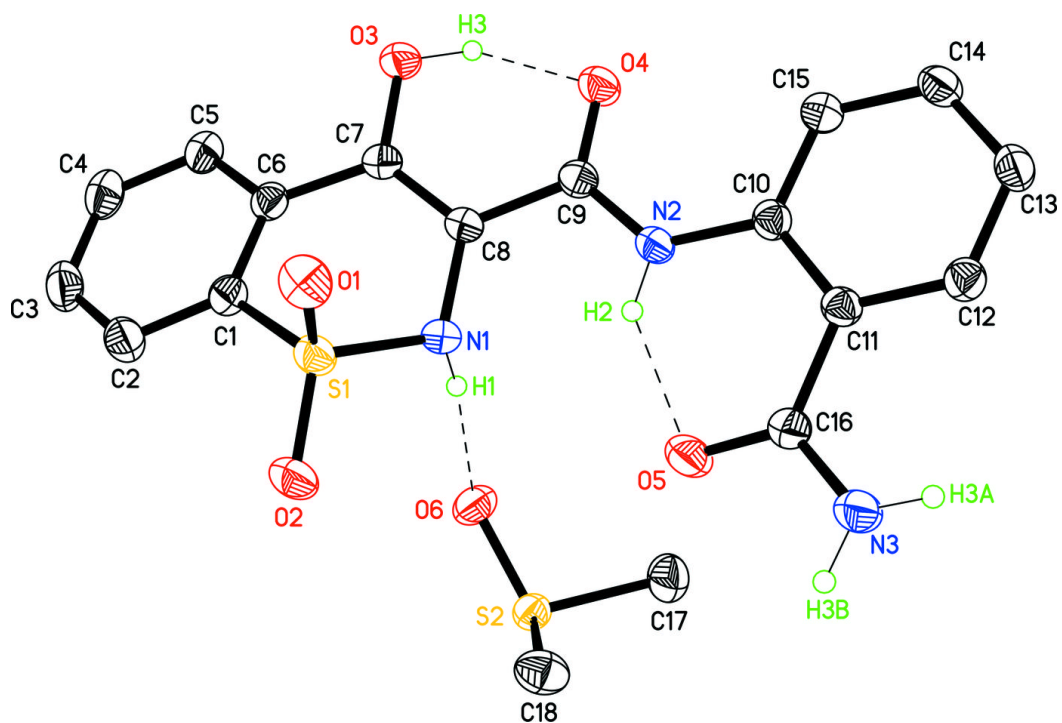


Fig. 2

