

## 4-Hydroxy-*N*-(2,4,6-tribromophenyl)-2*H*-1,2-benzothiazine-3-carboxamide 1,1-dioxide

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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å;  $R$  factor = 0.043;  $wR$  factor = 0.116; data-to-parameter ratio = 16.7.

In the title compound,  $\text{C}_{15}\text{H}_{19}\text{Br}_3\text{N}_2\text{O}_4\text{S}$ , the thiazine ring adopts a distorted half-chair conformation. The enolic H atom is involved in an intramolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bond, forming a six-membered ring. In the crystal, the molecules are linked into a three-dimensional network through intermolecular  $\text{N}-\text{H}\cdots\text{O}$ ,  $\text{N}-\text{H}\cdots\text{Br}$  and  $\text{O}-\text{H}\cdots\text{Br}$  hydrogen bonds.

### Related literature

For the synthesis of related molecules, see: Kojić-Prodić & Ružić-Toroš (1982); Zia-ur-Rehman, Choudary & Ahmad (2005). For the applications of 1,2-benzothiazine 1,1-dioxides and their precursor intermediates as non-steroidal anti-inflammatory compounds, see: Turck *et al.* (1996). For bond-length data, see: Weast *et al.* (1984).



### Experimental

#### Crystal data

$\text{C}_{15}\text{H}_{19}\text{Br}_3\text{N}_2\text{O}_4\text{S}$   
 $M_r = 553.03$   
 Triclinic,  $P\bar{1}$

$a = 7.5082$  (4) Å  
 $b = 8.7486$  (6) Å  
 $c = 13.0669$  (9) Å

$\alpha = 83.618$  (2)°  
 $\beta = 86.280$  (2)°  
 $\gamma = 87.684$  (2)°  
 $V = 850.72$  (9) Å<sup>3</sup>  
 $Z = 2$

Mo  $K\alpha$  radiation  
 $\mu = 7.26$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.18 \times 0.16 \times 0.11$  mm

#### Data collection

Bruker APEXII CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2007)  
 $T_{\min} = 0.355$ ,  $T_{\max} = 0.502$   
 16515 measured reflections  
 3794 independent reflections  
 2599 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.031$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$   
 $wR(F^2) = 0.116$   
 $S = 1.01$   
 3794 reflections  
 227 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 1.16$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.63$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O4}-\text{H4}\cdots\text{O3}$	0.82	1.83	2.561 (5)	147
$\text{N1}-\text{H1}\cdots\text{O1A}^i$	0.86	2.29	2.966 (5)	136
$\text{N2}-\text{H2A}\cdots\text{Br2}^{ii}$	0.86	2.79	3.597 (4)	157
$\text{O4}-\text{H4}\cdots\text{Br2}^{iii}$	0.82	2.88	3.403 (3)	124

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

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: PLATON (Spek, 2009) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5126).

### References

- Bruker (2007). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Kojić-Prodić, B. & Ružić-Toroš, Ž. (1982). *Acta Cryst.* **B38**, 2948–2951.  
 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. (2007). SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.  
 Turck, D., Busch, U., Heinzl, 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.  
 Zia-ur-Rehman, M., Choudary, J. A. & Ahmad, S. (2005). *Bull. Korean Chem. Soc.* **26**, 1771–1775.

**supplementary materials**

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## 4-Hydroxy-*N*-(2,4,6-tribromophenyl)-2*H*-1,2-benzothiazine-3-carboxamide 1,1-dioxide

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### Comment

Owing to the applications of 1,2-benzothiazine 1,1-dioxides and their precursor intermediates as non-steroidal anti-inflammatory compounds (Turck *et al.*, 1996), considerable attention has been given to their synthesis. As part of a research program synthesizing 1,2-benzothiazines (Zia-ur-Rehman *et al.*, 2005), we herein report the crystal structure of the title compound, (**I**) (Scheme and figure 1).

The thiazine ring, involving two double bonds, exhibits a sofa conformation; with S1/C1/C6/C7 relatively planar and N1 showing significant departure from plane due to its pyramidal geometry. The enolic hydrogen on O4 is involved in intramolecular hydrogen bonding [O4—H4···O3] with the carbonyl oxygen at C9 giving rise to a six-membered hydrogen bond ring (Table 1). The C1—S1 [1.757 (5) Å] bond is shorter than a normal C—S single bond (1.81–2.55 Å) (Weast *et al.*, 1984) due to partial double bond character and is in agreement with similar molecules (Kojić-Prodić & Ružić-Toroš, 1982). Each molecule is linked to its adjacent one through intermolecular N—H···Br forming a centrosymmetric dimer which is further linked to the next *via* O—H···Br hydrogen bonds giving rise to a zigzag chain along *b* (Figure 2). The title molecules are also linked to each other *via* N—H···O hydrogen bonds forming dimers along *b* which further links to the adjacent dimer through O—H···Br hydrogen bonds giving rise to a zigzag chain along *b* (Figure 3).

### Experimental

A mixture of methyl 4-hydroxy-2*H*-1,2-benzothiazine-3-carboxylate-1,1-dioxide (2.55 g; 10.0 mmol), 2,3-dimethyl aniline (4.947 g; 15.0 mmol) 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: 82%.

### Refinement

All hydrogen atoms were identified in the difference map and subsequently 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 freely 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 1.2U<sub>eq</sub> of the parent atoms or 1.5U<sub>eq</sub> for methyl groups.

### Figures

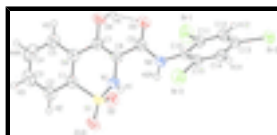


Fig. 1. The molecular structure of the title compound, with displacement ellipsoids at the 50% probability level.

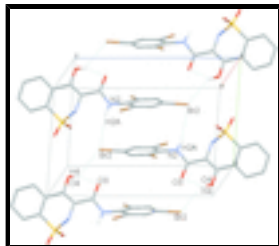


Fig. 2. Perspective view of the three-dimensional crystal packing showing N—H...Br and O—H...Br hydrogen-bonded interactions (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity.

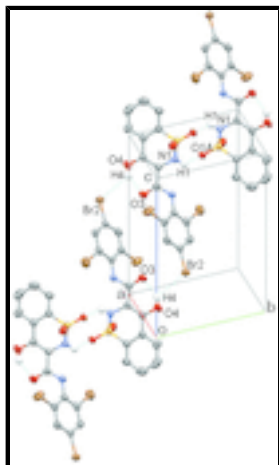


Fig. 3. Another perspective view of the three-dimensional crystal packing showing N—H...O and O—H...Br hydrogen-bonded interactions (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity.

#### 4-Hydroxy-*N*-(2,4,6-tribromophenyl)-2*H*-1,2-benzothiazine-3- carboxamide 1,1-dioxide

##### Crystal data

$C_{15}H_9Br_3N_2O_4S$

$M_r = 553.03$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 7.5082$  (4) Å

$b = 8.7486$  (6) Å

$c = 13.0669$  (9) Å

$\alpha = 83.618$  (2)°

$\beta = 86.280$  (2)°

$\gamma = 87.684$  (2)°

$V = 850.72$  (9) Å<sup>3</sup>

$Z = 2$

$F_{000} = 532$

$D_x = 2.159$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 5092 reflections

$\theta = 2.3$ – $24.2$ °

$\mu = 7.26$  mm<sup>-1</sup>

$T = 296$  K

Needle, light yellow

$0.18 \times 0.16 \times 0.11$  mm

##### Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296$  K

$\varphi$  and  $\omega$  scans

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

3794 independent reflections

2599 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.031$

$\theta_{max} = 27.6$ °

$\theta_{min} = 1.6$ °

$h = -9 \rightarrow 9$

$T_{\min} = 0.355$ ,  $T_{\max} = 0.502$   
16515 measured reflections

$k = -11 \rightarrow 11$   
 $l = -16 \rightarrow 16$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H-atom parameters constrained
$wR(F^2) = 0.116$	$w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 2.0189P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
3794 reflections	$(\Delta/\sigma)_{\max} < 0.001$
227 parameters	$\Delta\rho_{\max} = 1.16 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.63 \text{ e } \text{\AA}^{-3}$
	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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.82014 (8)	0.13368 (9)	0.54929 (5)	0.0701 (2)
Br2	0.30934 (9)	0.34516 (6)	0.26785 (4)	0.0603 (2)
Br3	0.17498 (9)	0.43843 (7)	0.68227 (5)	0.0629 (2)
C1	0.8054 (6)	0.2197 (6)	1.0575 (4)	0.0373 (11)
C2	0.8789 (7)	0.2424 (6)	1.1475 (4)	0.0462 (12)
H2	0.9024	0.3412	1.1617	0.055*
C3	0.9184 (8)	0.1144 (8)	1.2182 (5)	0.0591 (15)
H3	0.9641	0.1286	1.2808	0.071*
C4	0.8905 (7)	-0.0287 (8)	1.1956 (5)	0.0625 (17)
H4A	0.9211	-0.1127	1.2419	0.075*
C5	0.8152 (6)	-0.0538 (6)	1.1026 (4)	0.0454 (12)
H5	0.7971	-0.1533	1.0878	0.055*
C6	0.7687 (6)	0.0711 (5)	1.0339 (4)	0.0360 (10)
C7	0.6899 (6)	0.0507 (5)	0.9372 (4)	0.0364 (10)
C8	0.6194 (6)	0.1678 (5)	0.8750 (4)	0.0354 (10)
C9	0.5696 (6)	0.1459 (6)	0.7707 (4)	0.0382 (11)

## supplementary materials

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C10	0.4863 (6)	0.2808 (5)	0.6055 (4)	0.0361 (10)
C11	0.5909 (6)	0.2214 (5)	0.5271 (4)	0.0392 (11)
C12	0.5352 (7)	0.2323 (6)	0.4267 (4)	0.0430 (12)
H12	0.6034	0.1874	0.3753	0.052*
C13	0.3776 (7)	0.3105 (5)	0.4054 (4)	0.0383 (11)
C14	0.2718 (7)	0.3729 (5)	0.4799 (4)	0.0421 (12)
H14	0.1655	0.4259	0.4638	0.050*
C15	0.3259 (6)	0.3557 (5)	0.5797 (4)	0.0401 (11)
N1	0.6041 (5)	0.3207 (4)	0.9051 (3)	0.0383 (9)
H1	0.5118	0.3798	0.8931	0.046*
N2	0.5404 (6)	0.2763 (5)	0.7070 (3)	0.0430 (10)
H2A	0.5560	0.3627	0.7303	0.052*
O2	0.9238 (5)	0.3836 (4)	0.8929 (3)	0.0500 (9)
O3	0.5570 (5)	0.0166 (4)	0.7438 (3)	0.0492 (9)
O4	0.6955 (5)	-0.0922 (4)	0.9118 (3)	0.0470 (9)
H4	0.6471	-0.0940	0.8575	0.070*
O1A	0.7151 (5)	0.5147 (4)	1.0094 (3)	0.0481 (9)
S1	0.77172 (16)	0.37606 (13)	0.96331 (10)	0.0381 (3)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.0467 (3)	0.1064 (6)	0.0550 (4)	0.0271 (3)	-0.0078 (3)	-0.0070 (3)
Br2	0.0949 (5)	0.0455 (3)	0.0440 (3)	0.0073 (3)	-0.0311 (3)	-0.0082 (2)
Br3	0.0685 (4)	0.0635 (4)	0.0548 (4)	0.0185 (3)	0.0116 (3)	-0.0145 (3)
C1	0.032 (2)	0.046 (3)	0.034 (3)	0.0044 (19)	-0.0033 (19)	-0.005 (2)
C2	0.048 (3)	0.052 (3)	0.041 (3)	-0.002 (2)	-0.008 (2)	-0.011 (2)
C3	0.051 (3)	0.085 (5)	0.044 (3)	-0.008 (3)	-0.007 (3)	-0.018 (3)
C4	0.043 (3)	0.067 (4)	0.070 (4)	-0.005 (3)	-0.004 (3)	0.024 (3)
C5	0.038 (3)	0.050 (3)	0.047 (3)	-0.003 (2)	-0.003 (2)	0.005 (2)
C6	0.029 (2)	0.042 (3)	0.037 (3)	0.0009 (19)	0.0021 (19)	-0.006 (2)
C7	0.035 (2)	0.037 (3)	0.038 (3)	0.0014 (19)	0.002 (2)	-0.007 (2)
C8	0.036 (2)	0.037 (3)	0.035 (3)	0.0008 (19)	-0.0050 (19)	-0.013 (2)
C9	0.039 (3)	0.041 (3)	0.035 (3)	0.003 (2)	-0.004 (2)	-0.009 (2)
C10	0.043 (3)	0.034 (2)	0.033 (3)	-0.002 (2)	-0.007 (2)	-0.006 (2)
C11	0.043 (3)	0.038 (3)	0.037 (3)	0.004 (2)	-0.007 (2)	-0.005 (2)
C12	0.050 (3)	0.042 (3)	0.038 (3)	0.003 (2)	0.002 (2)	-0.010 (2)
C13	0.047 (3)	0.030 (2)	0.039 (3)	-0.001 (2)	-0.011 (2)	-0.006 (2)
C14	0.044 (3)	0.037 (3)	0.046 (3)	0.005 (2)	-0.010 (2)	-0.004 (2)
C15	0.043 (3)	0.035 (3)	0.042 (3)	0.000 (2)	0.005 (2)	-0.009 (2)
N1	0.040 (2)	0.037 (2)	0.041 (2)	0.0094 (17)	-0.0127 (17)	-0.0113 (18)
N2	0.062 (3)	0.036 (2)	0.033 (2)	0.0022 (19)	-0.0145 (19)	-0.0089 (18)
O2	0.048 (2)	0.050 (2)	0.050 (2)	0.0003 (16)	0.0008 (17)	0.0019 (17)
O3	0.067 (2)	0.041 (2)	0.042 (2)	-0.0016 (17)	-0.0091 (17)	-0.0152 (17)
O4	0.056 (2)	0.041 (2)	0.047 (2)	-0.0037 (16)	0.0011 (17)	-0.0202 (16)
O1A	0.053 (2)	0.045 (2)	0.052 (2)	0.0170 (16)	-0.0234 (17)	-0.0265 (17)
S1	0.0404 (6)	0.0357 (6)	0.0399 (7)	0.0042 (5)	-0.0088 (5)	-0.0094 (5)

*Geometric parameters (Å, °)*

Br1—C11	1.882 (5)	C8—C9	1.471 (6)
Br2—C13	1.890 (5)	C9—O3	1.230 (6)
Br3—C15	1.886 (5)	C9—N2	1.355 (6)
C1—C2	1.368 (7)	C10—C11	1.385 (7)
C1—C6	1.411 (7)	C10—C15	1.391 (7)
C1—S1	1.757 (5)	C10—N2	1.408 (6)
C2—C3	1.405 (8)	C11—C12	1.395 (7)
C2—H2	0.9300	C12—C13	1.371 (7)
C3—C4	1.345 (9)	C12—H12	0.9300
C3—H3	0.9300	C13—C14	1.363 (7)
C4—C5	1.415 (8)	C14—C15	1.381 (7)
C4—H4A	0.9300	C14—H14	0.9300
C5—C6	1.383 (7)	N1—S1	1.628 (4)
C5—H5	0.9300	N1—H1	0.8600
C6—C7	1.461 (7)	N2—H2A	0.8600
C7—O4	1.328 (5)	O2—S1	1.418 (4)
C7—C8	1.348 (7)	O4—H4	0.8200
C8—N1	1.435 (6)	O1A—S1	1.450 (3)
C2—C1—C6	121.6 (5)	C15—C10—N2	119.5 (4)
C2—C1—S1	119.9 (4)	C10—C11—C12	121.3 (4)
C6—C1—S1	118.2 (4)	C10—C11—Br1	121.5 (4)
C1—C2—C3	119.1 (5)	C12—C11—Br1	117.1 (4)
C1—C2—H2	120.4	C13—C12—C11	118.6 (4)
C3—C2—H2	120.4	C13—C12—H12	120.7
C4—C3—C2	120.2 (6)	C11—C12—H12	120.7
C4—C3—H3	119.9	C14—C13—C12	122.0 (5)
C2—C3—H3	119.9	C14—C13—Br2	118.1 (4)
C3—C4—C5	121.2 (6)	C12—C13—Br2	119.8 (4)
C3—C4—H4A	119.4	C13—C14—C15	118.5 (4)
C5—C4—H4A	119.4	C13—C14—H14	120.7
C6—C5—C4	119.4 (5)	C15—C14—H14	120.7
C6—C5—H5	120.3	C14—C15—C10	122.1 (4)
C4—C5—H5	120.3	C14—C15—Br3	117.9 (4)
C5—C6—C1	118.3 (5)	C10—C15—Br3	120.0 (4)
C5—C6—C7	121.3 (5)	C8—N1—S1	116.2 (3)
C1—C6—C7	120.4 (4)	C8—N1—H1	121.9
O4—C7—C8	120.9 (4)	S1—N1—H1	121.9
O4—C7—C6	115.6 (4)	C9—N2—C10	124.8 (4)
C8—C7—C6	123.4 (4)	C9—N2—H2A	117.6
C7—C8—N1	120.9 (4)	C10—N2—H2A	117.6
C7—C8—C9	121.2 (4)	C7—O4—H4	109.5
N1—C8—C9	117.7 (4)	O2—S1—O1A	117.6 (2)
O3—C9—N2	122.8 (4)	O2—S1—N1	108.5 (2)
O3—C9—C8	121.5 (4)	O1A—S1—N1	107.9 (2)
N2—C9—C8	115.8 (4)	O2—S1—C1	108.0 (2)
C11—C10—C15	117.4 (4)	O1A—S1—C1	111.6 (2)

## supplementary materials

C11—C10—N2	123.0 (4)	N1—S1—C1	101.9 (2)
C6—C1—C2—C3	-0.1 (7)	C10—C11—C12—C13	3.3 (7)
S1—C1—C2—C3	-174.8 (4)	Br1—C11—C12—C13	-172.9 (4)
C1—C2—C3—C4	2.5 (8)	C11—C12—C13—C14	-2.3 (8)
C2—C3—C4—C5	-2.3 (9)	C11—C12—C13—Br2	173.8 (4)
C3—C4—C5—C6	-0.3 (8)	C12—C13—C14—C15	-0.2 (8)
C4—C5—C6—C1	2.6 (7)	Br2—C13—C14—C15	-176.4 (4)
C4—C5—C6—C7	-179.7 (5)	C13—C14—C15—C10	1.8 (7)
C2—C1—C6—C5	-2.4 (7)	C13—C14—C15—Br3	-178.5 (4)
S1—C1—C6—C5	172.3 (3)	C11—C10—C15—C14	-0.8 (7)
C2—C1—C6—C7	179.9 (4)	N2—C10—C15—C14	175.5 (4)
S1—C1—C6—C7	-5.3 (6)	C11—C10—C15—Br3	179.5 (4)
C5—C6—C7—O4	-11.4 (6)	N2—C10—C15—Br3	-4.2 (6)
C1—C6—C7—O4	166.2 (4)	C7—C8—N1—S1	40.0 (6)
C5—C6—C7—C8	170.3 (5)	C9—C8—N1—S1	-135.0 (4)
C1—C6—C7—C8	-12.1 (7)	O3—C9—N2—C10	2.5 (7)
O4—C7—C8—N1	176.5 (4)	C8—C9—N2—C10	-178.0 (4)
C6—C7—C8—N1	-5.3 (7)	C11—C10—N2—C9	-64.1 (7)
O4—C7—C8—C9	-8.7 (7)	C15—C10—N2—C9	119.8 (5)
C6—C7—C8—C9	169.5 (4)	C8—N1—S1—O2	65.2 (4)
C7—C8—C9—O3	14.5 (7)	C8—N1—S1—O1A	-166.3 (3)
N1—C8—C9—O3	-170.5 (4)	C8—N1—S1—C1	-48.6 (4)
C7—C8—C9—N2	-165.0 (4)	C2—C1—S1—O2	93.0 (4)
N1—C8—C9—N2	10.0 (6)	C6—C1—S1—O2	-81.8 (4)
C15—C10—C11—C12	-1.8 (7)	C2—C1—S1—O1A	-37.8 (5)
N2—C10—C11—C12	-178.0 (5)	C6—C1—S1—O1A	147.4 (4)
C15—C10—C11—Br1	174.3 (4)	C2—C1—S1—N1	-152.7 (4)
N2—C10—C11—Br1	-1.9 (7)	C6—C1—S1—N1	32.4 (4)

### Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O4—H4...O3	0.82	1.83	2.561 (5)	147
N1—H1...O1A <sup>i</sup>	0.86	2.29	2.966 (5)	136
N2—H2A...Br2 <sup>ii</sup>	0.86	2.79	3.597 (4)	157
O4—H4...Br2 <sup>iii</sup>	0.82	2.88	3.403 (3)	124

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



Fig. 1

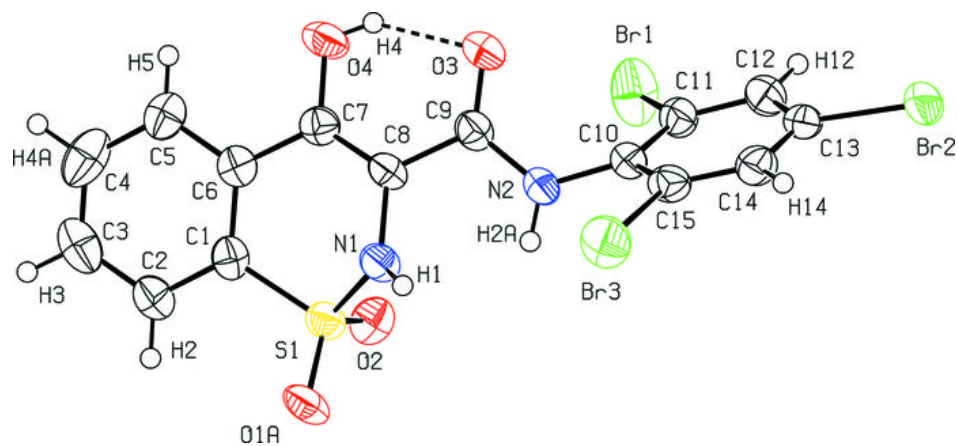


Fig. 2

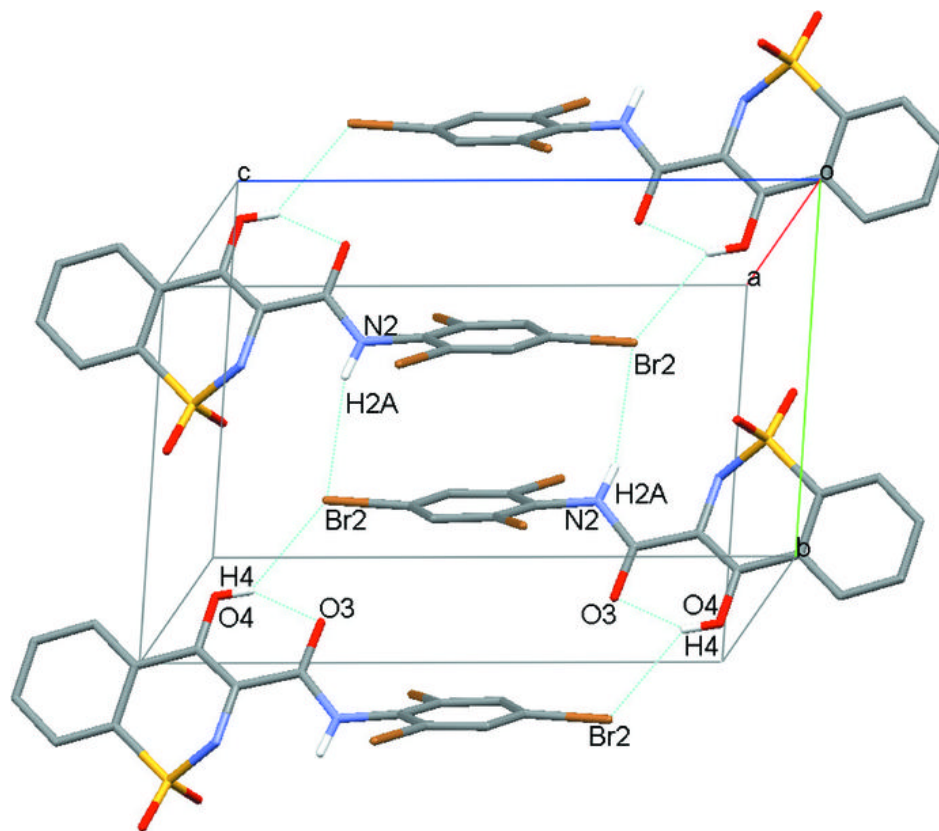


Fig. 3

