

## 4-[(2,4-Dihydroxybenzylidene)-ammonio]benzenesulfonate trihydrate

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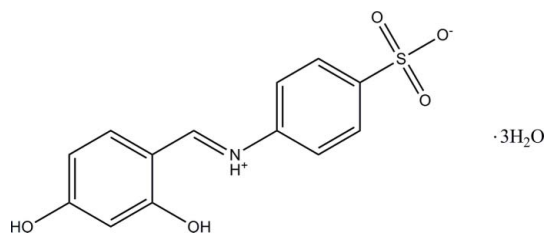
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.053;  $wR$  factor = 0.147; data-to-parameter ratio = 24.6.

The title Schiff base compound,  $\text{C}_{13}\text{H}_{11}\text{NO}_5\text{S}\cdot 3\text{H}_2\text{O}$ , formed from sulfanilic acid and 2,4-dihydroxybenzaldehyde, crystallized out as a zwitterion with the N atom protonated. The asymmetric unit consists of one 4-[(2,4-dihydroxybenzylidene)ammonio]benzenesulfonate and three water molecules. The zwitterion exists in an *E* configuration with respect to the central C=N double bond. The two benzene rings of the molecule are oriented at a dihedral angle of  $27.33$  ( $8^\circ$ ). An intramolecular N—H $\cdots$ O hydrogen bond stabilizes the molecular structure. In the crystal, the zwitterions are linked into chains along [101] by intermolecular O—H $\cdots$ O and N—H $\cdots$ O hydrogen bonds. The three water molecules link these chains into a three-dimensional framework by additional intermolecular O—H $\cdots$ O hydrogen bonds. A  $\pi\cdots\pi$  interaction [ $3.5485$  ( $9$ ) Å] further stabilizes the crystal structure.

### Related literature

For Schiff bases and their applications, see: Singh *et al.* (1975); Elmali *et al.* (1999); Patel *et al.* (1999). For details of sulfanilic acid, see: Rae & Maslen (1962); Banu & Golzar Hossain (2006); Hempel *et al.* (1999). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



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<sup>§</sup> Thomson Reuters ResearcherID: A-3561-2009.

### Experimental

#### Crystal data

$\text{C}_{13}\text{H}_{11}\text{NO}_5\text{S}\cdot 3\text{H}_2\text{O}$   
 $M_r = 347.34$   
 Triclinic,  $P\bar{1}$   
 $a = 7.7855$  (1) Å  
 $b = 9.0820$  (1) Å  
 $c = 11.8526$  (2) Å  
 $\alpha = 70.022$  (1) $^\circ$   
 $\beta = 79.271$  (1) $^\circ$

$\gamma = 76.141$  (1) $^\circ$   
 $V = 759.70$  (2) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.26$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.36 \times 0.16 \times 0.08$  mm

#### Data collection

Bruker SMART APEXII CCD  
 area-detector diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2009)  
 $T_{\min} = 0.914$ ,  $T_{\max} = 0.980$

19799 measured reflections  
 5420 independent reflections  
 4177 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.027$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$   
 $wR(F^2) = 0.147$   
 $S = 1.05$   
 5420 reflections  
 220 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.61$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.60$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å,  $^\circ$ ).

<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>
O1W—H1W1 $\cdots$ O3 <sup>i</sup>	0.89	2.17	3.020 (3)	161
O1W—H1W1 $\cdots$ O4 <sup>i</sup>	0.89	2.39	3.083 (3)	135
O1W—H2W1 $\cdots$ O2W	0.84	2.02	2.817 (4)	158
O2W—H2W2 $\cdots$ O2	0.95	1.89	2.812 (3)	161
O3W—H1W3 $\cdots$ O5 <sup>ii</sup>	0.96	1.83	2.756 (2)	161
O3W—H2W3 $\cdots$ O1W	0.86	1.86	2.701 (3)	166
N1—H1N1 $\cdots$ O1	0.86 (3)	2.07 (2)	2.6601 (18)	126 (2)
N1—H1N1 $\cdots$ O5 <sup>iii</sup>	0.86 (3)	2.19 (3)	2.948 (2)	148 (2)
O1—H1O1 $\cdots$ O3W	0.90 (3)	1.64 (4)	2.543 (2)	173 (4)
O2—H1O2 $\cdots$ O3 <sup>iv</sup>	0.80 (3)	1.85 (3)	2.627 (2)	164 (2)

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; 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 and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2719).

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**supplementary materials**

*Acta Cryst.* (2010). E66, o580-o581 [ doi:10.1107/S1600536810004526 ]

## 4-[(2,4-Dihydroxybenzylidene)ammonio]benzenesulfonate trihydrate

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### Comment

Schiff bases derived from aromatic amines and aromatic aldehydes have a wide variety of applications in many fields, *e.g.*, biological, inorganic and analytical chemistry (Singh *et al.*, 1975; Elmali *et al.*, 1999; Patel *et al.*, 1999). Schiff base compounds have been of great interest for many years. *p*-aminobenzenesulfonic acid is known as sulfanilic acid, which contains  $\text{NH}_3^+$  and  $\text{SO}_3^-$  groups. Sulfanilic acid is a salt, but of a rather special kind, called a zwitterion. It is the product of the reaction between an acidic group and a basic group that are part of the same molecule. The hydrogen is attached to nitrogen rather than oxygen simply because the  $\text{NH}_2$  group is a stronger base than the  $\text{SO}_3^-$  substituent. A zwitterionic structure was also observed in the crystal structure of sulfanilic acid monohydrate (Rae & Maslen, 1962; Banu & Golzar, 2006). The crystal structure of the Schiff base formed from sulfanilic acid and dimethylformamide has also been reported in the literature (Hempel *et al.*, 1999). The present work is part of a structural study of compounds of Schiff base systems and we report here the structure of the title compound, (I).

The 4-[(2,4-dihydroxybenzylidene)-amino]benzenesulfonic acid molecule crystallized out as a zwitterion, 4-[(2,4-dihydroxybenzylidene)-ammonio]benzenesulfonate. The asymmetric unit consists of one 4-[(2,4-dihydroxybenzylidene)-ammonio]benzenesulfonate and three water molecules (Fig. 1). The zwitterion exists in an *E* configuration with respect to the central C=N double bond. The two benzene rings [C1–C6 and C8–C13] are oriented at 27.33 (8)°. An intramolecular N1–H1N1···O1 hydrogen bond forms a six-membered ring, generating *S*(6) ring motif (Bernstein *et al.*, 1995). The zwitterions are linked into chains along [101] by intermolecular O2–H1O2···O3 and N1–H1N1···O5 hydrogen bonds. The three water molecules linked these chains into a three-dimensional framework by intermolecular O–H···O hydrogen bonds (Fig. 2, Table 2). An unusually short H2W1···H1W2 distance is also observed. A Cg1···Cg1 interaction of 3.5485 (9) Å; -*x*, 2-*y*, 1-*z*, further stabilizes the crystal structure [Cg1 is the centroid of the C1–C6 benzene ring].

### Experimental

2,4-Dihydroxybenzaldehyde (0.069 g) and sulfanilic acid (0.861 g) in ethanol/water (40 ml) were heated under reflux for 2 h with stirring. The colour of the solution gradually changed from colourless to lemon yellow. The solution was then cooled to room temperature. After few days, slow evaporation of the solvent yielded yellow crystals of compound (I).

### Refinement

The O and N bound H-atoms were located from difference Fourier map and refined freely. The H-atoms of the water molecules were located from a difference Fourier map and constrained to refine with the parent atom with  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$ . The C-bound H-atoms were positioned geometrically with a riding model with C–H = 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ .

## Figures

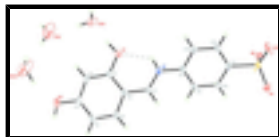


Fig. 1. The molecular structure of (I) with atom labels and 50% probability ellipsoids for non-H atoms. The intramolecular hydrogen bond is shown as a dashed line.

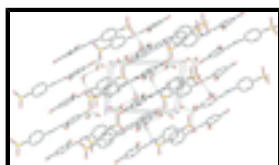


Fig. 2. The crystal packing of (I), viewed down the *b* axis, showing the molecules linked into a 3-dimensional framework. Intermolecular hydrogen bonds are shown as dashed lines.

## 4-[(2,4-Dihydroxybenzylidene)ammonio]benzenesulfonate trihydrate

### Crystal data

$C_{13}H_{11}NO_5S \cdot 3H_2O$

$M_r = 347.34$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 7.7855$  (1) Å

$b = 9.0820$  (1) Å

$c = 11.8526$  (2) Å

$\alpha = 70.022$  (1)°

$\beta = 79.271$  (1)°

$\gamma = 76.141$  (1)°

$V = 759.70$  (2) Å<sup>3</sup>

$Z = 2$

$F(000) = 364$

$D_x = 1.518$  Mg m<sup>-3</sup>

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

Cell parameters from 7701 reflections

$\theta = 2.6$ – $32.1$ °

$\mu = 0.26$  mm<sup>-1</sup>

$T = 100$  K

Block, yellow

$0.36 \times 0.16 \times 0.08$  mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan (SADABS; Bruker, 2009)

$T_{\min} = 0.914$ ,  $T_{\max} = 0.980$

19799 measured reflections

5420 independent reflections

4177 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 32.4$ °,  $\theta_{\min} = 1.8$ °

$h = -11 \rightarrow 11$

$k = -13 \rightarrow 13$

$l = -17 \rightarrow 17$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.053$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.147$

$S = 1.05$

5420 reflections

220 parameters

0 restraints

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.070P)^2 + 0.4045P]$$

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

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

$$\Delta\rho_{\max} = 0.61 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.60 \text{ e } \text{\AA}^{-3}$$

### Special details

**Experimental.** The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.71640 (6)	0.60676 (5)	1.18660 (4)	0.02504 (12)
O1	0.33982 (17)	0.77778 (14)	0.52286 (11)	0.0226 (2)
O2	0.00861 (17)	1.21132 (17)	0.24684 (11)	0.0256 (3)
O3	0.89905 (19)	0.52191 (18)	1.16735 (13)	0.0371 (3)
O4	0.71119 (3)	0.75067 (16)	1.21434 (16)	0.0530 (5)
O5	0.60457 (18)	0.50241 (15)	1.27306 (10)	0.0267 (3)
N1	0.41704 (17)	0.82152 (16)	0.71713 (11)	0.0170 (2)
C1	0.2641 (2)	0.93133 (18)	0.48282 (13)	0.0170 (3)
C2	0.1775 (2)	0.99664 (19)	0.37933 (13)	0.0190 (3)
H2A	0.1728	0.9331	0.3333	0.023*
C3	0.0980 (2)	1.15723 (19)	0.34488 (13)	0.0195 (3)
C4	0.1103 (2)	1.25788 (19)	0.40959 (14)	0.0207 (3)
H4A	0.0593	1.3658	0.3843	0.025*
C5	0.1987 (2)	1.19458 (18)	0.51051 (14)	0.0188 (3)
H5A	0.2090	1.2609	0.5528	0.023*
C6	0.2748 (2)	1.02971 (17)	0.55143 (13)	0.0162 (3)
C7	0.3521 (2)	0.97063 (18)	0.66134 (13)	0.0171 (3)
H7A	0.3572	1.0454	0.6975	0.021*
C8	0.4854 (2)	0.76888 (17)	0.83072 (13)	0.0169 (3)
C9	0.4177 (2)	0.85045 (19)	0.91434 (14)	0.0212 (3)
H9A	0.3246	0.9379	0.8979	0.025*
C10	0.4905 (2)	0.79988 (19)	1.02271 (14)	0.0219 (3)
H10A	0.4466	0.8537	1.0793	0.026*

## supplementary materials

C11	0.6291 (2)	0.66864 (18)	1.04650 (14)	0.0197 (3)
C12	0.6933 (2)	0.58417 (18)	0.96441 (14)	0.0203 (3)
H12A	0.7847	0.4954	0.9817	0.024*
C13	0.6196 (2)	0.63345 (18)	0.85627 (14)	0.0188 (3)
H13A	0.6595	0.5765	0.8014	0.023*
O1W	0.1128 (3)	0.7226 (3)	0.22066 (18)	0.0639 (6)
H1W1	0.0299	0.6832	0.2044	0.096*
H2W1	0.0754	0.8189	0.1836	0.096*
O2W	0.0858 (3)	1.0446 (3)	0.0769 (2)	0.0815 (7)
H1W2	0.1929	0.9994	0.0628	0.122*
H2W2	0.0424	1.1170	0.1224	0.122*
O3W	0.3001 (2)	0.59375 (18)	0.41308 (14)	0.0368 (3)
H1W3	0.4180	0.5558	0.3796	0.055*
H2W3	0.2254	0.6348	0.3604	0.055*
H1N1	0.415 (3)	0.747 (3)	0.689 (2)	0.041 (7)*
H1O1	0.327 (4)	0.719 (4)	0.478 (3)	0.057 (8)*
H1O2	-0.027 (3)	1.305 (3)	0.236 (2)	0.036 (7)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0369 (2)	0.01588 (18)	0.0241 (2)	-0.00608 (15)	-0.01801 (17)	-0.00021 (14)
O1	0.0310 (6)	0.0164 (5)	0.0212 (5)	-0.0008 (4)	-0.0084 (5)	-0.0063 (4)
O2	0.0257 (6)	0.0297 (7)	0.0188 (5)	-0.0058 (5)	-0.0101 (5)	-0.0001 (5)
O3	0.0291 (7)	0.0386 (8)	0.0338 (7)	-0.0045 (6)	-0.0170 (6)	0.0066 (6)
O4	0.0943 (14)	0.0183 (6)	0.0590 (10)	-0.0066 (7)	-0.0542 (10)	-0.0072 (6)
O5	0.0367 (7)	0.0239 (6)	0.0173 (5)	-0.0029 (5)	-0.0064 (5)	-0.0037 (4)
N1	0.0192 (6)	0.0167 (6)	0.0146 (5)	-0.0028 (5)	-0.0048 (4)	-0.0031 (5)
C1	0.0172 (6)	0.0175 (6)	0.0165 (6)	-0.0047 (5)	-0.0019 (5)	-0.0047 (5)
C2	0.0198 (7)	0.0228 (7)	0.0158 (6)	-0.0070 (5)	-0.0033 (5)	-0.0051 (5)
C3	0.0176 (7)	0.0241 (7)	0.0145 (6)	-0.0066 (5)	-0.0037 (5)	-0.0004 (5)
C4	0.0208 (7)	0.0186 (7)	0.0195 (7)	-0.0035 (5)	-0.0043 (6)	-0.0008 (5)
C5	0.0219 (7)	0.0161 (6)	0.0175 (6)	-0.0034 (5)	-0.0042 (5)	-0.0033 (5)
C6	0.0177 (6)	0.0159 (6)	0.0145 (6)	-0.0032 (5)	-0.0035 (5)	-0.0031 (5)
C7	0.0193 (7)	0.0165 (6)	0.0152 (6)	-0.0035 (5)	-0.0047 (5)	-0.0031 (5)
C8	0.0174 (6)	0.0165 (6)	0.0150 (6)	-0.0039 (5)	-0.0044 (5)	-0.0012 (5)
C9	0.0247 (7)	0.0184 (7)	0.0182 (7)	0.0005 (6)	-0.0065 (6)	-0.0039 (5)
C10	0.0288 (8)	0.0176 (7)	0.0183 (7)	-0.0018 (6)	-0.0067 (6)	-0.0038 (5)
C11	0.0234 (7)	0.0158 (6)	0.0194 (7)	-0.0068 (5)	-0.0082 (6)	0.0004 (5)
C12	0.0192 (7)	0.0170 (7)	0.0211 (7)	-0.0025 (5)	-0.0063 (5)	-0.0002 (5)
C13	0.0195 (7)	0.0176 (6)	0.0178 (6)	-0.0031 (5)	-0.0025 (5)	-0.0038 (5)
O1W	0.0496 (11)	0.1022 (17)	0.0610 (12)	-0.0363 (11)	-0.0095 (9)	-0.0358 (12)
O2W	0.0746 (15)	0.120 (2)	0.0831 (16)	-0.0358 (15)	0.0051 (12)	-0.0697 (16)
O3W	0.0402 (8)	0.0364 (7)	0.0420 (8)	-0.0097 (6)	0.0007 (6)	-0.0238 (6)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

S1—O4	1.4441 (14)	C5—H5A	0.9300
S1—O5	1.4512 (14)	C6—C7	1.417 (2)

S1—O3	1.4636 (15)	C7—H7A	0.9300
S1—C11	1.7706 (16)	C8—C9	1.389 (2)
O1—C1	1.3325 (18)	C8—C13	1.394 (2)
O1—H1O1	0.90 (3)	C9—C10	1.388 (2)
O2—C3	1.3500 (18)	C9—H9A	0.9300
O2—H1O2	0.80 (3)	C10—C11	1.390 (2)
N1—C7	1.3083 (19)	C10—H10A	0.9300
N1—C8	1.4247 (19)	C11—C12	1.389 (2)
N1—H1N1	0.85 (3)	C12—C13	1.390 (2)
C1—C2	1.389 (2)	C12—H12A	0.9300
C1—C6	1.422 (2)	C13—H13A	0.9300
C2—C3	1.390 (2)	O1W—H1W1	0.8863
C2—H2A	0.9300	O1W—H2W1	0.8403
C3—C4	1.408 (2)	O2W—H1W2	0.8500
C4—C5	1.372 (2)	O2W—H2W2	0.9519
C4—H4A	0.9300	O3W—H1W3	0.9617
C5—C6	1.419 (2)	O3W—H2W3	0.8594
O4—S1—O5	113.78 (10)	C7—C6—C1	123.35 (13)
O4—S1—O3	111.65 (11)	C5—C6—C1	118.67 (13)
O5—S1—O3	111.87 (8)	N1—C7—C6	126.60 (14)
O4—S1—C11	106.23 (8)	N1—C7—H7A	116.7
O5—S1—C11	106.54 (8)	C6—C7—H7A	116.7
O3—S1—C11	106.16 (8)	C9—C8—C13	121.03 (14)
C1—O1—H1O1	114.3 (19)	C9—C8—N1	120.33 (13)
C3—O2—H1O2	106.3 (18)	C13—C8—N1	118.64 (13)
C7—N1—C8	123.83 (13)	C10—C9—C8	119.21 (14)
C7—N1—H1N1	121.2 (17)	C10—C9—H9A	120.4
C8—N1—H1N1	114.9 (17)	C8—C9—H9A	120.4
O1—C1—C2	123.11 (14)	C9—C10—C11	119.89 (15)
O1—C1—C6	116.94 (13)	C9—C10—H10A	120.1
C2—C1—C6	119.94 (14)	C11—C10—H10A	120.1
C1—C2—C3	119.77 (14)	C12—C11—C10	120.89 (14)
C1—C2—H2A	120.1	C12—C11—S1	120.66 (12)
C3—C2—H2A	120.1	C10—C11—S1	118.41 (12)
O2—C3—C2	116.77 (15)	C11—C12—C13	119.41 (14)
O2—C3—C4	121.90 (15)	C11—C12—H12A	120.3
C2—C3—C4	121.33 (14)	C13—C12—H12A	120.3
C5—C4—C3	119.07 (14)	C12—C13—C8	119.48 (14)
C5—C4—H4A	120.5	C12—C13—H13A	120.3
C3—C4—H4A	120.5	C8—C13—H13A	120.3
C4—C5—C6	121.14 (14)	H1W1—O1W—H2W1	97.0
C4—C5—H5A	119.4	H1W2—O2W—H2W2	127.8
C6—C5—H5A	119.4	H1W3—O3W—H2W3	113.2
C7—C6—C5	117.94 (13)		
O1—C1—C2—C3	178.43 (14)	C7—N1—C8—C13	-150.63 (15)
C6—C1—C2—C3	-1.5 (2)	C13—C8—C9—C10	2.8 (2)
C1—C2—C3—O2	-176.86 (14)	N1—C8—C9—C10	-177.92 (15)
C1—C2—C3—C4	3.1 (2)	C8—C9—C10—C11	-0.2 (2)



## supplementary materials

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O2—C3—C4—C5	178.14 (14)	C9—C10—C11—C12	-1.7 (3)
C2—C3—C4—C5	-1.8 (2)	C9—C10—C11—S1	-179.29 (13)
C3—C4—C5—C6	-1.1 (2)	O4—S1—C11—C12	146.08 (15)
C4—C5—C6—C7	-175.27 (15)	O5—S1—C11—C12	-92.28 (15)
C4—C5—C6—C1	2.5 (2)	O3—S1—C11—C12	27.10 (16)
O1—C1—C6—C7	-3.5 (2)	O4—S1—C11—C10	-36.33 (17)
C2—C1—C6—C7	176.45 (14)	O5—S1—C11—C10	85.31 (14)
O1—C1—C6—C5	178.82 (14)	O3—S1—C11—C10	-155.30 (14)
C2—C1—C6—C5	-1.2 (2)	C10—C11—C12—C13	1.1 (2)
C8—N1—C7—C6	-176.86 (14)	S1—C11—C12—C13	178.61 (12)
C5—C6—C7—N1	174.98 (15)	C11—C12—C13—C8	1.5 (2)
C1—C6—C7—N1	-2.7 (3)	C9—C8—C13—C12	-3.4 (2)
C7—N1—C8—C9	30.1 (2)	N1—C8—C13—C12	177.29 (14)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1W1 $\cdots$ O3 <sup>i</sup>	0.89	2.17	3.020 (3)	161.
O1W—H1W1 $\cdots$ O4 <sup>i</sup>	0.89	2.39	3.083 (3)	135.
O1W—H2W1 $\cdots$ O2W	0.84	2.02	2.817 (4)	158.
O2W—H2W2 $\cdots$ O2	0.95	1.89	2.812 (3)	161.
O3W—H1W3 $\cdots$ O5 <sup>ii</sup>	0.96	1.83	2.756 (2)	161.
O3W—H2W3 $\cdots$ O1W	0.86	1.86	2.701 (3)	166.
N1—H1N1 $\cdots$ O1	0.86 (3)	2.07 (2)	2.6601 (18)	126 (2)
N1—H1N1 $\cdots$ O5 <sup>iii</sup>	0.86 (3)	2.19 (3)	2.948 (2)	148 (2)
O1—H1O1 $\cdots$ O3W	0.90 (3)	1.64 (4)	2.543 (2)	173 (4)
O2—H1O2 $\cdots$ O3 <sup>iv</sup>	0.80 (3)	1.85 (3)	2.627 (2)	164 (2)

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

Fig. 1

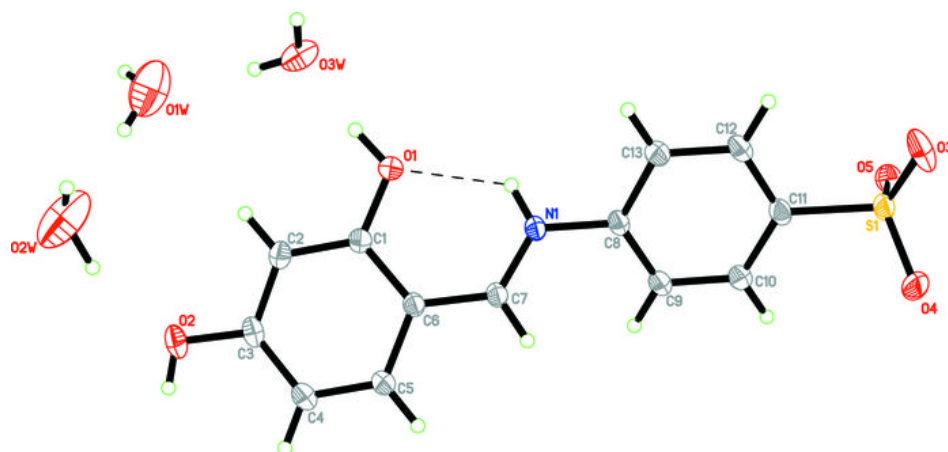


Fig. 2

