metal-organic compounds

Disodium 5,5′-diamino-2,2′-ethylene-dibenzenesulfonate tetrahydrate

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In the title disodium salt, $2Na^+ \cdot C_{14}H_{12}N_2O_6S_2{}^{2-} \cdot 4H_2O$, the two aryl units of the centrosymmetric anion are coplanar. The E geometric isomer was exclusively found, while the mean plane of the unsaturated group is twisted by 10.1 (2)° from the mean plane described by the two aminobenzenes. Four water molecules cocrystallize and participate in intermolecular hydrogen bonding. The anions lie in parallel planes separated by 3.367 (16) Å and their symmetry-related benzene rings are separated by 3.84 (1) Å, leading to weak intermolecular π-stacking.

Related literature

For general background, see: Dufresne *et al.* (2007); Pérez Guarìn *et al.* (2007) and references therein; Zhao *et al.* (1996). For related literature, see: Wang *et al.* (2005); Zhang *et al.* (2005); Etter (1990).
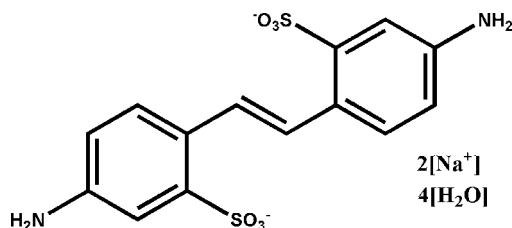
Experimental

Crystal data

$2Na^+ \cdot C_{14}H_{12}N_2O_6S_2{}^{2-} \cdot 4H_2O$
$M_r = 486.43$
Monoclinic, $P2_1/c$
$a = 8.1415$ (6) Å
$b = 18.6885$ (15) Å
$c = 6.3230$ (5) Å
$\beta = 99.860$ (4)°

$V = 947.85$ (13) Å^3
$Z = 2$
Cu $K\alpha$ radiation
$\mu = 3.56$ mm^{-1}
$T = 220$ (2) K
$0.18 \times 0.18 \times 0.07$ mm

Data collection

Bruker SMART 2000 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.560$, $T_{max} = 0.779$

4637 measured reflections
1778 independent reflections
1617 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.186$
$S = 1.07$
1778 reflections
154 parameters
9 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.79$ e Å^{-3}
$\Delta\rho_{min} = -0.77$ e Å^{-3}

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H4A$\cdots$N1^i	0.83 (2)	2.015 (10)	2.818 (3)	163 (3)
O5—H5A$\cdots$O2^{ii}	0.83 (2)	2.26 (3)	2.942 (3)	139 (4)
O5—H5B$\cdots$O2^{iii}	0.83 (3)	2.11 (3)	2.915 (3)	164 (4)
N1—H1A$\cdots$O3^{iii}	0.87 (2)	2.449 (17)	3.236 (3)	151 (3)
N1—H1B$\cdots$O5^{iv}	0.87 (3)	2.58 (3)	3.403 (3)	158 (2)

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

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *UdMX* (Marris, 2004).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AV3102).

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supplementary materials

Disodium 5,5'-diamino-2,2'-ethylenedibenzenesulfonate tetrahydrate

S. Dufresne, M. Gaultois and W. G. Skene

Comment

Stilbenes are an interesting class of compounds that can be photoexcited with standard UV irradiation sources because of their high degree of conjugation. Even though diamininostilbene (I) is an extended stilbene, photoinduced conversion does not proceed resulting in high fluorescence (Zhao *et al.*, 1996). Owing to our on-going azomethine (–N=C–) research, (Pérez Guarìn *et al.*, 2007), we examined (I) as a novel monomer for the synthesis of unprecedented water soluble conjugated functional materials. (Dufresne *et al.*, 2007) Its diamine residues are suitable for condensation with aryl aldehydes for preparation of the pi-conjugated compounds. During the course of this research we isolated (I), whose crystallographic study is discussed herein. This study is of importance for assigning the geometric isomer, co-planarity of the stilbene moiety, and cation exchange, all of which cannot be unequivocally confirmed by standard characterization techniques such as ^{1}H and ^{13}C NMR.

The resulting crystal structure found for (I) confirmed that the compound contained the stilbenoid structure with the two sulfonate groups in the 2,2' positions. Of importance, the exchange of the original two H atoms with sodium cations on the two sulfonic acid groups was confirmed from the X-ray analysis. Four water molecules were found to coordinate with the sulfonic moieties to further stabilize the charges of the ionic groups. Confirmation of the amine substitution in the *para* position of the stilbene was also possible. The exclusive formation of the thermodynamic *E* isomer was also confirmed.

Even though stilbenes are generally understood to be completely planar, the structure obtained for (I) illustrates this is not the case. The mean plane described by the phenyl rings is rotated 10.14 (24) ° from the mean plane of the unsaturated alkene to which they are covalently bonded. For comparison, the angle described by similar planes for analogous unsubstituted stilbenes is much smaller ranging between 2.7 (4) ° (Wang *et al.*, 2005) and 1.9 (3) ° (Zhang *et al.*, 2005). The larger degree of twisting from planarity for the phenyl rings of (I) is a result of the bulky sulfonic groups that reduce the amount of intermolecular *p*-stacking.

Besides the cation required to counter balance the sulfonate group, three additional sodium cations were found in close proximity to the ionic group. The distance separating the sodium cations of adjacent molecules is 3.588 (1) Å. One counter cation is located 2.539 (2) Å from the anionic O3 while the two other cations are 2.399 (2) and 2.421 (2) Å from O1. All the cations are within sufficient proximity to be described as counter ions of the sulfonate group. The sodium cations are symmetry related and the negative charge is uniformly spread over all oxygen atoms of the sulfonate. There are additionally two water molecules per sodium cation (Fig. 2) that are co-crystallized within the structure. These solvate the sodium cations and are located 2.419 (2) and 2.383 (3) Å from the cation.

In addition to these ionic interactions, hydrogen bonding also occurs in an anticipated manner predicted by Etter's topological descriptors (Etter, 1990). Three such hydrogen bonds take place between the hydrogen donors of the co-crystallized water molecules and (I), represented in Fig. 3. Specifically the interactions are: O4—H4A⋯N1[ii], O5—H5A⋯O2[iii] and O5—H5B⋯O2[iv] whose lengths are 2.818 (3), 2.942 (3), and 2.915 (3) Å, respectively. The amino groups act both as

donors and acceptors *via* the hydrogen and its nitrogen to form N—H···O and H···N interactions, respectively. Such hydrogen bonding occurs *via* N1—H1A···O3^{v} of the sulfonate and N1—H1B···O5^{v} of a water molecule. The distances of these hydrogen bonds are 3.236 (3) and 3.403 (3) Å, respectively. Each hydrogen present in (I) participates in hydrogen bonds with the exception for H4B.

Overlapping of the aryl groups resulting in pi-stacking normally takes place with conjugated aromatic compounds such as stilbenes. This is not entirely the case with (I). While the distance of 3.367 (16) Å separating similarly parallel molecules of (I) is ideal for pi-stacking, such a strong interaction is not expected because the aryl units are not completely superimposed. However, there is some overlap between the symmetrically related phenyl rings C2—C7 whose distance between the ring centroids is 3.84 (1) Å. This leads to weaker pi-stacking in comparison to its unsubstituted analogue.

Experimental

The commercially available 4,4'-diamino-2,2'-stilbene-disulfonic acid from Aldrich was dissolved in a NaOH solution. It was then purified by successive acid and base washes. The desired water soluble product was obtained by adjusting the pH ≥ 8 with NaOH and then it was recrystallized by slow evaporation in ethanol.

Refinement

H atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2\ U_{eq}(C)$. Some special constraints were added for hydrogen on nitrogen (N—H = 0.87 /%A) with $U_{iso}(H) = 1.5\ U_{eq}(C)$ and for hydrogen on oxygen (O—H = 0.83 /%A) with $U_{iso}(H) = 1.5\ U_{eq}(C)$. More constraints were used on these protons to get more realistic angle for amine and water protons.

Figures
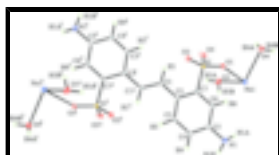
Fig. 1. *ORTEP* representation of (I) with the numbering scheme adopted (Farrugia 1997). Ellipsoids drawn at 30% probability level. [Symmetry code: (i) −*x*, 1 − *y*, −*z*.]
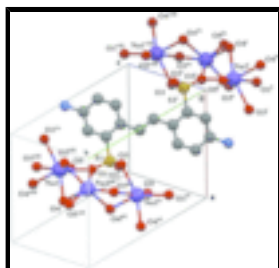
Fig. 2. The three-dimensional network demonstrating the well arranged ionic assembly between sulfonate group, sodium cations and water molecule. Hydrogen atoms have been omitted for clarity. [Symmetry codes: (i) −*x*, 1 − *y*, −*z*; (iv) *x*, *y*, 1 + *z*; (viii) −*x*, 1 − *y*, 1 − *z*; (ix) −*x*, −1/2 + *y*, 1/2 − *z*; (x) −*x*, −1/2 + *y*, −1/2 + *z*; (xi) *x*, *y*, −1 + *z*; (xii) *x*, 1.5 − *y*, −1/2 + *z*; (xiii) *x*, 1.5 − *y*, 1/2 + *z*.]

Fig. 3. Supramolecular structure showing the intermolecular H-bonding giving the structural arrangement. Dashed lines indicate hydrogen bonds. Only H-bonding hydrogen atoms are shown for clarity. [Symmetry codes: (i) −*x*, 1 − *y*, −*z*; (ii) 1 + *x*, *y*, *z*; (iii) 1 − *x*, *y*, *z*; (iv) *x*, *y*, 1 + *z*; (v) 1 − *x*, 1 − *y*, 1 − *z*; (vi) 1 − *x*, 1 − *y*, −*z*; (vii) 1 + *x*, *y*, 1 + *z*; (viii) −*x*, 1 − *y*, 1 − *z*.]

Disodium 5,5'-diamino-2,2'-ethylenedibenzenesulfonate tetrahydrate

Crystal data

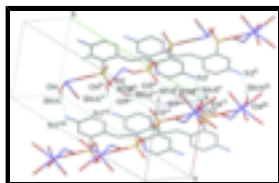|	
$2Na^+ \cdot C_{14}H_{12}N_2O_6S_2^{2-} \cdot 4H_2O$	$F_{000} = 504$
$M_r = 486.43$	$D_x = 1.704$ Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation $\lambda = 1.54178$ Å
Hall symbol: -P 2ybc	Cell parameters from 2942 reflections
$a = 8.1415$ (6) Å	$\theta = 4.7–72.1º$
$b = 18.6885$ (15) Å	$\mu = 3.56$ mm^{-1}
$c = 6.3230$ (5) Å	$T = 220$ (2) K
$\beta = 99.860$ (4)º	Block, orange
$V = 947.85$ (13) Å^3	$0.18 \times 0.18 \times 0.07$ mm
$Z = 2$	

Data collection

Bruker SMART 2000 diffractometer	1778 independent reflections
Radiation source: X-ray Sealed Tube	1617 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.049$
Detector resolution: 5.5 pixels mm^{-1}	$\theta_{max} = 72.1º$
$T = 220(2)$ K	$\theta_{min} = 4.7º$
ω scans	$h = -8 \rightarrow 10$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -23 \rightarrow 21$
$T_{min} = 0.560$, $T_{max} = 0.779$	$l = -7 \rightarrow 7$
4637 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
$R[F^2 > 2\sigma(F^2)] = 0.068$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.186$	$w = 1/[\sigma^2(F_o^2) + (0.1547P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.001$
1778 reflections	$\Delta\rho_{max} = 0.79$ e Å^{-3}
154 parameters	$\Delta\rho_{min} = -0.77$ e Å^{-3}
9 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

supplementary materials

Special details

Experimental. X-ray crystallographic data for I were collected from a single-crystal sample, which was mounted on a loop fiber. Data were collected using a Bruker Platform diffractometer, equipped with a Bruker *SMART* 2 K Charged-Coupled Device (CCD) Area Detector using the program *SMART* and normal focus sealed tube source graphite monochromated Cu—Kα radiation. The crystal-to-detector distance was 4.908 cm, and the data collection was carried out in 512 *x* 512 pixel mode, utilizing 4 *x* 4 pixel binning. The initial unit-cell parameters were determined by a least-squares fit of the angular setting of strong reflections, collected by a 9.0 degree scan in 30 frames over four different parts of the reciprocal space (120 frames total). One complete sphere of data was collected, to better than 0.8Å resolution. Upon completion of the data collection, the first 101 frames were recollected in order to improve the decay correction analysis.

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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 ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$
Na1	0.47929 (13)	0.70461 (6)	0.82314 (16)	0.0220 (3)
S1	0.31622 (6)	0.63408 (3)	0.27575 (8)	0.0134 (3)
O1	0.4039 (2)	0.68007 (10)	0.4429 (3)	0.0235 (5)
O2	0.4037 (2)	0.56729 (10)	0.2570 (3)	0.0233 (5)
O3	0.2760 (2)	0.67132 (12)	0.0699 (3)	0.0249 (5)
O4	0.6775 (2)	0.78332 (11)	0.6530 (3)	0.0232 (5)
H4A	0.759 (2)	0.7566 (12)	0.688 (5)	0.035*
H4B	0.697 (4)	0.8187 (10)	0.733 (4)	0.035*
O5	0.5550 (3)	0.58184 (12)	0.8734 (4)	0.0391 (6)
H5A	0.527 (6)	0.5482 (13)	0.789 (4)	0.059*
H5B	0.522 (5)	0.5698 (19)	0.985 (3)	0.059*
N1	−0.0974 (3)	0.67174 (14)	0.7935 (3)	0.0226 (5)
H1A	−0.010 (2)	0.6876 (17)	0.879 (3)	0.034*
H1B	−0.168 (3)	0.6459 (17)	0.849 (4)	0.034*
C1	0.0441 (3)	0.52710 (13)	0.0485 (4)	0.0160 (5)
H1	0.1357	0.5430	−0.0112	0.019*
C2	0.0102 (3)	0.56404 (12)	0.2411 (4)	0.0138 (5)
C3	−0.1390 (3)	0.55170 (14)	0.3206 (4)	0.0179 (5)
H3	−0.2168	0.5190	0.2484	0.021*
C4	−0.1748 (3)	0.58591 (14)	0.5005 (4)	0.0188 (6)
H4	−0.2759	0.5766	0.5481	0.023*
C5	−0.0613 (3)	0.63444 (13)	0.6121 (4)	0.0158 (6)
C6	0.0858 (3)	0.64861 (14)	0.5362 (4)	0.0165 (5)
H6	0.1616	0.6823	0.6071	0.020*

C7	0.1217 (3)	0.61327 (13)	0.3559 (3)	0.0126 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na1	0.0202 (6)	0.0241 (6)	0.0203 (6)	−0.0008 (4)	−0.0003 (4)	−0.0023 (4)
S1	0.0093 (4)	0.0144 (4)	0.0157 (4)	−0.00317 (18)	−0.0002 (3)	−0.00306 (18)
O1	0.0183 (9)	0.0256 (10)	0.0263 (10)	−0.0104 (8)	0.0035 (8)	−0.0093 (7)
O2	0.0164 (9)	0.0205 (10)	0.0337 (10)	0.0007 (7)	0.0066 (8)	−0.0051 (7)
O3	0.0194 (9)	0.0303 (11)	0.0246 (10)	−0.0028 (8)	0.0032 (8)	0.0092 (8)
O4	0.0189 (9)	0.0234 (10)	0.0242 (10)	0.0016 (7)	−0.0051 (7)	−0.0037 (7)
O5	0.0607 (16)	0.0227 (11)	0.0404 (12)	0.0024 (10)	0.0267 (12)	−0.0028 (9)
N1	0.0217 (11)	0.0282 (12)	0.0179 (11)	0.0057 (9)	0.0029 (9)	−0.0026 (8)
C1	0.0125 (11)	0.0186 (11)	0.0163 (11)	−0.0034 (9)	0.0007 (9)	−0.0018 (9)
C2	0.0128 (11)	0.0137 (11)	0.0134 (11)	−0.0010 (8)	−0.0023 (9)	0.0014 (8)
C3	0.0103 (11)	0.0206 (12)	0.0210 (12)	−0.0045 (9)	−0.0019 (9)	0.0005 (9)
C4	0.0116 (11)	0.0232 (14)	0.0215 (12)	0.0000 (9)	0.0022 (10)	0.0028 (10)
C5	0.0146 (12)	0.0177 (13)	0.0143 (12)	0.0060 (8)	0.0002 (9)	0.0031 (8)
C6	0.0132 (11)	0.0179 (11)	0.0162 (12)	0.0011 (9)	−0.0036 (9)	−0.0029 (9)
C7	0.0093 (10)	0.0133 (11)	0.0140 (11)	−0.0001 (8)	−0.0012 (8)	0.0010 (8)

Geometric parameters (Å, °)

Na1—O5	2.383 (3)	N1—H1a	0.87 (2)
Na1—O1	2.421 (2)	N1—H1b	0.87 (3)
Na1—O4	2.553 (2)	C1—C2	1.467 (3)
C1—C1^i	1.329 (3)	C1—H1	0.94
S1—O1	1.4516 (18)	C2—C7	1.404 (3)
S1—O2	1.452 (2)	C2—C3	1.411 (3)
S1—O3	1.4627 (19)	C3—C4	1.378 (4)
S1—C7	1.787 (2)	C3—H3	0.94
O4—H4a	0.83 (2)	C4—C5	1.397 (4)
O4—H4b	0.83 (2)	C4—H4	0.94
O5—H5a	0.83 (2)	C5—C6	1.390 (4)
O5—H5b	0.83 (3)	C6—C7	1.391 (3)
N1—C5	1.415 (3)	C6—H6	0.94
O5—NA1—O1^{ii}	153.80 (8)	O3—S1—C7	106.35 (11)
O5—NA1—O4^{ii}	81.70 (9)	O2—S1—NA1^{iv}	126.86 (8)
O1^{ii}—NA1—O4^{ii}	79.22 (7)	O3—S1—NA1^{iv}	70.74 (9)
O5—NA1—O1	88.05 (8)	C7—S1—NA1^{iv}	122.52 (8)
O1^{ii}—NA1—O1	116.66 (8)	S1—O1—NA1^{iv}	115.00 (11)
O4^{ii}—NA1—O1	153.13 (8)	S1—O1—NA1	147.64 (12)
O5—NA1—O3^{iii}	82.13 (8)	NA1^{iv}—O1—NA1	96.21 (7)
O1^{ii}—NA1—O3^{iii}	78.29 (8)	S1—O3—NA1^v	126.38 (11)
O4^{ii}—NA1—O3^{iii}	84.37 (7)	NA1^{iv}—O4—NA1	92.32 (7)
O1—NA1—O3^{iii}	118.86 (7)	NA1^{iv}—O4—H4A	132 (2)

O5—NA1—O4	116.31 (8)	NA1—O4—H4A	94 (2)
O1^{ii}—NA1—O4	80.20 (7)	NA1^{iv}—O4—H4B	118.70 (19)
O4^{ii}—NA1—O4	86.16 (7)	NA1—O4—H4B	106 (3)
O1—NA1—O4	76.22 (7)	H4A—O4—H4B	105 (3)
O3^{iii}—NA1—O4	157.77 (8)	NA1—O5—H5A	127 (3)
O5—NA1—S1^{ii}	170.56 (7)	NA1—O5—H5B	105 (3)
O1^{ii}—NA1—S1^{ii}	23.59 (5)	H5A—O5—H5B	104 (3)
O4^{ii}—NA1—S1^{ii}	101.10 (6)	C5—N1—H1A	114 (2)
O1—NA1—S1^{ii}	93.06 (6)	C5—N1—H1B	107 (2)
O3^{iii}—NA1—S1^{ii}	89.15 (6)	H1A—N1—H1B	118 (2)
O4—NA1—S1^{ii}	73.01 (5)	C1^{i}—C1—C2	125.3 (3)
O5—NA1—NA1^{iv}	122.26 (6)	C1^{i}—C1—H1	117.3
O1^{ii}—NA1—NA1^{iv}	83.80 (6)	C2—C1—H1	117.3
O4^{ii}—NA1—NA1^{iv}	127.95 (6)	C7—C2—C3	116.3 (2)
O1—NA1—NA1^{iv}	41.65 (5)	C7—C2—C1	122.8 (2)
O3^{iii}—NA1—NA1^{iv}	139.09 (5)	C3—C2—C1	121.0 (2)
O4—NA1—NA1^{iv}	42.36 (5)	C4—C3—C2	122.3 (2)
S1^{ii}—NA1—NA1^{iv}	63.06 (3)	C4—C3—H3	118.9
O5—NA1—NA1^{ii}	112.13 (6)	C2—C3—H3	118.9
O1^{ii}—NA1—NA1^{ii}	42.13 (5)	C3—C4—C5	120.2 (2)
O4^{ii}—NA1—NA1^{ii}	45.32 (5)	C3—C4—H4	119.9
O1—NA1—NA1^{ii}	157.86 (7)	C5—C4—H4	119.9
O3^{iii}—NA1—NA1^{ii}	58.39 (5)	C6—C5—C4	119.0 (2)
O4—NA1—NA1^{ii}	101.20 (6)	C6—C5—N1	119.8 (2)
S1^{ii}—NA1—NA1^{ii}	65.53 (3)	C4—C5—N1	121.2 (2)
NA1^{iv}—NA1—NA1^{ii}	123.56 (6)	C5—C6—C7	120.4 (2)
O1—S1—O2	112.45 (12)	C5—C6—H6	119.8
O1—S1—O3	111.93 (13)	C7—C6—H6	119.8
O2—S1—O3	112.00 (12)	C6—C7—C2	121.8 (2)
O1—S1—C7	105.75 (11)	C6—C7—S1	116.73 (18)
O2—S1—C7	107.88 (11)	C2—C7—S1	121.45 (17)
O2—S1—O1—NA1^{iv}	120.85 (12)	O4^{ii}—NA1—O4—NA1^{iv}	−171.25 (9)
O3—S1—O1—NA1^{iv}	−6.25 (16)	O1—NA1—O4—NA1^{iv}	29.20 (7)
C7—S1—O1—NA1^{iv}	−121.64 (11)	O3^{iii}—NA1—O4—NA1^{iv}	−106.34 (19)
O2—S1—O1—NA1	−75.6 (2)	S1^{ii}—NA1—O4—NA1^{iv}	−68.41 (5)
O3—S1—O1—NA1	157.3 (2)	NA1^{ii}—NA1—O4—NA1^{iv}	−128.27 (6)
C7—S1—O1—NA1	41.9 (3)	C1^{i}—C1—C2—C7	169.8 (3)
NA1^{iv}—S1—O1—NA1	163.5 (3)	C1^{i}—C1—C2—C3	−10.4 (5)
O5—NA1—O1—S1	47.6 (2)	C7—C2—C3—C4	0.1 (4)
O1^{ii}—NA1—O1—S1	−123.28 (18)	C1—C2—C3—C4	−179.6 (2)
O4^{ii}—NA1—O1—S1	114.9 (2)	C2—C3—C4—C5	−0.4 (4)
O3^{iii}—NA1—O1—S1	−32.3 (3)	C3—C4—C5—C6	1.3 (4)

O4—NA1—O1—S1	165.3 (2)	C3—C4—C5—N1	178.2 (2)
S1^{ii}—NA1—O1—S1	−123.0 (2)	C4—C5—C6—C7	−2.0 (4)
NA1^{iv}—NA1—O1—S1	−165.0 (3)	N1—C5—C6—C7	−178.9 (2)
NA1^{ii}—NA1—O1—S1	−108.8 (3)	C5—C6—C7—C2	1.8 (4)
O5—NA1—O1—NA1^{iv}	−147.35 (8)	C5—C6—C7—S1	−178.65 (18)
O1^{ii}—NA1—O1—NA1^{iv}	41.75 (14)	C3—C2—C7—C6	−0.8 (3)
O4^{ii}—NA1—O1—NA1^{iv}	−80.11 (18)	C1—C2—C7—C6	178.9 (2)
O3^{iii}—NA1—O1—NA1^{iv}	132.75 (8)	C3—C2—C7—S1	179.65 (18)
O4—NA1—O1—NA1^{iv}	−29.64 (7)	C1—C2—C7—S1	−0.6 (3)
S1^{ii}—NA1—O1—NA1^{iv}	42.03 (7)	O1—S1—C7—C6	6.5 (2)
NA1^{ii}—NA1—O1—NA1^{iv}	56.23 (19)	O2—S1—C7—C6	127.03 (19)
O1—S1—O3—NA1^{v}	73.67 (18)	O3—S1—C7—C6	−112.6 (2)
O2—S1—O3—NA1^{v}	−53.67 (17)	NA1^{iv}—S1—C7—C6	−35.4 (2)
C7—S1—O3—NA1^{v}	−171.30 (13)	O1—S1—C7—C2	−173.98 (19)
NA1^{iv}—S1—O3—NA1^{v}	69.30 (12)	O2—S1—C7—C2	−53.4 (2)
O5—NA1—O4—NA1^{iv}	109.98 (9)	O3—S1—C7—C2	66.9 (2)
O1^{ii}—NA1—O4—NA1^{iv}	−91.53 (7)	NA1^{iv}—S1—C7—C2	144.13 (16)

Symmetry codes: (i) −x, −y+1, −z; (ii) x, −y+3/2, z+1/2; (iii) x, y, z+1; (iv) x, −y+3/2, z−1/2; (v) x, y, z−1.

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O4—H4A⋯N1^{vi}	0.83 (2)	2.015 (10)	2.818 (3)	163 (3)
O5—H5A⋯O2^{vii}	0.83 (2)	2.26 (3)	2.942 (3)	139 (4)
O5—H5B⋯O2^{iii}	0.83 (3)	2.11 (3)	2.915 (3)	164 (4)
N1—H1A⋯O3^{iii}	0.87 (2)	2.449 (17)	3.236 (3)	151 (3)
N1—H1B⋯O5^{viii}	0.87 (3)	2.58 (3)	3.403 (3)	158 (2)

Symmetry codes: (vi) x+1, y, z; (vii) −x+1, −y+1, −z+1; (iii) x, y, z+1; (viii) x−1, y, z.

Fig. 1

Fig. 2

Fig. 3