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Dimethyl(4-methylphenyl)ammonium naphthalene-1,5-disulfonate dihydrate

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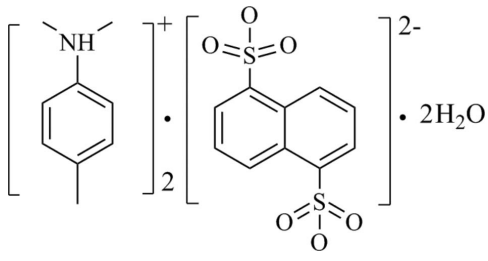
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; some non-H atoms missing; R factor = 0.040; wR factor = 0.115; data-to-parameter ratio = 17.4.

The asymmetric unit of the organic–inorganic hybrid salt, $2C_9H_{14}N^+\cdot C_{10}H_6O_6S_2^{2-}\cdot 2H_2O$, consists of one dimethyl(4-methylphenyl)ammonium cation, one half of a naphthalene-1,5-disulfonate anion lying on a crystallographic centre of inversion, and one water molecule. In the crystal, O—H···O(S) and N—H···OH_2 hydrogen bonds link the cations and anions forming ring motifs.

Related literature

The title compound was obtained during attempts to obtain dielectric-ferroelectric materials. For general background to ferroelectric metal-organic frameworks, see: Wu *et al.* (2011); Fu *et al.* (2009); Ye *et al.* (2006); Zhang *et al.* (2008); Zhang *et al.* (2010).



Experimental

Crystal data

$2C_9H_{14}N^+\cdot C_{10}H_6O_6S_2^{2-}\cdot 2H_2O$
M_r = 594.74
Triclinic, $P\overline{1}$
a = 9.2660 (19) Å
b = 9.882 (2) Å
c = 10.260 (2) Å
α = 109.59 (3)°
β = 115.79 (3)°
γ = 98.39 (3)°
V = 748.3 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.955, T_{max} = 0.955
7762 measured reflections
3421 independent reflections
2951 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.115
S = 1.06
3421 reflections
197 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4B···O3^{i}	0.85 (3)	1.93 (3)	2.778 (3)	176 (3)
O4—H4B···S1^{i}	0.85 (3)	2.96 (3)	3.753 (3)	157 (2)
N1—H10···O4^{ii}	0.89 (2)	1.84 (2)	2.723 (2)	174.2 (19)
O4—H4A···O1^{iii}	0.84 (3)	2.01 (3)	2.846 (2)	171 (2)
O4—H4A···S1^{iii}	0.84 (3)	2.87 (3)	3.6569 (18)	156 (2)

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

The author is grateful to the starter fund of Southeast University for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2329).

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supporting information

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Dimethyl(4-methylphenyl)ammonium naphthalene-1,5-disulfonate dihydrate

Bin Wei

S1. Comment

Dielectric-ferroelectric constitute an interesting class of materials, comprising organic ligands, metal-organic coordination compounds and organic-inorganic hybrids(Fu *et al.*, 2009; Zhang *et al.*, 2010; Zhang *et al.*, 2008; Ye *et al.*, 2006). Unfortunately,the dielectric constant of the title compound as a function of temperature indicates that the permittivity is basically temperature-independent, below the melting point (385k-387k) of the compound, we have found that cyclohexylammonium 4-methoxy-benzoate has no dielectric disuniform from 80 K to 405 K. Herein we descibe the crystal structure of this compound.

Regarding its crystal structure,the asymmetric unit of the title compound consists of a dimethyl(4-methylphenyl)-ammonium cation, a half of naphthalene-1,5-disulfonate anion and a water molecule(Fig. 1). The free water molecules connected cations and anions by intermolecular hydrogen bonds involving O—H···S, O—H···O and N—H···O which makes great contribution to the stability of the crystal structure,and these hydrogen bonds link the cations, water molecules and anions into a chains along the *c* axis(Fig. 2 and Tab. 1).

S2. Experimental

The title compound was obtained by the addition of naphthalene-1,5-disulfonate acid (3.62 g, 0.01 mol) to a solution of dimethyl(4-methylphenyl)amine (2.72 g, 0.02 mol) in water, in the stoichiometric ratio 1: 2. Good quality single crystals were obtained by slow evaporation after two days(the chemical yield is 35%).

S3. Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H = 0.96 Å, O—H = 0.84 to 0.85 Å, N—H = 0.89 Å and with U_{iso}(H) = 1.2 U_{iso}(C, O) or 1.5 U_{iso}(C) for methyl H atoms.

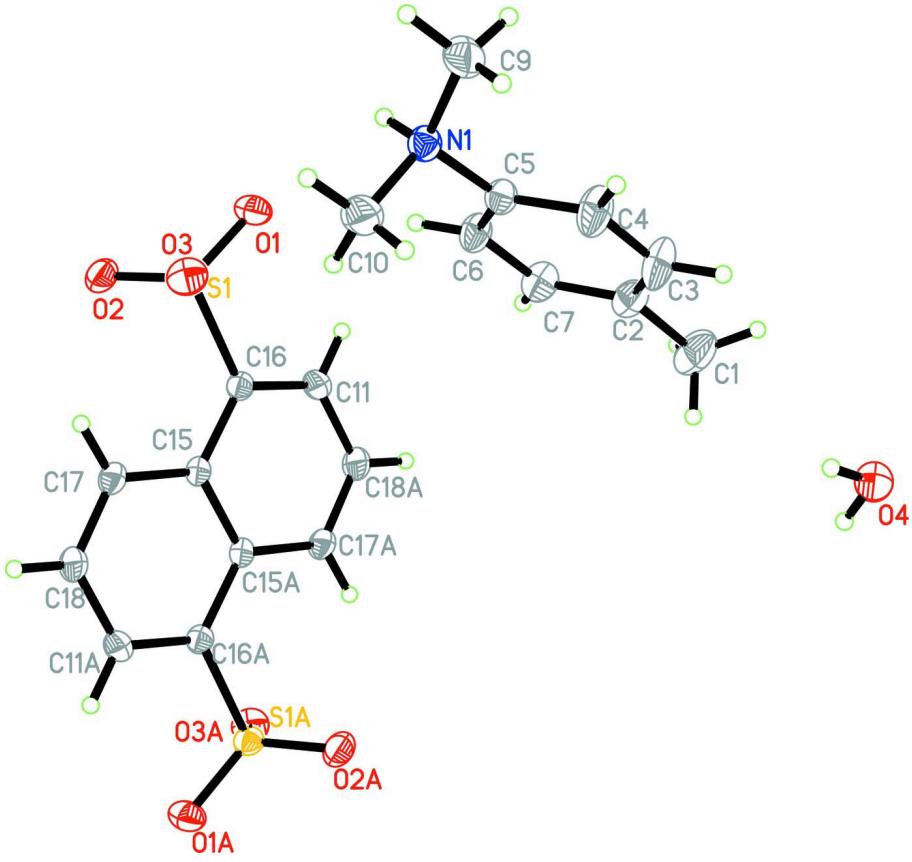

Figure 1

The molecular structure of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

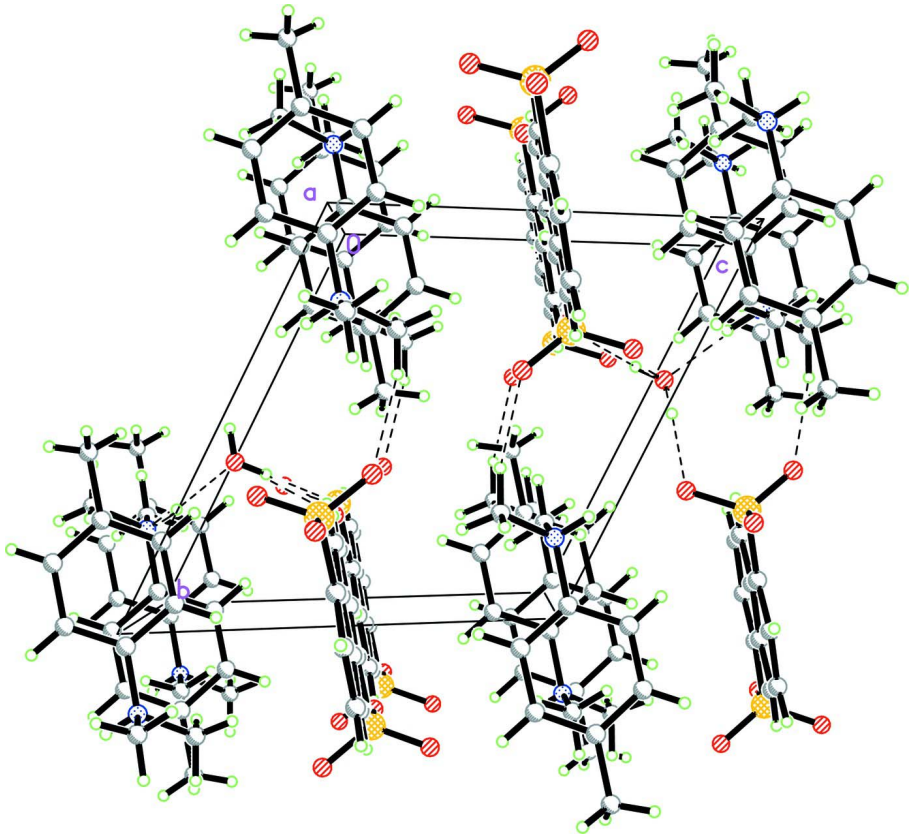

Figure 2

A view of the packing of the title compound, stacking along the *a* axis. Dashed lines indicate hydrogen bonds.

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Crystal data

$2C_9H_{14}N^+·C_{10}H_6O_6S_2^{2-}·2H_2O$
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c = 10.260 (2) Å
α = 109.59 (3)°
β = 115.79 (3)°
γ = 98.39 (3)°
V = 748.3 (3) Å^3
Z = 1
$F(000)$ = 314
D_x = 1.315 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
θ = 3.0–27.5°
μ = 0.23 mm^{-1}
T = 293 K
Block, colorless
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.955, T_{max} = 0.955
7762 measured reflections
3421 independent reflections
2951 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°, θ_{min} = 3.3°
h = −12→12
k = −12→12
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.115$
$S = 1.06$
3421 reflections
197 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.1813P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.015$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.030 (5)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5316 (3)	0.4244 (2)	0.3073 (3)	0.0732 (7)
H1A	0.5825	0.4690	0.2607	0.110*
H1B	0.6194	0.4366	0.4096	0.110*
H1C	0.4552	0.4746	0.3232	0.110*
N1	0.14101 (18)	−0.21478 (16)	−0.12073 (17)	0.0374 (3)
O1	0.29693 (16)	−0.24956 (14)	0.32982 (15)	0.0444 (3)
S1	0.13241 (5)	−0.27559 (4)	0.31891 (4)	0.03323 (14)
C2	0.4338 (2)	0.2567 (2)	0.1950 (2)	0.0487 (4)
O2	0.10384 (18)	−0.37468 (13)	0.38791 (16)	0.0482 (3)
C3	0.3514 (3)	0.2034 (2)	0.0300 (3)	0.0649 (6)
H3	0.3587	0.2722	−0.0123	0.078*
O3	−0.00765 (16)	−0.32258 (14)	0.15568 (14)	0.0466 (3)
C4	0.2579 (3)	0.0502 (2)	−0.0752 (2)	0.0590 (6)
H4	0.2035	0.0167	−0.1865	0.071*
O4	0.26401 (18)	0.60704 (17)	0.02223 (18)	0.0468 (3)
C5	0.2464 (2)	−0.05151 (19)	−0.0131 (2)	0.0369 (4)
C6	0.3283 (2)	−0.0021 (2)	0.1516 (2)	0.0442 (4)
H6	0.3209	−0.0714	0.1933	0.053*
C7	0.4220 (3)	0.1516 (2)	0.2548 (2)	0.0495 (5)
H7	0.4779	0.1848	0.3661	0.059*
C9	0.1372 (3)	−0.2773 (2)	−0.2766 (2)	0.0580 (5)
H9A	0.0704	−0.2369	−0.3454	0.087*
H9B	0.0872	−0.3868	−0.3291	0.087*

H9C	0.2515	−0.2485	−0.2556	0.087*
C10	−0.0362 (3)	−0.2446 (2)	−0.1518 (3)	0.0573 (5)
H10A	−0.0325	−0.2135	−0.0508	0.086*
H10B	−0.1006	−0.3519	−0.2186	0.086*
H10C	−0.0897	−0.1877	−0.2060	0.086*
C11	0.2843 (2)	0.02932 (18)	0.5176 (2)	0.0371 (4)
H11	0.3775	0.0157	0.5088	0.045*
C15	−0.00608 (18)	−0.07490 (15)	0.45203 (17)	0.0275 (3)
C16	0.13837 (19)	−0.09301 (16)	0.43905 (17)	0.0294 (3)
C17	−0.1605 (2)	−0.19878 (17)	0.37400 (19)	0.0350 (3)
H17	−0.1695	−0.2968	0.3124	0.042*
C18	−0.2949 (2)	−0.17579 (18)	0.3883 (2)	0.0412 (4)
H18	−0.3954	−0.2581	0.3351	0.049*
H10	0.186 (3)	−0.267 (2)	−0.068 (2)	0.043 (5)*
H4A	0.268 (3)	0.640 (3)	0.111 (4)	0.073 (8)*
H4B	0.187 (3)	0.519 (3)	−0.036 (3)	0.075 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0718 (15)	0.0389 (11)	0.0853 (17)	0.0116 (10)	0.0304 (13)	0.0197 (11)
N1	0.0403 (8)	0.0372 (7)	0.0359 (7)	0.0127 (6)	0.0197 (6)	0.0179 (6)
O1	0.0425 (7)	0.0480 (7)	0.0438 (7)	0.0180 (5)	0.0269 (6)	0.0144 (6)
S1	0.0390 (2)	0.0300 (2)	0.0306 (2)	0.01212 (16)	0.01956 (17)	0.01138 (16)
C2	0.0452 (10)	0.0376 (9)	0.0546 (11)	0.0129 (8)	0.0207 (9)	0.0190 (8)
O2	0.0708 (9)	0.0355 (6)	0.0547 (8)	0.0226 (6)	0.0408 (7)	0.0247 (6)
C3	0.0811 (16)	0.0455 (11)	0.0614 (13)	0.0120 (10)	0.0263 (12)	0.0354 (10)
O3	0.0474 (7)	0.0445 (7)	0.0310 (6)	0.0119 (5)	0.0147 (5)	0.0084 (5)
C4	0.0767 (14)	0.0477 (11)	0.0410 (10)	0.0108 (10)	0.0198 (10)	0.0266 (9)
O4	0.0493 (8)	0.0446 (8)	0.0410 (7)	0.0129 (6)	0.0218 (6)	0.0170 (6)
C5	0.0387 (8)	0.0366 (8)	0.0355 (8)	0.0126 (7)	0.0177 (7)	0.0186 (7)
C6	0.0547 (11)	0.0397 (9)	0.0390 (9)	0.0147 (8)	0.0212 (8)	0.0231 (8)
C7	0.0532 (11)	0.0429 (10)	0.0383 (9)	0.0118 (8)	0.0155 (8)	0.0163 (8)
C9	0.0728 (14)	0.0515 (11)	0.0478 (11)	0.0116 (10)	0.0390 (11)	0.0138 (9)
C10	0.0454 (11)	0.0502 (11)	0.0707 (14)	0.0124 (9)	0.0332 (10)	0.0187 (10)
C11	0.0299 (8)	0.0354 (8)	0.0457 (9)	0.0086 (6)	0.0218 (7)	0.0159 (7)
C15	0.0293 (7)	0.0249 (7)	0.0263 (7)	0.0061 (5)	0.0135 (6)	0.0120 (6)
C16	0.0325 (7)	0.0273 (7)	0.0286 (7)	0.0095 (6)	0.0161 (6)	0.0127 (6)
C17	0.0341 (8)	0.0242 (7)	0.0382 (8)	0.0047 (6)	0.0168 (7)	0.0099 (6)
C18	0.0314 (8)	0.0292 (8)	0.0512 (10)	0.0013 (6)	0.0193 (7)	0.0121 (7)

Geometric parameters (Å, º)

C1—C2	1.506 (3)	C5—C6	1.376 (2)
C1—H1A	0.9600	C6—C7	1.385 (3)
C1—H1B	0.9600	C6—H6	0.9300
C1—H1C	0.9600	C7—H7	0.9300
N1—C5	1.478 (2)	C9—H9A	0.9600

N1—C9	1.490 (2)	C9—H9B	0.9600
N1—C10	1.492 (2)	C9—H9C	0.9600
N1—H10	0.89 (2)	C10—H10A	0.9600
O1—S1	1.4562 (13)	C10—H10B	0.9600
S1—O2	1.4430 (13)	C10—H10C	0.9600
S1—O3	1.4548 (15)	C11—C16	1.364 (2)
S1—C16	1.7883 (16)	C11—C18^{i}	1.406 (2)
C2—C3	1.374 (3)	C11—H11	0.9300
C2—C7	1.386 (3)	C15—C17	1.422 (2)
C3—C4	1.384 (3)	C15—C16	1.432 (2)
C3—H3	0.9299	C15—C15^{i}	1.432 (3)
C4—C5	1.373 (2)	C17—C18	1.358 (2)
C4—H4	0.9300	C17—H17	0.9300
O4—H4A	0.84 (3)	C18—C11^{i}	1.406 (2)
O4—H4B	0.85 (3)	C18—H18	0.9300

C2—C1—H1A	109.5	C5—C6—H6	120.3
C2—C1—H1B	109.5	C7—C6—H6	120.3
H1A—C1—H1B	109.5	C6—C7—C2	121.13 (17)
C2—C1—H1C	109.5	C6—C7—H7	119.4
H1A—C1—H1C	109.5	C2—C7—H7	119.4
H1B—C1—H1C	109.5	N1—C9—H9A	109.5
C5—N1—C9	114.42 (14)	N1—C9—H9B	109.5
C5—N1—C10	111.22 (14)	H9A—C9—H9B	109.5
C9—N1—C10	110.28 (16)	N1—C9—H9C	109.5
C5—N1—H10	107.1 (13)	H9A—C9—H9C	109.5
C9—N1—H10	107.0 (13)	H9B—C9—H9C	109.5
C10—N1—H10	106.4 (13)	N1—C10—H10A	109.5
O2—S1—O3	113.10 (9)	N1—C10—H10B	109.5
O2—S1—O1	113.24 (8)	H10A—C10—H10B	109.5
O3—S1—O1	112.12 (8)	N1—C10—H10C	109.5
O2—S1—C16	106.42 (7)	H10A—C10—H10C	109.5
O3—S1—C16	105.32 (8)	H10B—C10—H10C	109.5
O1—S1—C16	105.85 (8)	C16—C11—C18^{i}	120.47 (15)
C3—C2—C7	117.88 (17)	C16—C11—H11	119.8
C3—C2—C1	121.06 (19)	C18^{i}—C11—H11	119.8
C7—C2—C1	121.1 (2)	C17—C15—C16	122.94 (13)
C2—C3—C4	121.90 (18)	C17—C15—C15^{i}	118.74 (17)
C2—C3—H3	119.0	C16—C15—C15^{i}	118.32 (16)
C4—C3—H3	119.1	C11—C16—C15	120.69 (14)
C5—C4—C3	119.14 (18)	C11—C16—S1	118.17 (12)
C5—C4—H4	120.4	C15—C16—S1	121.14 (11)
C3—C4—H4	120.4	C18—C17—C15	120.86 (15)
H4A—O4—H4B	105 (2)	C18—C17—H17	119.6
C4—C5—C6	120.44 (17)	C15—C17—H17	119.6
C4—C5—N1	121.04 (15)	C17—C18—C11^{i}	120.91 (15)

C6—C5—N1	118.48 (15)	C17—C18—H18	119.5
C5—C6—C7	119.50 (16)	C11^{i}—C18—H18	119.5

Symmetry code: (i) −*x*, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*B*···O3^{ii}	0.85 (3)	1.93 (3)	2.778 (3)	176 (3)
O4—H4*B*···S1^{ii}	0.85 (3)	2.96 (3)	3.753 (3)	157 (2)
N1—H10···O4^{iii}	0.89 (2)	1.84 (2)	2.723 (2)	174.2 (19)
O4—H4*A*···O1^{iv}	0.84 (3)	2.01 (3)	2.846 (2)	171 (2)
O4—H4*A*···S1^{iv}	0.84 (3)	2.87 (3)	3.6569 (18)	156 (2)

Symmetry codes: (ii) −*x*, −*y*, −*z*; (iii) *x*, *y*−1, *z*; (iv) *x*, *y*+1, *z*.