Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(pyridinium) naphthalene-1,5-disulfonate dihydrate

Bin Wei

Ordered Matter Science Research Center, Southeast University, Nanjing 211189, People's Republic of China
Correspondence e-mail: seuwei@126.com

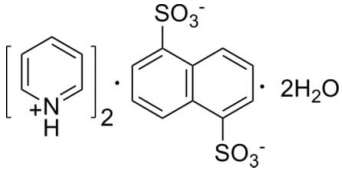
Received 21 March 2012; accepted 18 April 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.056; wR factor = 0.150; data-to-parameter ratio = 17.1.

The asymmetric unit of the title organic salt, $2C_5H_6N^+\cdot C_{10}H_6O_6S_2^{2-}\cdot 2H_2O$, consists of a pyridinium cation, half a naphthalene-1,5-disulfonate dianion and a water molecule. The dianion has a crystallographically imposed centre of symmetry. In the crystal, N—H···O and O—H···O hydrogen bonds link cations, anions and water molecules into a three-dimensional network.

Related literature

For general background to ferroelectric metal-organic frameworks, see: Ye *et al.* (2006); Zhang *et al.* (2008, 2010); Fu *et al.* (2009).



Experimental

Crystal data

$2C_5H_6N^+\cdot C_{10}H_6O_6S_2^{2-}\cdot 2H_2O$
M_r = 482.52
Monoclinic, $P2_1/n$
a = 9.5876 (19) Å
b = 12.065 (2) Å
c = 9.843 (2) Å
β = 103.51 (3)°
V = 1107.0 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 293 K
0.55 × 0.44 × 0.36 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.860, T_{max} = 0.902
11131 measured reflections
2533 independent reflections
2150 reflections with $I > 2\sigma(I)$
R_{int} = 0.072

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.150
S = 1.10
2533 reflections
148 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4B···O1	0.82	2.07	2.801 (3)	148
O4—H4A···O2^{i}	0.82	1.91	2.731 (3)	174
N1—H10···O4^{ii}	0.86	1.82	2.667 (3)	170

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

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

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: RZ2728).

References

Fu, D.-W., Ge, J.-Z., Dai, J., Ye, H.-Y. & Qu, Z.-R. (2009). *Inorg. Chem. Commun.* **12**, 994–997.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Ye, Q., Song, Y.-M., Wang, G.-X., Chen, K. & Fu, D.-W. (2006). *J. Am. Chem. Soc.* **128**, 6554–6555.
Zhang, W., Xiong, R.-G. & Huang, S.-P. D. (2008). *J. Am. Chem. Soc.* **130**, 10468–10469.
Zhang, W., Ye, H.-Y., Cai, H.-L., Ge, J.-Z. & Xiong, R.-G. (2010). *J. Am. Chem. Soc.* **132**, 7300–7302.

supplementary materials

Acta Cryst. (2012). E**68**, o1489 [doi:10.1107/S1600536812016960]

Bis(pyridinium) naphthalene-1,5-disulfonate dihydrate

Bin Wei

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 (411k-412k) of the compound, we have found that title compound has no dielectric disuniform from 80 K to 405 K. Herein we descibe the crystal structure of this compound.

The asymmetric unit of the title compound (Fig. 1) consists of a pyridinium cation, a half of a naphthalene-1,5-disulfonate anion and a free water molecule, the anion having crystallographically imposed centre of symmetry. The pyridinium and naphthalene rings are oriented to form a dihedral angle of 13.89 (6)°. In the crystal, cations, anions and water molecules are connected by N—H···O and O—H···O intermolecular hydrogen bonds into a three-dimensional structure (Fig. 2; Table 1)

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 pyridine (1.6 g, 0.02 mol) in methanol, in the stoichiometric ratio 1: 2. Good quality single crystals were obtained by slow evaporation of the solvent after six days (yield 52%).

Refinement

The water H atoms were located in a difference Fourier map and refined as riding, with the O—H distances restrained to 0.82 Å and with U_{iso}(H) = 1.5U_{eq}(O). All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å, N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq}(C, N).

Computing details

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear* (Rigaku, 2005); data reduction: *CrystalClear* (Rigaku, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

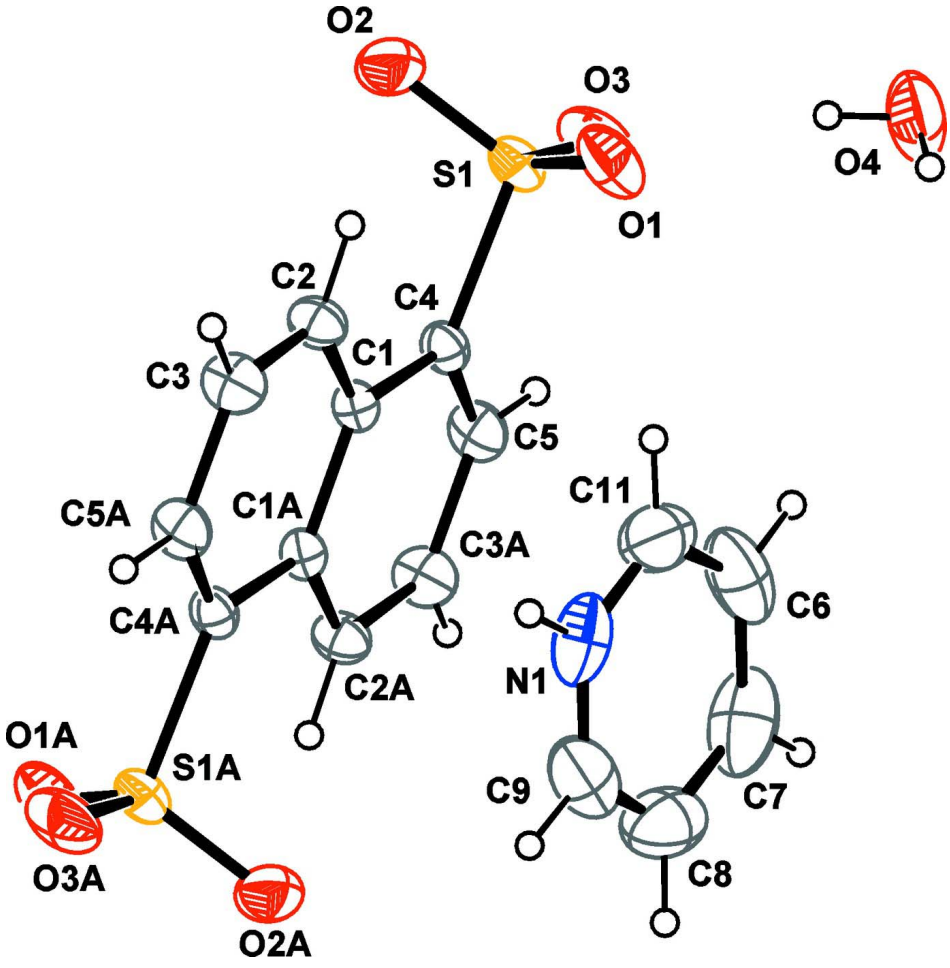


Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. Atoms labelled with suffix A are generated by the symmetry operator (2-x, -y, 1-z).

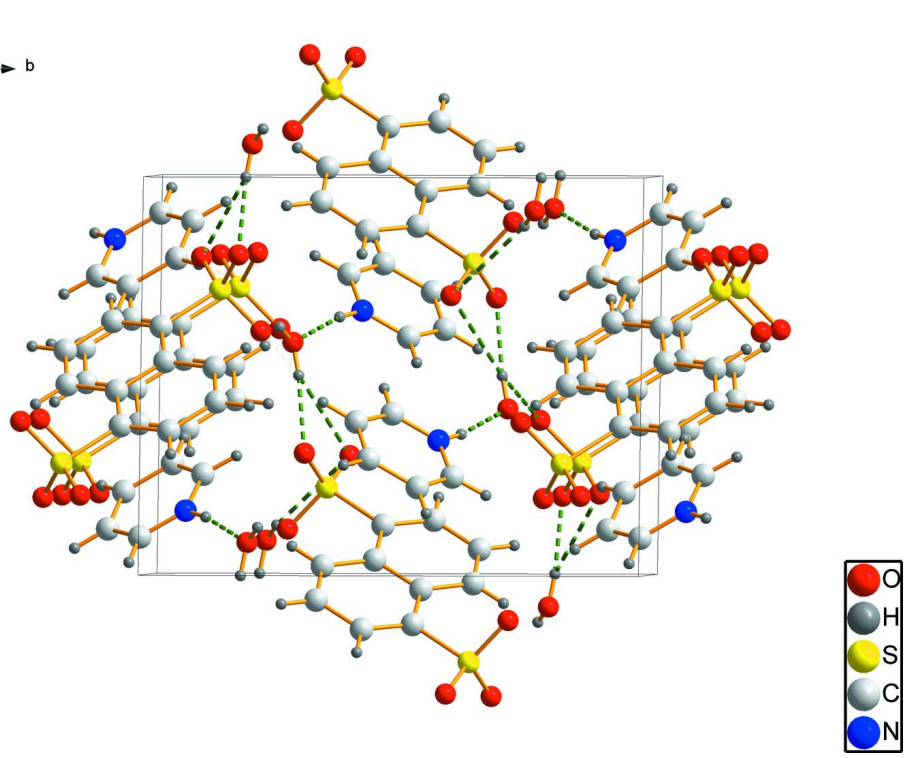


Figure 2

Crystal packing of the title compound viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

Bis(pyridinium) naphthalene-1,5-disulfonate dihydrate

Crystal data

$2C_5H_6N^+\cdot C_{10}H_6O_6S_2^{2-}\cdot 2H_2O$
M_r = 482.52
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 9.5876 (19) Å
b = 12.065 (2) Å
c = 9.843 (2) Å
β = 103.51 (3)°
V = 1107.0 (4) Å^3
Z = 2
$F(000)$ = 504
D_x = 1.448 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3638 reflections
θ = 3.0–27.5°
μ = 0.29 mm^{-1}
T = 293 K
Block, colourless
0.55 × 0.44 × 0.36 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.860, T_{max} = 0.902
11131 measured reflections
2533 independent reflections
2150 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 27.5°, θ_{min} = 3.2°
$h = -12 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = -12 \rightarrow 12$
2 standard reflections every 150 reflections

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.150$
$S = 1.10$
2533 reflections
148 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.5924P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

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}
O4	0.4852 (3)	0.2836 (3)	0.5829 (3)	0.1102 (12)
H4A	0.4806	0.2959	0.4999	0.19 (3)*
H4B	0.5648	0.2599	0.6216	0.15 (2)*
S1	0.83962 (6)	0.15020 (5)	0.71768 (6)	0.0461 (2)
C1	0.9846 (2)	0.04729 (16)	0.53702 (19)	0.0316 (4)
C2	1.0533 (2)	0.14879 (17)	0.5220 (2)	0.0411 (5)
H2	1.0350	0.2108	0.5712	0.049*
C4	0.8853 (2)	0.03502 (17)	0.6239 (2)	0.0348 (4)
C3	1.1457 (3)	0.15711 (19)	0.4368 (3)	0.0483 (6)
H3	1.1884	0.2249	0.4269	0.058*
O3	0.7426 (2)	0.10851 (18)	0.7975 (2)	0.0700 (6)
C5	0.8229 (3)	−0.0643 (2)	0.6363 (2)	0.0450 (5)
H5	0.7588	−0.0708	0.6937	0.054*
O2	0.9728 (2)	0.19099 (18)	0.8046 (2)	0.0659 (6)
O1	0.7752 (2)	0.23031 (17)	0.6128 (2)	0.0712 (6)
N1	0.7941 (3)	0.0855 (3)	0.1614 (3)	0.0736 (8)
H10	0.8605	0.1283	0.1461	0.088*
C9	0.7696 (4)	−0.0066 (4)	0.0948 (4)	0.0811 (11)
H9	0.8240	−0.0255	0.0314	0.097*
C6	0.6178 (5)	0.0513 (4)	0.2786 (4)	0.0933 (13)
H6	0.5660	0.0718	0.3435	0.112*
C8	0.6691 (6)	−0.0745 (3)	0.1146 (4)	0.0943 (12)
H8	0.6521	−0.1410	0.0656	0.113*
C11	0.7207 (4)	0.1164 (3)	0.2526 (4)	0.0804 (10)
H11	0.7408	0.1838	0.2989	0.097*
C7	0.5903 (4)	−0.0464 (4)	0.2074 (6)	0.1002 (15)

H7	0.5183	−0.0932	0.2223	0.120*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.0640 (15)	0.206 (4)	0.0579 (14)	0.0563 (18)	0.0094 (11)	−0.0121 (17)
S1	0.0446 (3)	0.0501 (4)	0.0488 (4)	0.0077 (2)	0.0215 (3)	−0.0075 (2)
C1	0.0306 (9)	0.0345 (10)	0.0306 (9)	0.0028 (7)	0.0090 (7)	0.0042 (8)
C2	0.0482 (12)	0.0334 (10)	0.0450 (12)	−0.0010 (9)	0.0177 (10)	−0.0007 (8)
C4	0.0337 (10)	0.0403 (11)	0.0329 (10)	0.0044 (8)	0.0125 (8)	0.0016 (8)
C3	0.0544 (14)	0.0389 (11)	0.0579 (14)	−0.0099 (10)	0.0263 (12)	0.0035 (10)
O3	0.0734 (12)	0.0738 (13)	0.0809 (14)	−0.0020 (11)	0.0546 (11)	−0.0165 (11)
C5	0.0456 (12)	0.0509 (13)	0.0453 (12)	−0.0030 (10)	0.0244 (10)	0.0037 (10)
O2	0.0611 (11)	0.0809 (13)	0.0583 (11)	−0.0078 (10)	0.0192 (9)	−0.0258 (10)
O1	0.0746 (14)	0.0612 (12)	0.0818 (14)	0.0320 (10)	0.0263 (11)	0.0060 (10)
N1	0.0460 (13)	0.094 (2)	0.0767 (18)	−0.0075 (13)	0.0056 (12)	0.0324 (16)
C9	0.087 (2)	0.102 (3)	0.0581 (18)	0.036 (2)	0.0238 (17)	0.0150 (18)
C6	0.080 (2)	0.125 (4)	0.087 (2)	0.039 (2)	0.046 (2)	0.026 (2)
C8	0.116 (3)	0.061 (2)	0.091 (3)	0.006 (2)	−0.005 (3)	−0.0072 (19)
C11	0.090 (3)	0.0665 (19)	0.074 (2)	0.0054 (18)	−0.0035 (19)	−0.0072 (17)
C7	0.063 (2)	0.091 (3)	0.141 (4)	−0.017 (2)	0.012 (2)	0.050 (3)

Geometric parameters (Å, º)

O4—H4A	0.8207	C5—C3^{i}	1.401 (3)
O4—H4B	0.8202	C5—H5	0.9300
S1—O3	1.4413 (19)	N1—C9	1.283 (5)
S1—O1	1.443 (2)	N1—C11	1.317 (5)
S1—O2	1.447 (2)	N1—H10	0.8600
S1—C4	1.779 (2)	C9—C8	1.314 (6)
C1—C2	1.415 (3)	C9—H9	0.9300
C1—C1^{i}	1.422 (4)	C6—C11	1.332 (6)
C1—C4	1.428 (3)	C6—C7	1.365 (6)
C2—C3	1.358 (3)	C6—H6	0.9300
C2—H2	0.9300	C8—C7	1.357 (6)
C4—C5	1.358 (3)	C8—H8	0.9300
C3—C5^{i}	1.401 (3)	C11—H11	0.9300
C3—H3	0.9300	C7—H7	0.9300
H4A—O4—H4B	110.7	C4—C5—H5	119.8
O3—S1—O1	113.62 (13)	C3^{i}—C5—H5	119.8
O3—S1—O2	112.87 (13)	C9—N1—C11	122.1 (3)
O1—S1—O2	111.49 (14)	C9—N1—H10	119.0
O3—S1—C4	106.19 (11)	C11—N1—H10	119.0
O1—S1—C4	105.55 (11)	N1—C9—C8	121.1 (4)
O2—S1—C4	106.42 (11)	N1—C9—H9	119.4
C2—C1—C1^{i}	118.9 (2)	C8—C9—H9	119.4
C2—C1—C4	122.98 (18)	C11—C6—C7	118.3 (4)
C1^{i}—C1—C4	118.1 (2)	C11—C6—H6	120.9
C3—C2—C1	121.0 (2)	C7—C6—H6	120.9

C3—C2—H2	119.5	C9—C8—C7	119.1 (4)
C1—C2—H2	119.5	C9—C8—H8	120.4
C5—C4—C1	120.97 (19)	C7—C8—H8	120.4
C5—C4—S1	118.43 (16)	N1—C11—C6	120.1 (4)
C1—C4—S1	120.60 (16)	N1—C11—H11	120.0
C2—C3—C5^{i}	120.5 (2)	C6—C11—H11	120.0
C2—C3—H3	119.8	C8—C7—C6	119.4 (4)
C5^{i}—C3—H3	119.8	C8—C7—H7	120.3
C4—C5—C3^{i}	120.48 (19)	C6—C7—H7	120.3

Symmetry code: (i) −*x*+2, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*B*···O1	0.82	2.07	2.801 (3)	148
O4—H4*A*···O2^{ii}	0.82	1.91	2.731 (3)	174
N1—H10···O4^{iii}	0.86	1.82	2.667 (3)	170

Symmetry codes: (ii) *x*−1/2, −*y*+1/2, *z*−1/2; (iii) *x*+1/2, −*y*+1/2, *z*−1/2.