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Bis(2-methylpiperidinium) naphthalene-1,5-disulfonate

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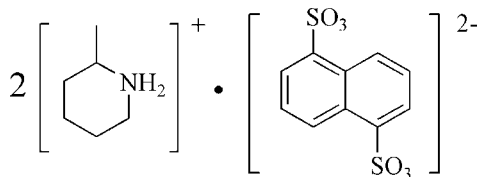
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.058; wR factor = 0.134; data-to-parameter ratio = 19.3.

In the structure of the title molecular salt, $2\text{C}_6\text{H}_{14}\text{N}^+\cdot\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2^{2-}$, the asymmetric unit consists of one 2-methylpiperidinium cation and one-half of a naphthalene-1,5-disulfonate anion; the anion lies across a centre of symmetry. In the crystal, the cations and anions are linked through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a two-dimensional network.

Related literature

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



Experimental

Crystal data

$2\text{C}_6\text{H}_{14}\text{N}^+\cdot\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2^{2-}$
 $M_r = 486.63$
 Monoclinic, $P2_1/c$
 $a = 12.040$ (2) Å
 $b = 8.8133$ (18) Å

$c = 12.715$ (3) Å
 $\beta = 112.62$ (3)°
 $V = 1245.4$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.25$ mm⁻¹
 $T = 293$ K

$0.32 \times 0.27 \times 0.22$ mm

Data collection

Rigaku SCXmini diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\text{min}} = 0.924$, $T_{\text{max}} = 0.947$
 12383 measured reflections

2814 independent reflections
 1942 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.069$
 Standard reflections: ?

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.134$
 $S = 1.04$
 2814 reflections

146 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1B}\cdots\text{O2}$	0.90	1.91	2.795 (3)	169
$\text{N1}-\text{H1A}\cdots\text{O1}^1$	0.90	1.93	2.820 (3)	169

Symmetry code: (i) $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$.

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: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

This work was supported by Southeast University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GO2054).

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supplementary materials

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Bis(2-methylpiperidinium) naphthalene-1,5-disulfonate

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Comment

The title compound, (I), was synthesized to assess its ferroelectric properties by dielectric measurements as a function of temperature (Fu *et al.*, 2009; Ye *et al.*, 2006; Zhang *et al.*, 2008; Zhang *et al.*, 2010). In the range from 190K to near its melting point (m.p. >370K), no dielectric anomaly was observed.

A view of (I) is shown in Fig.1. Two intermolecular N–H···O hydrogen bonds form a two-dimensional network, Table, 1, Fig. 2.

Experimental

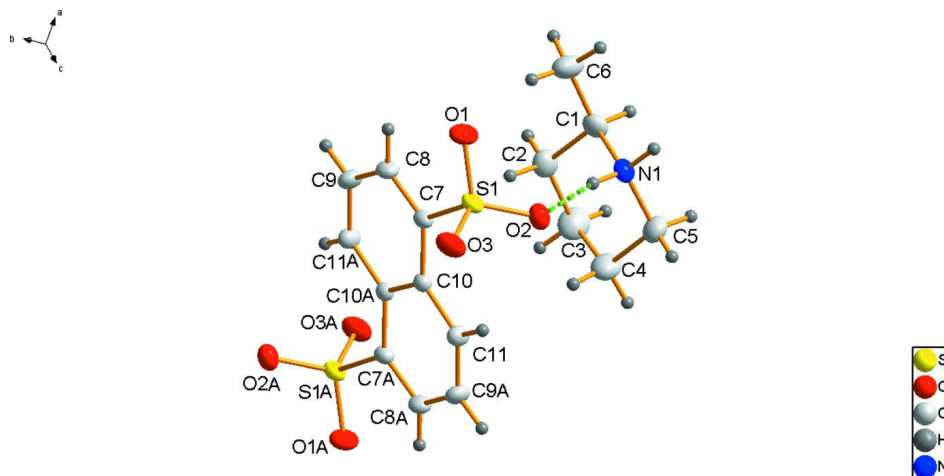
A mixture of 2-methylpiperidine (0.98 g, 10 mmol), naphthalene-1,5-disulfonic acid (2.5 g, 10 mmol) in water was stirred for several days at ambient temperature, colourless crystals were obtained.

Refinement

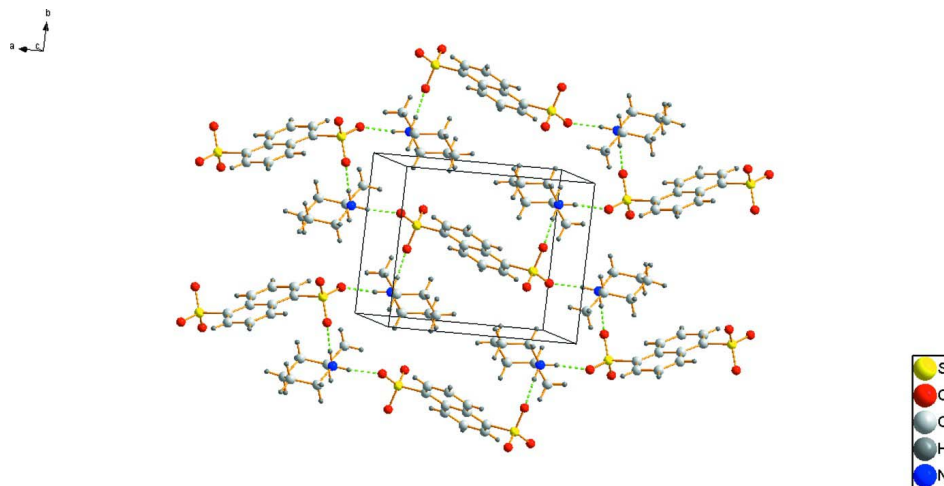
Hydrogen atom positions were calculated and allowed to ride on their respective C atoms and N atoms with C–H distances of 0.93–0.98 Å and N–H = 0.90 Å, and with $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C or N})$, $1.5 U_{\text{iso}}(\text{C})$ for methyl H atoms.

Computing details

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear* (Rigaku, 2005); data reduction: *CrystalClear* (Rigaku, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).


Figure 1

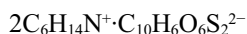
The molecular structure of the title compound, with the displacement ellipsoids drawn at the 30% probability level. Symmetry code for A: $1 - x, 1 - y, 1 - z$.


Figure 2

Packing diagram of the title compound, hydrogen bonds are shown as dashed lines.

Bis(2-methylpiperidinium) naphthalene-1,5-disulfonate

Crystal data



$M_r = 486.63$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 12.040\ (2)\ \text{\AA}$

$b = 8.8133\ (18)\ \text{\AA}$

$c = 12.715\ (3)\ \text{\AA}$

$\beta = 112.62\ (3)^\circ$

$V = 1245.4\ (4)\ \text{\AA}^3$

$Z = 2$

$F(000) = 520$

$D_x = 1.298\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2814 reflections

$\theta = 3.1\text{--}27.5^\circ$

$\mu = 0.25\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, colourless

$0.32 \times 0.27 \times 0.22\ \text{mm}$

Data collection

Rigaku SCXmini diffractometer	12383 measured reflections
Radiation source: fine-focus sealed tube	2814 independent reflections
Graphite monochromator	1942 reflections with $I > 2\sigma(I)$
CCD_Profile_fitting scans	$R_{\text{int}} = 0.069$
Absorption correction: multi-scan (<i>CrystalClear</i> ; Rigaku, 2005)	$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.0^\circ$
$T_{\text{min}} = 0.924$, $T_{\text{max}} = 0.947$	$h = -15 \rightarrow 15$
	$k = -11 \rightarrow 11$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.058$	H-atom parameters constrained
$wR(F^2) = 0.134$	$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.481P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
2814 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
146 parameters	$\Delta\rho_{\text{max}} = 0.22 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.28 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.78457 (5)	0.59654 (7)	0.69286 (5)	0.0339 (2)
O1	0.88140 (15)	0.6629 (2)	0.66486 (16)	0.0499 (5)
O2	0.81475 (16)	0.4419 (2)	0.73778 (15)	0.0452 (5)
O3	0.74714 (16)	0.6921 (2)	0.76559 (16)	0.0508 (5)
C7	0.6587 (2)	0.5777 (3)	0.5596 (2)	0.0306 (5)
C8	0.6710 (2)	0.6257 (3)	0.4618 (2)	0.0376 (6)
H8	0.7426	0.6706	0.4663	0.045*
C9	0.5757 (2)	0.6074 (3)	0.3545 (2)	0.0439 (7)
H9	0.5848	0.6414	0.2890	0.053*
C10	0.5479 (2)	0.5101 (3)	0.55522 (19)	0.0286 (5)
C11	0.5297 (2)	0.4597 (3)	0.6538 (2)	0.0388 (6)
H11	0.5907	0.4727	0.7253	0.047*
N1	0.88441 (18)	0.1722 (2)	0.66473 (18)	0.0395 (5)
H1A	0.9617	0.1614	0.7127	0.047*
H1B	0.8586	0.2628	0.6790	0.047*
C1	0.8782 (2)	0.1714 (3)	0.5439 (2)	0.0475 (7)
H1	0.9076	0.0732	0.5292	0.057*

C2	0.7463 (3)	0.1887 (3)	0.4635 (2)	0.0509 (8)
H2A	0.7176	0.2877	0.4750	0.061*
H2B	0.7404	0.1834	0.3854	0.061*
C3	0.6666 (3)	0.0666 (4)	0.4827 (3)	0.0713 (10)
H3A	0.6888	-0.0317	0.4624	0.086*
H3B	0.5833	0.0860	0.4341	0.086*
C4	0.6799 (3)	0.0642 (4)	0.6077 (3)	0.0668 (10)
H4A	0.6468	0.1569	0.6248	0.080*
H4B	0.6345	-0.0204	0.6196	0.080*
C5	0.8109 (3)	0.0494 (3)	0.6881 (3)	0.0552 (8)
H5A	0.8172	0.0568	0.7663	0.066*
H5B	0.8415	-0.0490	0.6780	0.066*
C6	0.9591 (3)	0.2953 (4)	0.5302 (3)	0.0726 (10)
H6A	0.9276	0.3927	0.5383	0.109*
H6B	0.9623	0.2879	0.4561	0.109*
H6C	1.0387	0.2838	0.5875	0.109*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0244 (3)	0.0376 (4)	0.0378 (4)	-0.0008 (3)	0.0099 (2)	-0.0067 (3)
O1	0.0283 (9)	0.0686 (14)	0.0508 (12)	-0.0130 (9)	0.0130 (8)	-0.0050 (10)
O2	0.0429 (11)	0.0441 (11)	0.0426 (11)	0.0092 (8)	0.0099 (8)	0.0024 (8)
O3	0.0425 (11)	0.0563 (13)	0.0549 (12)	-0.0018 (9)	0.0201 (9)	-0.0229 (10)
C7	0.0239 (12)	0.0284 (12)	0.0400 (14)	-0.0010 (10)	0.0128 (10)	0.0001 (10)
C8	0.0287 (13)	0.0395 (15)	0.0466 (16)	-0.0077 (11)	0.0166 (12)	0.0011 (12)
C9	0.0392 (14)	0.0593 (18)	0.0380 (15)	-0.0038 (13)	0.0200 (12)	0.0103 (13)
C10	0.0260 (12)	0.0274 (12)	0.0318 (13)	0.0005 (10)	0.0103 (9)	0.0013 (10)
C11	0.0304 (13)	0.0516 (16)	0.0307 (14)	-0.0030 (12)	0.0077 (11)	0.0052 (11)
N1	0.0324 (11)	0.0387 (13)	0.0425 (13)	0.0040 (9)	0.0088 (10)	0.0010 (10)
C1	0.0460 (16)	0.0509 (18)	0.0490 (17)	0.0072 (13)	0.0221 (14)	-0.0056 (13)
C2	0.0547 (18)	0.0561 (19)	0.0350 (16)	-0.0007 (15)	0.0097 (13)	-0.0042 (13)
C3	0.061 (2)	0.074 (2)	0.061 (2)	-0.0227 (18)	0.0029 (17)	-0.0099 (18)
C4	0.0486 (19)	0.075 (2)	0.070 (2)	-0.0180 (17)	0.0150 (17)	0.0046 (17)
C5	0.0578 (19)	0.0477 (18)	0.0576 (19)	-0.0046 (14)	0.0196 (16)	0.0110 (14)
C6	0.055 (2)	0.102 (3)	0.064 (2)	-0.0095 (19)	0.0280 (17)	0.017 (2)

Geometric parameters (\AA , $^\circ$)

S1—O3	1.4452 (18)	C1—C6	1.518 (4)
S1—O1	1.4652 (18)	C1—C2	1.530 (4)
S1—O2	1.4687 (19)	C1—H1	0.9800
S1—C7	1.794 (3)	C2—C3	1.523 (4)
C7—C8	1.374 (3)	C2—H2A	0.9700
C7—C10	1.442 (3)	C2—H2B	0.9700
C8—C9	1.414 (4)	C3—C4	1.535 (5)
C8—H8	0.9300	C3—H3A	0.9700
C9—C11 ⁱ	1.367 (3)	C3—H3B	0.9700
C9—H9	0.9300	C4—C5	1.519 (4)
C10—C11	1.424 (3)	C4—H4A	0.9700

C10—C10 ⁱ	1.445 (4)	C4—H4B	0.9700
C11—C9 ⁱ	1.367 (3)	C5—H5A	0.9700
C11—H11	0.9300	C5—H5B	0.9700
N1—C5	1.498 (3)	C6—H6A	0.9600
N1—C1	1.509 (3)	C6—H6B	0.9600
N1—H1A	0.90	C6—H6C	0.9600
N1—H1B	0.90		
O3—S1—O1	113.33 (12)	C2—C1—H1	108.6
O3—S1—O2	112.56 (12)	C3—C2—C1	112.2 (2)
O1—S1—O2	111.33 (12)	C3—C2—H2A	109.2
O3—S1—C7	107.48 (11)	C1—C2—H2A	109.2
O1—S1—C7	105.63 (11)	C3—C2—H2B	109.2
O2—S1—C7	105.90 (11)	C1—C2—H2B	109.2
C8—C7—C10	120.8 (2)	H2A—C2—H2B	107.9
C8—C7—S1	118.69 (18)	C2—C3—C4	110.8 (2)
C10—C7—S1	120.49 (18)	C2—C3—H3A	109.5
C7—C8—C9	120.6 (2)	C4—C3—H3A	109.5
C7—C8—H8	119.7	C2—C3—H3B	109.5
C9—C8—H8	119.7	C4—C3—H3B	109.5
C11 ⁱ —C9—C8	120.6 (2)	H3A—C3—H3B	108.1
C11 ⁱ —C9—H9	119.7	C5—C4—C3	111.5 (3)
C8—C9—H9	119.7	C5—C4—H4A	109.3
C11—C10—C7	123.2 (2)	C3—C4—H4A	109.3
C11—C10—C10 ⁱ	118.8 (3)	C5—C4—H4B	109.3
C7—C10—C10 ⁱ	118.0 (3)	C3—C4—H4B	109.3
C9 ⁱ —C11—C10	121.3 (2)	H4A—C4—H4B	108.0
C9 ⁱ —C11—H11	119.3	N1—C5—C4	110.1 (2)
C10—C11—H11	119.3	N1—C5—H5A	109.6
C5—N1—C1	113.4 (2)	C4—C5—H5A	109.6
C5—N1—H1A	109.0	N1—C5—H5B	109.6
C1—N1—H1A	108.8	C4—C5—H5B	109.6
C5—N1—H1B	108.9	H5A—C5—H5B	108.1
C1—N1—H1B	108.9	C1—C6—H6A	109.5
H1A—N1—H1B	107.7	C1—C6—H6B	109.5
N1—C1—C6	109.3 (2)	H6A—C6—H6B	109.5
N1—C1—C2	108.2 (2)	C1—C6—H6C	109.5
C6—C1—C2	113.5 (3)	H6A—C6—H6C	109.5
N1—C1—H1	108.6	H6B—C6—H6C	109.5
C6—C1—H1	108.6		
O3—S1—C7—C8	120.4 (2)	S1—C7—C10—C10 ⁱ	-176.5 (2)
O1—S1—C7—C8	-0.8 (2)	C7—C10—C11—C9 ⁱ	-178.3 (2)
O2—S1—C7—C8	-119.0 (2)	C10 ⁱ —C10—C11—C9 ⁱ	0.4 (4)
O3—S1—C7—C10	-61.0 (2)	C5—N1—C1—C6	-177.8 (2)
O1—S1—C7—C10	177.70 (18)	C5—N1—C1—C2	58.2 (3)
O2—S1—C7—C10	59.5 (2)	N1—C1—C2—C3	-56.1 (3)
C10—C7—C8—C9	-1.1 (4)	C6—C1—C2—C3	-177.6 (3)
S1—C7—C8—C9	177.49 (19)	C1—C2—C3—C4	55.1 (4)

C7—C8—C9—C11 ⁱ	-0.7 (4)	C2—C3—C4—C5	-53.6 (4)
C8—C7—C10—C11	-179.3 (2)	C1—N1—C5—C4	-58.2 (3)
S1—C7—C10—C11	2.1 (3)	C3—C4—C5—N1	54.5 (4)
C8—C7—C10—C10 ⁱ	2.0 (4)		

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1B...O2	0.90	1.91	2.795 (3)	169
N1—H1A...O1 ⁱⁱ	0.90	1.93	2.820 (3)	169

Symmetry code: (ii) $-x+2, y-1/2, -z+3/2$.