organic compounds

Benzene-1,2-di(aminium) naphthalene-1,5-disulfonate methanol monosolvate trihydrate

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In the title salt, $C_6H_{10}N_2^{2+} \cdot C_{10}H_6O_6S_2^{2-} \cdot CH_3OH \cdot 3H_2O$, the cation lies on a mirror plane and the anion on a center of inversion. One lattice water molecule is located on a mirror plane, another is equally disordered over two sites. The methanol solvent molecule is disordered about a mirror plane. In the crystal, the cations, anions, water and methanol molecules are linked by O—H⋯O and N—H⋯O hydrogen bonds, forming a three-dimensional network.

Related literature

For other diammonium napthalene-1,5-disulfonates, see: Wei (2011); Zhu et al. (2009).
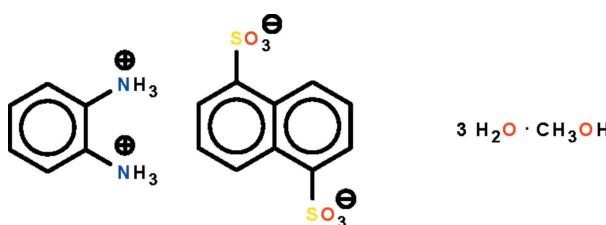
Experimental

Crystal data

$C_6H_{10}N_2^{2+} \cdot C_{10}H_6O_6S_2^{2-} \cdot CH_4O \cdot 3H_2O$
$M_r = 482.52$
Monoclinic, $P2_1/m$
$a = 8.1727$ (15) Å

$b = 13.681$ (2) Å
$c = 9.5173$ (15) Å
$\beta = 98.390$ (5)°
$V = 1052.8$ (3) Å^3
$Z = 2$

Mo $K\alpha$ radiation
$\mu = 0.31$ mm^{-1}
$T = 293$ K
$0.25 \times 0.22 \times 0.19$ mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.926$, $T_{max} = 0.943$

10378 measured reflections
2503 independent reflections
1935 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.176$
$S = 1.13$
2503 reflections

174 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.85$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O4—H4⋯O3	0.84	1.94	2.783 (6)	175
O1w—H1w⋯O2w	0.84	2.32	2.915 (6)	128
O1w—H1w⋯O2w′	0.84	2.19	2.799 (7)	130
O2w—H2w2⋯O3[i]	0.84	2.35	2.873 (5)	121
O2w′—H2w4⋯O3[i]	0.84	2.25	2.808 (6)	124
N1—H12⋯O1[ii]	0.88	2.07	2.880 (3)	153
N1—H11⋯O2w′	0.88	2.03	2.816 (5)	149
N2—H22⋯O1	0.88	1.99	2.815 (5)	156
N2—H21⋯O1w[iii]	0.88	1.88	2.735 (4)	164

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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supplementary materials

Benzene-1,2-di(aminium) naphthalene-1,5-disulfonate methanol monosolvate trihydrate

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Comment

Naphthalene-1,5-disulfonic acid has been characterized as several diammonium salts, *e.g.*, as the ethane-1,2-diammonium (Zhu *et al.*, 2009), the piperizine-1,4-diium (Wei, 2011) derivatives. In the salt, $C_6H_{10}N_2^{2+} \cdot C_{10}H_6O_6S_2^{2-} \cdot 3H_2O \cdot CH_3OH$ (Scheme I, Fig. 1), the cation lies on a mirror plane and the anion on a center-of-inversion. The cation, anion, water and methanol molecules are linked by O–H···O hydrogen bonds to form a three-dimensional network (Table 1).

Experimental

A methanol solution (5 ml) of 1,2-benzenediamine (1 mmol, 108 mg) was added to an aqueous solution (5 ml) of 1,5-naphthalenedisulfonic acid tetrahydrate (0.5 mmol, 180 mg). The mixture was heated at 343 K until the reactants dissolved completely. The solution was filtered; colorless crystals were isolated after several days.suitable for X-ray diffraction were isolated from the filtrate afterfour days.

Refinement

Carbon- and nitrogen-bound H-atoms were placed in calculated positions (C–H 0.93, N–H 0.88 Å) and were included in the refinement in the riding model approximation, with U(H) set to $1.2U$(C,N). The water H-atoms were placed in chemcally sensible positions on the basis of hydrogen bonding interactions (O–H 0.84 Å) and their temperature factors similar tied. One of the water molecules is disordered over a center-of-inversion.

Computing details

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).
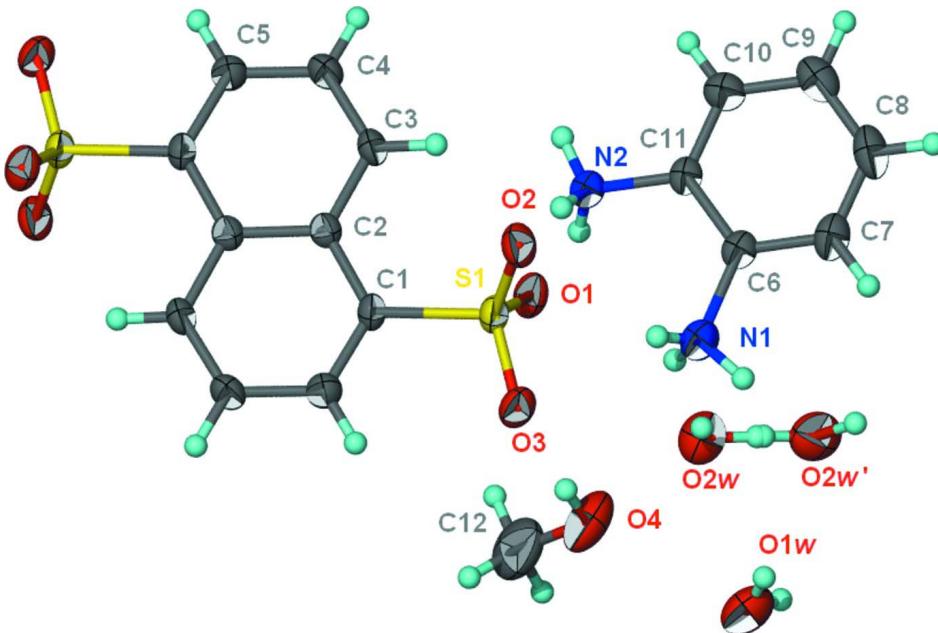
Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_6H_{10}N_2^{2+} \cdot C_{10}H_6O_6S_2^{2-} \cdot 3H_2O \cdot CH_3OH$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The symmetry-related atoms of the anion are not labeled.

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Crystal data

$C_6H_{10}N_2^{2+} \cdot C_{10}H_6O_6S_2^{2-} \cdot CH_4O \cdot 3H_2O$	$F(000) = 508$
$M_r = 482.52$	$D_x = 1.522$ Mg m^{-3}
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yb	Cell parameters from 6427 reflections
$a = 8.1727$ (15) Å	$\theta = 3.1–27.5°$
$b = 13.681$ (2) Å	$\mu = 0.31$ mm^{-1}
$c = 9.5173$ (15) Å	$T = 293$ K
$\beta = 98.390$ (5)°	Prism, colorless
$V = 1052.8$ (3) Å^3	$0.25 \times 0.22 \times 0.19$ mm
$Z = 2$	

Data collection

Rigaku R-AXIS RAPID IP diffractometer	10378 measured reflections
	2503 independent reflections
Radiation source: fine-focus sealed tube	1935 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.044$
ω scan	$\theta_{max} = 27.5°, \theta_{min} = 3.1°$
Absorption correction: multi-scan	$h = -10 \rightarrow 10$
(*ABSCOR*; Higashi, 1995)	$k = -16 \rightarrow 17$
$T_{min} = 0.926, T_{max} = 0.943$	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.055$	
$wR(F^2) = 0.176$	H-atom parameters constrained
$S = 1.13$	$w = 1/[\sigma^2(F_o^2) + (0.0909P)^2 + 0.6772P]$
2503 reflections	where $P = (F_o^2 + 2F_c^2)/3$
174 parameters	$(\Delta/\sigma)_{max} = 0.001$
0 restraints	$\Delta\rho_{max} = 0.85$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.41$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.71163 (7)	0.51770 (5)	0.72850 (6)	0.0322 (2)	
O1	0.6469 (2)	0.61258 (14)	0.6776 (2)	0.0425 (5)	
O2	0.6097 (2)	0.43755 (15)	0.6701 (2)	0.0443 (5)	
O3	0.7450 (3)	0.51585 (15)	0.8836 (2)	0.0433 (5)	
O1w	0.5509 (5)	0.7500	1.2108 (4)	0.0707 (10)	
H1w	0.4934	0.6998	1.1902	0.106*	
N1	0.4198 (5)	0.7500	0.7729 (4)	0.0509 (9)	
H11	0.3788	0.7500	0.8535	0.076*	
H12	0.4809	0.8025	0.7685	0.076*	
N2	0.4862 (4)	0.7500	0.4847 (3)	0.0368 (7)	
H21	0.4867	0.7500	0.3923	0.055*	
H22	0.5374	0.6975	0.5221	0.055*	
C1	0.9077 (3)	0.50513 (18)	0.6689 (3)	0.0300 (5)	
C2	0.9198 (3)	0.50489 (17)	0.5204 (3)	0.0292 (5)	
C3	0.7803 (3)	0.51357 (19)	0.4131 (3)	0.0338 (6)	
H3	0.6752	0.5191	0.4388	0.041*	
C4	0.7984 (3)	0.5140 (2)	0.2731 (3)	0.0387 (6)	
H4A	0.7057	0.5207	0.2045	0.046*	
C5	0.9552 (3)	0.5044 (2)	0.2309 (3)	0.0373 (6)	
H5	0.9659	0.5044	0.1349	0.045*	
C6	0.2850 (5)	0.7500	0.6545 (4)	0.0354 (8)	
C7	0.1240 (5)	0.7500	0.6818 (5)	0.0453 (10)	
H7	0.1025	0.7500	0.7751	0.054*	
C8	−0.0050 (5)	0.7500	0.5712 (6)	0.0527 (11)	
H8	−0.1133	0.7500	0.5904	0.063*	
C9	0.0245 (6)	0.7500	0.4325 (5)	0.0499 (10)	
H9	−0.0631	0.7500	0.3582	0.060*	
C10	0.1866 (5)	0.7500	0.4045 (4)	0.0431 (9)	
H10	0.2077	0.7500	0.3111	0.052*	
C11	0.3153 (4)	0.7500	0.5139 (4)	0.0315 (7)	
O4	0.7361 (8)	0.6961 (4)	1.0177 (6)	0.0782 (17)	0.50
H4	0.7426	0.6406	0.9812	0.117*	0.50
C12	0.8778 (11)	0.751 (2)	1.0006 (8)	0.090 (3)	0.5
H12A	0.8778	0.8110	1.0533	0.135*	0.50
H12B	0.8767	0.7658	0.9018	0.135*	0.50

H12C	0.9753	0.7143	1.0351	0.135*	0.50
O2w	0.4770 (6)	0.6054 (4)	0.9855 (5)	0.0635 (13)	0.50
H2w1	0.3897	0.6342	0.9987	0.095*	0.50
H2w2	0.4637	0.5451	0.9955	0.095*	0.50
O2w′	0.2979 (7)	0.6729 (4)	1.0117 (5)	0.0657 (13)	0.50
H2w3	0.3789	0.6375	1.0001	0.099*	0.50
H2w4	0.2196	0.6366	1.0271	0.099*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0272 (4)	0.0378 (4)	0.0335 (4)	0.0033 (2)	0.0108 (2)	0.0024 (2)
O1	0.0421 (11)	0.0428 (11)	0.0451 (10)	0.0138 (9)	0.0152 (8)	0.0056 (9)
O2	0.0313 (10)	0.0483 (12)	0.0553 (12)	−0.0061 (8)	0.0127 (8)	−0.0051 (10)
O3	0.0434 (11)	0.0557 (12)	0.0333 (10)	0.0051 (9)	0.0143 (8)	0.0054 (9)
O1w	0.086 (3)	0.083 (3)	0.0472 (18)	0.000	0.0224 (18)	0.000
N1	0.044 (2)	0.074 (3)	0.0366 (17)	0.000	0.0125 (15)	0.000
N2	0.0354 (17)	0.0422 (17)	0.0341 (16)	0.000	0.0096 (13)	0.000
C1	0.0241 (12)	0.0339 (12)	0.0335 (13)	0.0028 (9)	0.0096 (9)	0.0008 (10)
C2	0.0265 (13)	0.0297 (12)	0.0314 (12)	0.0016 (9)	0.0045 (9)	0.0021 (10)
C3	0.0219 (12)	0.0428 (14)	0.0367 (13)	0.0009 (10)	0.0050 (10)	0.0003 (11)
C4	0.0281 (13)	0.0520 (17)	0.0343 (14)	0.0034 (11)	−0.0007 (10)	0.0015 (11)
C5	0.0339 (14)	0.0480 (15)	0.0299 (12)	0.0026 (11)	0.0045 (10)	0.0024 (11)
C6	0.038 (2)	0.0342 (18)	0.0360 (18)	0.000	0.0101 (15)	0.000
C7	0.043 (2)	0.044 (2)	0.054 (2)	0.000	0.0200 (19)	0.000
C8	0.032 (2)	0.048 (2)	0.081 (3)	0.000	0.016 (2)	0.000
C9	0.038 (2)	0.045 (2)	0.064 (3)	0.000	−0.0007 (19)	0.000
C10	0.043 (2)	0.045 (2)	0.040 (2)	0.000	0.0021 (17)	0.000
C11	0.0317 (18)	0.0271 (16)	0.0372 (18)	0.000	0.0100 (14)	0.000
O4	0.098 (4)	0.069 (3)	0.079 (4)	−0.002 (3)	0.050 (3)	−0.012 (3)
C12	0.097 (6)	0.093 (6)	0.086 (5)	0.035 (14)	0.031 (4)	−0.009 (16)
O2w	0.067 (3)	0.060 (3)	0.066 (3)	0.004 (2)	0.020 (2)	0.002 (2)
O2w′	0.071 (3)	0.077 (3)	0.051 (3)	−0.010 (3)	0.015 (2)	0.011 (2)

Geometric parameters (Å, º)

S1—O2	1.439 (2)	C6—C7	1.377 (5)
S1—O1	1.4578 (19)	C6—C11	1.396 (5)
S1—O3	1.461 (2)	C7—C8	1.377 (7)
S1—C1	1.785 (2)	C7—H7	0.9300
O1w—H1w	0.8391	C8—C9	1.376 (7)
N1—C6	1.456 (5)	C8—H8	0.9300
N1—H11	0.8800	C9—C10	1.389 (6)
N1—H12	0.8800	C9—H9	0.9300
N2—C11	1.463 (4)	C10—C11	1.368 (5)
N2—H21	0.8800	C10—H10	0.9300
N2—H22	0.8800	O4—C12	1.41 (2)
C1—C5ⁱ	1.367 (4)	O4—H4	0.8400
C1—C2	1.430 (3)	C12—H12A	0.9600

C2—C3	1.420 (4)	C12—H12B	0.9600
C2—C2i	1.428 (5)	C12—H12C	0.9600
C3—C4	1.362 (4)	O2w—H2w1	0.8401
C3—H3	0.9300	O2w—H2w2	0.8400
C4—C5	1.404 (4)	O2w—H2w3	0.9423
C4—H4A	0.9300	O2w′—H2w1	0.9412
C5—C1i	1.367 (4)	O2w′—H2w3	0.8400
C5—H5	0.9300	O2w′—H2w4	0.8400
O2—S1—O1	112.86 (13)	C1i—C5—H5	120.1
O2—S1—O3	112.92 (12)	C4—C5—H5	120.1
O1—S1—O3	110.88 (12)	C7—C6—C11	119.2 (4)
O2—S1—C1	107.45 (12)	C7—C6—N1	119.3 (3)
O1—S1—C1	106.22 (12)	C11—C6—N1	121.5 (3)
O3—S1—C1	105.98 (12)	C8—C7—C6	120.1 (4)
C6—N1—H11	109.5	C8—C7—H7	119.9
C6—N1—H12	109.5	C6—C7—H7	119.9
H11—N1—H12	109.5	C9—C8—C7	120.8 (4)
C11—N2—H21	109.5	C9—C8—H8	119.6
C11—N2—H22	109.5	C7—C8—H8	119.6
H21—N2—H22	109.5	C8—C9—C10	119.3 (4)
C5i—C1—C2	121.4 (2)	C8—C9—H9	120.3
C5i—C1—S1	118.00 (19)	C10—C9—H9	120.3
C2—C1—S1	120.56 (19)	C11—C10—C9	120.2 (4)
C3—C2—C2i	119.0 (3)	C11—C10—H10	119.9
C3—C2—C1	123.1 (2)	C9—C10—H10	119.9
C2i—C2—C1	117.9 (3)	C10—C11—C6	120.4 (3)
C4—C3—C2	120.9 (2)	C10—C11—N2	120.3 (3)
C4—C3—H3	119.6	C6—C11—N2	119.3 (3)
C2—C3—H3	119.6	H2w1—O2w—H2w2	108.5
C3—C4—C5	120.9 (2)	H2w2—O2w—H2w3	108.3
C3—C4—H4A	119.5	H2w1—O2w′—H2w4	109.5
C5—C4—H4A	119.5	H2w3—O2w′—H2w4	108.5
C1i—C5—C4	119.9 (2)		
O2—S1—C1—C5i	−120.4 (2)	C3—C4—C5—C1i	−0.3 (4)
O1—S1—C1—C5i	118.6 (2)	C11—C6—C7—C8	0.000 (2)
O3—S1—C1—C5i	0.6 (3)	N1—C6—C7—C8	180.000 (1)
O2—S1—C1—C2	59.5 (2)	C6—C7—C8—C9	0.000 (2)
O1—S1—C1—C2	−61.5 (2)	C7—C8—C9—C10	0.000 (2)
O3—S1—C1—C2	−179.51 (19)	C8—C9—C10—C11	0.000 (1)
C5i—C1—C2—C3	179.3 (3)	C9—C10—C11—C6	0.0
S1—C1—C2—C3	−0.5 (3)	C9—C10—C11—N2	180.0
C5i—C1—C2—C2i	−0.5 (4)	C7—C6—C11—C10	0.000 (1)
S1—C1—C2—C2i	179.7 (2)	N1—C6—C11—C10	180.000 (1)
C2i—C2—C3—C4	−0.9 (4)	C7—C6—C11—N2	180.000 (1)
C1—C2—C3—C4	179.3 (2)	N1—C6—C11—N2	0.000 (1)
C2—C3—C4—C5	0.9 (4)		

Symmetry code: (i) −x+2, −y+1, −z+1.

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O4—H4⋯O3	0.84	1.94	2.783 (6)	175
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N2—H21⋯O1w^{iv}	0.88	1.88	2.735 (4)	164

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