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Bis(3-aminopropan-1-aminium) naphthalene-1,5-disulfonate dihydrate

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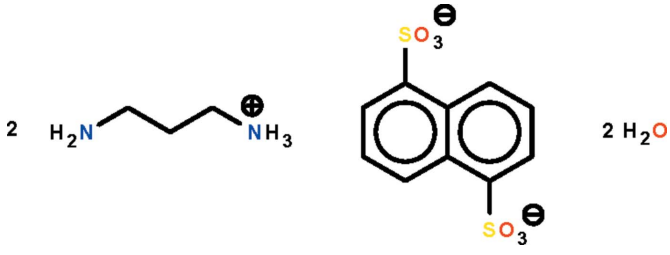
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.127; data-to-parameter ratio = 15.5.

In the title hydrated salt, $2C_3H_{11}N_2^+\cdot C_{10}H_6O_6S_2^{2-}\cdot 2H_2O$, the anion lies on a center of inversion; its sulfonate $-SO_3$ group features one S—O bond that is longer than the other two. The O atom of this longer bond is the hydrogen-bond acceptor to the amino H atom of one cation and the ammonium H atom of another cation. In the crystal, N—H···O and O—H···O hydrogen bonds link the cations, anions and water molecules into a three-dimensional network.

Related literature

For a related structure, see: Gao & Ng (2012).



Experimental

Crystal data

$2C_3H_{11}N_2^+\cdot C_{10}H_6O_6S_2^{2-}\cdot 2H_2O$
M_r = 472.58
Triclinic, $P\overline{1}$
a = 8.0913 (16) Å
b = 8.1917 (16) Å
c = 9.8062 (18) Å
α = 71.182 (5)°
β = 65.950 (5)°
γ = 79.945 (5)°
V = 561.12 (19) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 293 K
0.21 × 0.17 × 0.17 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.942, T_{max} = 0.953
5584 measured reflections
2552 independent reflections
2268 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.127
S = 1.05
2552 reflections
165 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1w—H1w1···O1	0.84 (1)	2.00 (1)	2.826 (2)	166 (3)
O1w—H1w2···N1	0.84 (1)	1.91 (1)	2.736 (2)	172 (3)
N1—H12···O3^{i}	0.88 (1)	2.55 (2)	3.317 (2)	147 (2)
N2—H21···O1w^{ii}	0.89 (1)	1.84 (1)	2.715 (2)	172 (2)
N2—H22···O2^{iii}	0.89 (1)	1.98 (1)	2.845 (2)	165 (3)
N2—H23···O3^{iv}	0.88 (1)	1.97 (1)	2.825 (2)	164 (2)

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

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 data and figures for this paper are available from the IUCr electronic archives (Reference: XU5521).

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supporting information

Acta Cryst. (2012). E**68**, o1575 [doi:10.1107/S1600536812018296]

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S1. Comment

The study continues with the preceding study on ammonium naphthalene-1,5-disulfonates (Gao & Ng, 2012). The synthesis of the propane-1,3-diammonium derivative yielded instead the bis(3-aminopropylammonium) salt, which exists as a dihydrate (Scheme I), *i.e.*, only one of the two amino groups is protonated so that the cation has a monopositive charge only. The anion of $2(C_3H_{11}N_2)^{2+}\cdot(C_{10}H_6O_6S_2)^{2-}\cdot 2H_2O$ lies on a center-of-inversion (Fig. 1). The sulfonate $-SO_3$ group features one S–O bond that is longer than the other two; the O atom of this longer bond is hydrogen-bond acceptor two the amino H atom of one cation and the ammonium H atom of another cation. Hydrogen bonds link the cation, anion and water molecule into a three-dimensional network.

S2. Experimental

A methanol solution (5 ml) of 1,3-propanediamine (98% pure, 1 mmol, 0.08 ml) was added to an aqueous solution (5 ml) of 1,5-naphthalenedisulfonic acid tetrahydrate (0.5 mmol, 180 mg). The mixture heated at 343 K until the reacctants dissolved completely; colorless crystals were isolated from the filtrate after several days. three days.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U(C). The amino and water H-atoms were located in a difference Fourier map, and were refined with distance restraints N–H 0.88±0.01 Å, O–H 0.84±0.01 Å and H···H 1.37±0.01 Å; their temperature factors were refined.

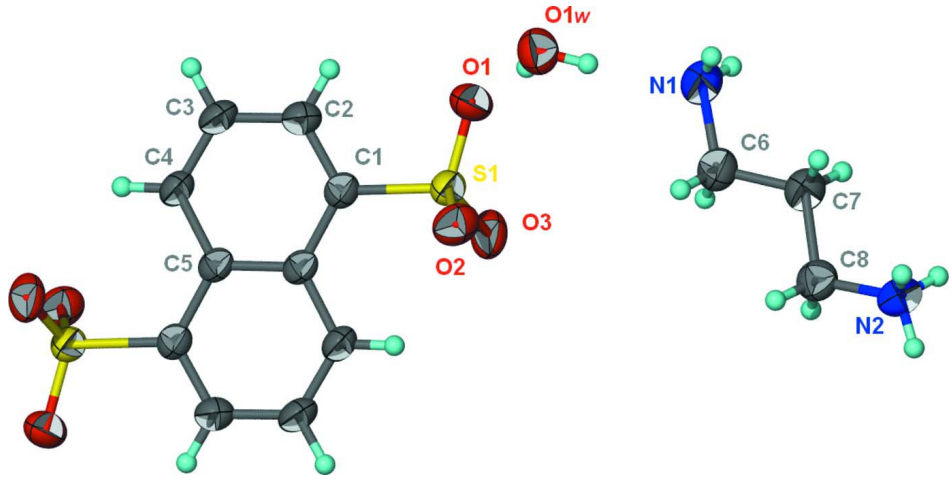

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $2(C_3H_{11}N_2)^{2+}\cdot(C_{10}H_6O_6S_2)^{2-}\cdot 2H_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. Symmetry-related atoms are not labeled.

Bis(3-aminopropan-1-aminium) naphthalene-1,5-disulfonate dihydrate

Crystal data

$2C_3H_{11}N_2^+\cdot C_{10}H_6O_6S_2^{2-}\cdot 2H_2O$
$M_r = 472.58$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.0913\ (16)$ Å
$b = 8.1917\ (16)$ Å
$c = 9.8062\ (18)$ Å
$\alpha = 71.182\ (5)°$
$\beta = 65.950\ (5)°$
$\gamma = 79.945\ (5)°$
$V = 561.12\ (19)$ Å^3
$Z = 1$
$F(000) = 252$
$D_x = 1.399$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3803 reflections
$\theta = 3.0–27.5°$
$\mu = 0.29$ mm^{-1}
$T = 293$ K
Prism, colorless
0.21 × 0.17 × 0.17 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.942$, $T_{max} = 0.953$
5584 measured reflections
2552 independent reflections
2268 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -10\rightarrow 9$
$k = -10\rightarrow 10$
$l = -12\rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.127$
$S = 1.05$
2552 reflections
165 parameters
7 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

$w = 1/[\sigma^2(F_o^2) + (0.0736P)^2 + 0.1592P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.081 (12)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.27701 (5)	0.72860 (5)	0.28439 (4)	0.02880 (18)
O1	0.40860 (17)	0.78029 (17)	0.12668 (14)	0.0406 (3)
O2	0.35741 (17)	0.62846 (16)	0.39549 (15)	0.0402 (3)
O3	0.12666 (17)	0.64501 (17)	0.29413 (17)	0.0425 (3)
O1W	0.75757 (19)	0.63049 (19)	0.10446 (16)	0.0421 (3)
H1W1	0.6524 (18)	0.661 (3)	0.106 (3)	0.057 (7)*
H1W2	0.766 (4)	0.5307 (17)	0.096 (3)	0.063 (8)*
N1	0.8221 (3)	0.3040 (2)	0.0702 (2)	0.0437 (4)
H11	0.9387 (16)	0.288 (3)	0.053 (3)	0.063 (8)*
H12	0.792 (4)	0.294 (4)	−0.003 (2)	0.067 (8)*
N2	0.7492 (2)	−0.3142 (2)	0.36576 (18)	0.0367 (4)
H21	0.744 (3)	−0.340 (3)	0.287 (2)	0.059 (7)*
H22	0.701 (3)	−0.400 (3)	0.450 (2)	0.065 (7)*
H23	0.8614 (16)	−0.323 (3)	0.361 (3)	0.046 (6)*
C1	0.1830 (2)	0.9236 (2)	0.33721 (18)	0.0269 (3)
C2	0.2538 (2)	1.0749 (2)	0.23475 (18)	0.0320 (4)
H2	0.3496	1.0745	0.1411	0.038*
C3	0.1822 (2)	1.2322 (2)	0.27039 (19)	0.0341 (4)
H3	0.2318	1.3352	0.2003	0.041*
C4	0.0411 (2)	1.2350 (2)	0.40653 (18)	0.0297 (4)
H4A	−0.0057	1.3403	0.4274	0.036*
C5	−0.03570 (19)	1.08034 (19)	0.51689 (17)	0.0251 (3)
C6	0.7250 (3)	0.1771 (2)	0.2140 (2)	0.0387 (4)
H6A	0.5962	0.1970	0.2356	0.046*
H6B	0.7462	0.1951	0.2985	0.046*
C7	0.7781 (3)	−0.0097 (2)	0.2119 (2)	0.0385 (4)
H7A	0.7530	−0.0300	0.1302	0.046*
H7B	0.9073	−0.0301	0.1882	0.046*
C8	0.6774 (2)	−0.1352 (2)	0.3650 (2)	0.0360 (4)
H8A	0.6896	−0.1063	0.4488	0.043*
H8B	0.5496	−0.1262	0.3825	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0281 (3)	0.0244 (2)	0.0307 (3)	−0.00188 (15)	−0.00813 (18)	−0.00714 (17)
O1	0.0406 (7)	0.0360 (7)	0.0315 (6)	0.0011 (5)	−0.0028 (5)	−0.0076 (5)
O2	0.0405 (7)	0.0329 (7)	0.0413 (7)	0.0000 (5)	−0.0168 (6)	−0.0019 (5)
O3	0.0366 (7)	0.0390 (7)	0.0580 (8)	−0.0049 (5)	−0.0135 (6)	−0.0256 (6)
O1W	0.0412 (7)	0.0395 (8)	0.0456 (8)	0.0018 (6)	−0.0141 (6)	−0.0169 (6)

N1	0.0528 (10)	0.0322 (8)	0.0420 (9)	−0.0036 (7)	−0.0147 (8)	−0.0085 (7)
N2	0.0376 (8)	0.0327 (8)	0.0368 (8)	−0.0065 (6)	−0.0146 (7)	−0.0028 (6)
C1	0.0298 (7)	0.0245 (8)	0.0278 (7)	−0.0035 (6)	−0.0122 (6)	−0.0063 (6)
C2	0.0339 (8)	0.0307 (9)	0.0262 (7)	−0.0073 (6)	−0.0066 (6)	−0.0047 (6)
C3	0.0418 (9)	0.0238 (8)	0.0311 (8)	−0.0107 (7)	−0.0097 (7)	−0.0011 (6)
C4	0.0368 (8)	0.0210 (8)	0.0297 (8)	−0.0063 (6)	−0.0110 (7)	−0.0043 (6)
C5	0.0273 (7)	0.0233 (8)	0.0265 (7)	−0.0050 (6)	−0.0123 (6)	−0.0046 (6)
C6	0.0396 (9)	0.0340 (9)	0.0424 (9)	−0.0042 (7)	−0.0133 (8)	−0.0123 (8)
C7	0.0425 (9)	0.0321 (9)	0.0390 (9)	−0.0020 (7)	−0.0143 (8)	−0.0087 (7)
C8	0.0337 (8)	0.0347 (9)	0.0395 (9)	−0.0041 (7)	−0.0125 (7)	−0.0106 (7)

Geometric parameters (Å, º)

S1—O1	1.4484 (13)	C2—H2	0.9300
S1—O2	1.4484 (13)	C3—C4	1.362 (2)
S1—O3	1.4532 (13)	C3—H3	0.9300
S1—C1	1.7848 (16)	C4—C5	1.416 (2)
O1W—H1W1	0.840 (10)	C4—H4A	0.9300
O1W—H1W2	0.837 (10)	C5—C5^{i}	1.427 (3)
N1—C6	1.457 (2)	C5—C1^{i}	1.436 (2)
N1—H11	0.883 (10)	C6—C7	1.520 (2)
N1—H12	0.879 (10)	C6—H6A	0.9700
N2—C8	1.479 (2)	C6—H6B	0.9700
N2—H21	0.886 (10)	C7—C8	1.508 (2)
N2—H22	0.885 (10)	C7—H7A	0.9700
N2—H23	0.881 (9)	C7—H7B	0.9700
C1—C2	1.360 (2)	C8—H8A	0.9700
C1—C5^{i}	1.436 (2)	C8—H8B	0.9700
C2—C3	1.408 (2)		
O1—S1—O2	113.11 (8)	C3—C4—C5	121.11 (15)
O1—S1—O3	111.96 (8)	C3—C4—H4A	119.4
O2—S1—O3	112.63 (8)	C5—C4—H4A	119.4
O1—S1—C1	106.07 (7)	C4—C5—C5^{i}	119.01 (17)
O2—S1—C1	106.32 (8)	C4—C5—C1^{i}	123.18 (14)
O3—S1—C1	106.10 (7)	C5^{i}—C5—C1^{i}	117.81 (16)
H1W1—O1W—H1W2	100 (2)	N1—C6—C7	114.59 (16)
C6—N1—H11	108.1 (17)	N1—C6—H6A	108.6
C6—N1—H12	107.7 (18)	C7—C6—H6A	108.6
H11—N1—H12	115 (3)	N1—C6—H6B	108.6
C8—N2—H21	110.4 (16)	C7—C6—H6B	108.6
C8—N2—H22	119.3 (18)	H6A—C6—H6B	107.6
H21—N2—H22	107 (2)	C8—C7—C6	112.28 (15)
C8—N2—H23	108.1 (15)	C8—C7—H7A	109.1
H21—N2—H23	111 (2)	C6—C7—H7A	109.1
H22—N2—H23	101 (2)	C8—C7—H7B	109.1
C2—C1—C5^{i}	121.35 (15)	C6—C7—H7B	109.1
C2—C1—S1	118.04 (13)	H7A—C7—H7B	107.9

C5^{i}—C1—S1	120.60 (12)	N2—C8—C7	110.76 (14)
C1—C2—C3	120.06 (15)	N2—C8—H8A	109.5
C1—C2—H2	120.0	C7—C8—H8A	109.5
C3—C2—H2	120.0	N2—C8—H8B	109.5
C4—C3—C2	120.65 (15)	C7—C8—H8B	109.5
C4—C3—H3	119.7	H8A—C8—H8B	108.1
C2—C3—H3	119.7		
O1—S1—C1—C2	−4.68 (15)	S1—C1—C2—C3	179.05 (12)
O2—S1—C1—C2	115.97 (14)	C1—C2—C3—C4	−0.5 (3)
O3—S1—C1—C2	−123.91 (14)	C2—C3—C4—C5	1.0 (3)
O1—S1—C1—C5^{i}	174.63 (12)	C3—C4—C5—C5^{i}	−0.7 (3)
O2—S1—C1—C5^{i}	−64.71 (14)	C3—C4—C5—C1^{i}	179.21 (15)
O3—S1—C1—C5^{i}	55.41 (14)	N1—C6—C7—C8	178.34 (16)
C5^{i}—C1—C2—C3	−0.3 (2)	C6—C7—C8—N2	−172.59 (15)

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

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N2—H23···O3^{v}	0.88 (1)	1.97 (1)	2.825 (2)	164 (2)

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