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Bis(butan-1-aminium) naphthalene-1,5-disulfonate

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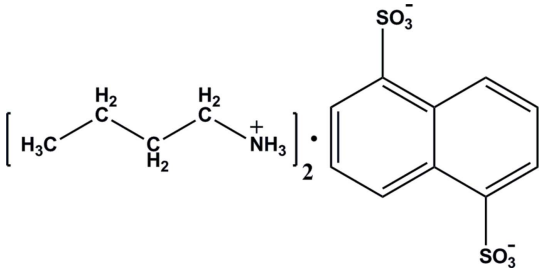
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.094; data-to-parameter ratio = 18.1.

In the title compound, $2C_4H_{12}N^+\cdot C_{10}H_6O_6S_2^{2-}$, the anion lies on an inversion center, so the asymmetric unit contains half an anion and one cation which are linked by a strong N—H···O hydrogen bond. The crystal structure comprises discrete ions, which are linked into centrosymmetric $R_4^4(12)$ loops by N—H···O interactions.

Related literature

For related structures, see: Jin (2011*a*,*b*, 2012). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$2C_4H_{12}N^+\cdot C_{10}H_6O_6S_2^{2-}$
M_r = 434.56
Monoclinic, $P2_1/c$
a = 8.1532 (16) Å
b = 9.2582 (19) Å
c = 14.108 (5) Å
β = 108.02 (3)°
V = 1012.7 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 293 K
0.3 × 0.3 × 0.2 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.489, T_{max} = 1.000
10171 measured reflections
2316 independent reflections
2039 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.094
S = 1.14
2316 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1	0.89	1.95	2.840 (2)	177
N1—H1C···O2^{i}	0.89	1.97	2.857 (2)	177
N1—H1B···O3^{ii}	0.89	2.05	2.911 (2)	162

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

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2406).

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Jin, Y. (2012). *Acta Cryst.* E**68**, o90.
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supplementary materials

Acta Cryst. (2012). E**68**, o1595 [doi:10.1107/S1600536812018880]

Bis(butan-1-aminium) naphthalene-1,5-disulfonate

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Comment

As an extension of the research about naphthalene-1,5-disulfonate salts (Jin, 2011*a*; Jin, 2011*b*; Jin, 2012), I report here the synthesis and the crystal structure of the title complex, $2C_4H_{12}N^+C_{10}H_6S_2O_6^{2-}$. In the title compound 2 $C_4H_{12}N^+$. $C_{10}H_6S_2O_6^2$, the anion lies on inversion center, so the asymmetric unit contains one-half anion and one cation which are linked by one strong N—H···O hydrogen bond interaction, Fig 1. The crystal structure comprises discrete ions which are linked into centrosymmetric $R_4^4(12)$ dimers by simple N—H···O interactions, (Bernstein, *et al.*, 1995), Fig 2, Table1.

Experimental

$2C_4H_{12}N^+$•$C_{10}H_6S_2O_6^{2-}$ was synthetized from a mixture of $CH_3(CH_2)_3NH_2$ (146.28 mg, 2.00 mmol), $C_{10}H_8O_6S_2$ (288.28 mg, 1.00 mmol), and distilled water (10 ml), which was stirred a few minutes at room temperature, giving a clear transparent solution. After evaporation for a few days, block colorless crystals suitable for X-ray diffraction were obtained in about 88% yield and filtered and washed with distilled water.

Refinement

H atoms bound to carbon and nitrogen were placed at idealized positions [C—H =0.93 to 0.97 Å and N—H = 0.89 Å] and allowed to ride on their parent atoms with U_{iso} fixed at 1.2 U_{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: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

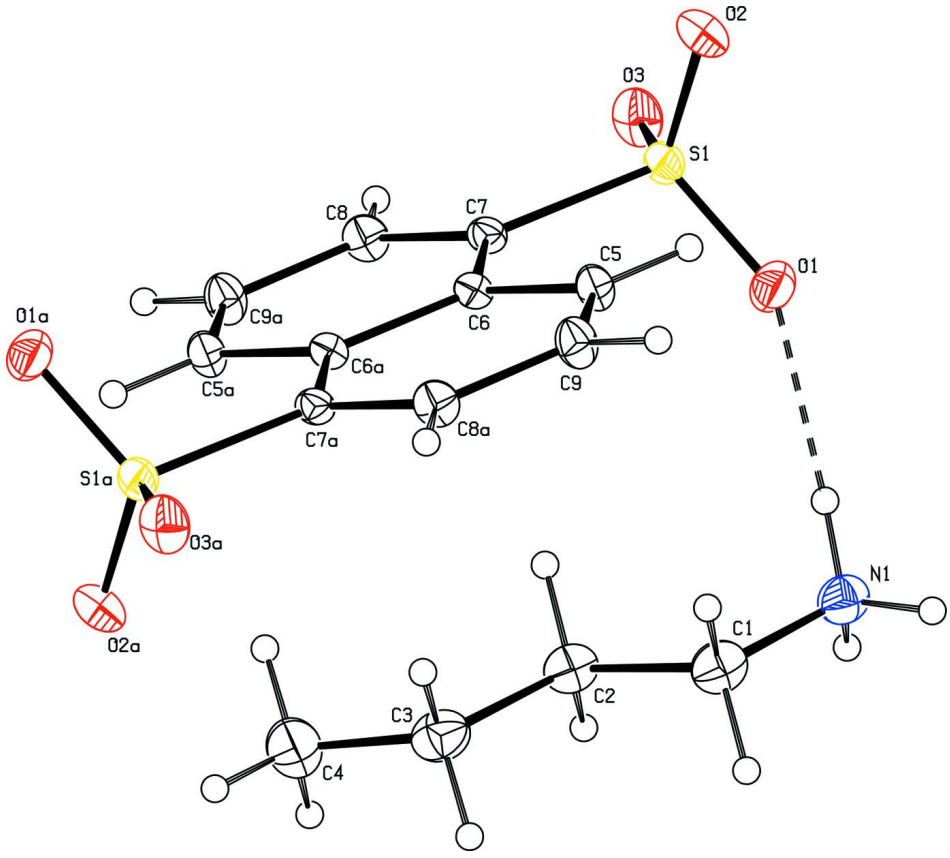

Figure 1

A view of the title compound showing the labelling of the non-H atoms. Displacement ellipsoids drawn at the 30% probability level. [Symmetry code: (a) 1-x, 2-y, 2-z]

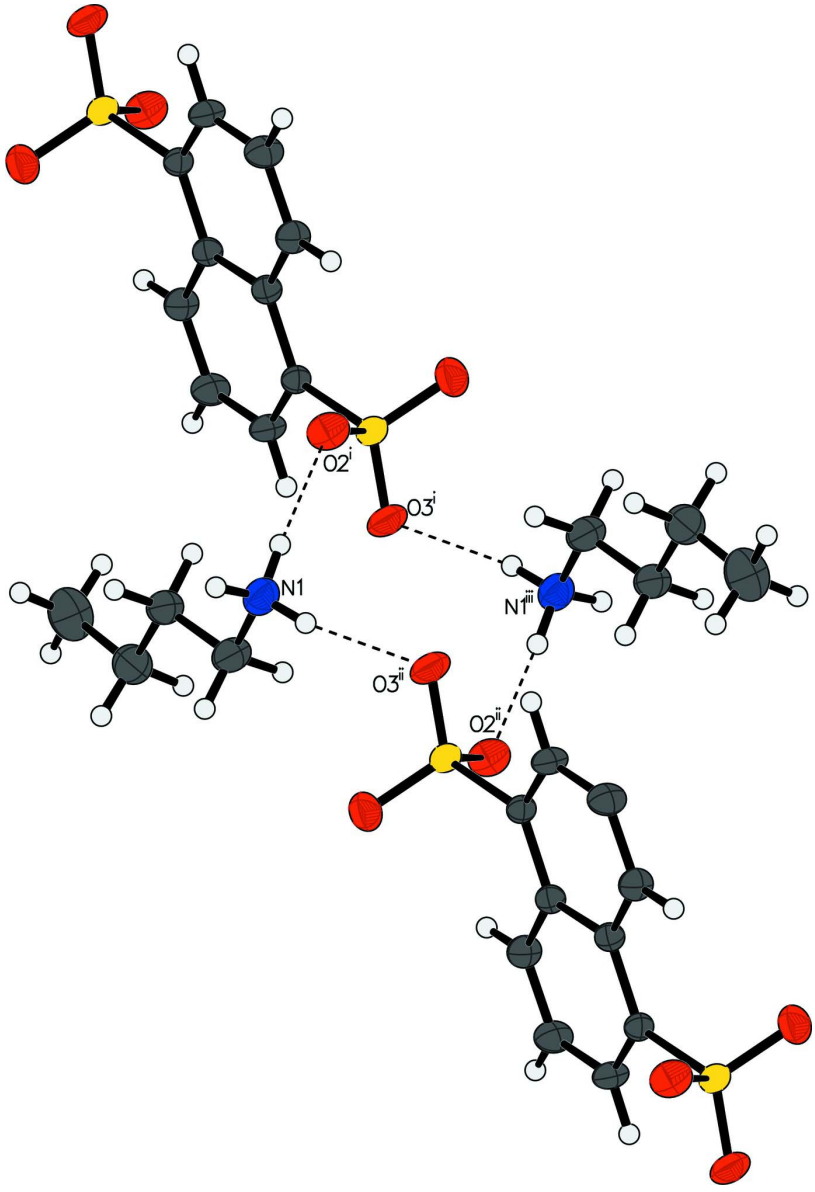

Figure 2

Part of the crystal structure of (I) , showing discrete ions which are linked into centrosymmetric $R_4^4(12)$ dimers. [Symmetry codes : (i) x, 3/2-y, -1/2+z; (ii) -x, 1/2+y, 3/2-z; (iii) -x,2-y,1-z]

Bis(butan-1-aminium) naphthalene-1,5-disulfonate

Crystal data

$2C_4H_{12}N^+·C_{10}H_6O_6S_2^{2-}$
M_r = 434.56
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.1532 (16) Å
b = 9.2582 (19) Å
c = 14.108 (5) Å
β = 108.02 (3)°
V = 1012.7 (5) Å^3
Z = 2
F(000) = 464
D_x = 1.425 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3450 reflections
θ = 6.2–55.3°
μ = 0.30 mm^{-1}

T = 293 K
Block, colourless

0.3 × 0.3 × 0.2 mm

Data collection

Rigaku Mercury CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.489, T_{max} = 1.000

10171 measured reflections
2316 independent reflections
2039 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −10→10
k = −12→12
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.094
S = 1.14
2316 reflections
128 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.4216P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.2319 (2)	1.0297 (2)	0.69402 (14)	0.0364 (4)
H1D	0.2261	1.0939	0.7473	0.044*
H1E	0.2235	1.0880	0.6356	0.044*
C2	0.4015 (2)	0.95077 (19)	0.72490 (13)	0.0332 (4)
H2A	0.4062	0.8866	0.6714	0.040*
H2B	0.4085	0.8919	0.7829	0.040*
C3	0.5553 (3)	1.0519 (2)	0.74938 (15)	0.0405 (5)
H3A	0.5439	1.1157	0.6932	0.049*
H3B	0.5553	1.1112	0.8061	0.049*
C4	0.7256 (3)	0.9722 (3)	0.77313 (17)	0.0524 (6)
H4A	0.7286	0.9168	0.7161	0.079*
H4B	0.7376	0.9087	0.8286	0.079*
H4C	0.8186	1.0406	0.7896	0.079*
C5	0.2728 (2)	1.06642 (17)	0.97032 (12)	0.0253 (3)
H5	0.1617	1.0294	0.9555	0.030*

C6	0.41452 (18)	0.97062 (16)	0.98793 (11)	0.0198 (3)
C7	0.39614 (19)	0.81664 (16)	0.98343 (11)	0.0204 (3)
C8	0.5368 (2)	0.72878 (17)	1.00271 (12)	0.0259 (3)
H8	0.5228	0.6290	1.0009	0.031*
C9	0.2973 (2)	1.21166 (18)	0.97477 (13)	0.0286 (4)
H9	0.2025	1.2727	0.9627	0.034*
N1	0.0850 (2)	0.92591 (16)	0.67115 (11)	0.0338 (3)
H1A	0.0840	0.8815	0.7269	0.041*
H1B	−0.0136	0.9735	0.6455	0.041*
H1C	0.0971	0.8608	0.6274	0.041*
O1	0.09524 (16)	0.78595 (15)	0.85199 (9)	0.0380 (3)
O2	0.11042 (16)	0.77995 (14)	1.02545 (9)	0.0343 (3)
O3	0.21663 (16)	0.57911 (13)	0.95327 (10)	0.0353 (3)
S1	0.18812 (5)	0.73440 (4)	0.95082 (3)	0.02387 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0436 (11)	0.0273 (9)	0.0380 (10)	0.0057 (8)	0.0123 (8)	0.0024 (7)
C2	0.0409 (10)	0.0277 (9)	0.0313 (9)	0.0033 (7)	0.0118 (8)	−0.0002 (7)
C3	0.0491 (12)	0.0362 (10)	0.0351 (10)	−0.0046 (9)	0.0113 (9)	0.0010 (8)
C4	0.0407 (12)	0.0699 (16)	0.0470 (12)	−0.0053 (11)	0.0139 (10)	−0.0027 (11)
C5	0.0186 (7)	0.0255 (8)	0.0320 (8)	−0.0005 (6)	0.0078 (6)	0.0015 (6)
C6	0.0197 (7)	0.0203 (7)	0.0206 (7)	−0.0011 (6)	0.0081 (6)	0.0008 (5)
C7	0.0212 (7)	0.0202 (7)	0.0204 (7)	−0.0037 (6)	0.0073 (6)	0.0008 (6)
C8	0.0282 (8)	0.0166 (7)	0.0333 (8)	−0.0006 (6)	0.0101 (7)	0.0011 (6)
C9	0.0225 (8)	0.0235 (8)	0.0400 (9)	0.0052 (6)	0.0100 (7)	0.0025 (7)
N1	0.0363 (8)	0.0338 (8)	0.0320 (8)	0.0092 (6)	0.0116 (6)	0.0033 (6)
O1	0.0318 (7)	0.0443 (8)	0.0306 (7)	−0.0089 (6)	−0.0010 (5)	0.0079 (5)
O2	0.0315 (6)	0.0355 (7)	0.0417 (7)	−0.0059 (5)	0.0198 (6)	−0.0001 (5)
O3	0.0332 (7)	0.0219 (6)	0.0481 (8)	−0.0085 (5)	0.0084 (6)	−0.0019 (5)
S1	0.0220 (2)	0.0218 (2)	0.0267 (2)	−0.00585 (14)	0.00604 (15)	0.00118 (15)

Geometric parameters (Å, º)

C1—N1	1.491 (2)	C5—H5	0.9300
C1—C2	1.505 (3)	C6—C7	1.433 (2)
C1—H1D	0.9700	C6—C6^{i}	1.436 (3)
C1—H1E	0.9700	C7—C8	1.363 (2)
C2—C3	1.517 (3)	C7—S1	1.7847 (15)
C2—H2A	0.9700	C8—C9^{i}	1.403 (2)
C2—H2B	0.9700	C8—H8	0.9300
C3—C4	1.516 (3)	C9—C8^{i}	1.403 (2)
C3—H3A	0.9700	C9—H9	0.9300
C3—H3B	0.9700	N1—H1A	0.8900
C4—H4A	0.9600	N1—H1B	0.8900
C4—H4B	0.9600	N1—H1C	0.8900
C4—H4C	0.9600	O1—S1	1.4464 (14)
C5—C9	1.358 (2)	O2—S1	1.4493 (13)
C5—C6	1.416 (2)	O3—S1	1.4550 (13)

N1—C1—C2	110.73 (15)	C6—C5—H5	119.6
N1—C1—H1D	109.5	C5—C6—C7	123.16 (14)
C2—C1—H1D	109.5	C5—C6—C6^{i}	118.93 (17)
N1—C1—H1E	109.5	C7—C6—C6^{i}	117.91 (17)
C2—C1—H1E	109.5	C8—C7—C6	120.99 (14)
H1D—C1—H1E	108.1	C8—C7—S1	118.08 (12)
C1—C2—C3	112.76 (16)	C6—C7—S1	120.93 (11)
C1—C2—H2A	109.0	C7—C8—C9^{i}	120.22 (15)
C3—C2—H2A	109.0	C7—C8—H8	119.9
C1—C2—H2B	109.0	C9^{i}—C8—H8	119.9
C3—C2—H2B	109.0	C5—C9—C8^{i}	121.16 (15)
H2A—C2—H2B	107.8	C5—C9—H9	119.4
C4—C3—C2	112.72 (18)	C8^{i}—C9—H9	119.4
C4—C3—H3A	109.0	C1—N1—H1A	109.5
C2—C3—H3A	109.0	C1—N1—H1B	109.5
C4—C3—H3B	109.0	H1A—N1—H1B	109.5
C2—C3—H3B	109.0	C1—N1—H1C	109.5
H3A—C3—H3B	107.8	H1A—N1—H1C	109.5
C3—C4—H4A	109.5	H1B—N1—H1C	109.5
C3—C4—H4B	109.5	O1—S1—O2	112.83 (9)
H4A—C4—H4B	109.5	O1—S1—O3	112.40 (8)
C3—C4—H4C	109.5	O2—S1—O3	111.89 (8)
H4A—C4—H4C	109.5	O1—S1—C7	106.27 (8)
H4B—C4—H4C	109.5	O2—S1—C7	106.42 (8)
C9—C5—C6	120.77 (15)	O3—S1—C7	106.46 (7)
C9—C5—H5	119.6		

N1—C1—C2—C3	−179.79 (15)	S1—C7—C8—C9^{i}	177.76 (13)
C1—C2—C3—C4	−175.79 (16)	C6—C5—C9—C8^{i}	−0.3 (3)
C9—C5—C6—C7	−179.48 (15)	C8—C7—S1—O1	−119.32 (13)
C9—C5—C6—C6^{i}	0.9 (3)	C6—C7—S1—O1	59.88 (14)
C5—C6—C7—C8	−178.83 (15)	C8—C7—S1—O2	120.19 (13)
C6^{i}—C6—C7—C8	0.8 (3)	C6—C7—S1—O2	−60.61 (14)
C5—C6—C7—S1	2.0 (2)	C8—C7—S1—O3	0.70 (15)
C6^{i}—C6—C7—S1	−178.37 (13)	C6—C7—S1—O3	179.90 (12)
C6—C7—C8—C9^{i}	−1.4 (2)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1	0.89	1.95	2.840 (2)	177
N1—H1*C*···O2^{ii}	0.89	1.97	2.857 (2)	177
N1—H1*B*···O3^{iii}	0.89	2.05	2.911 (2)	162

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