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

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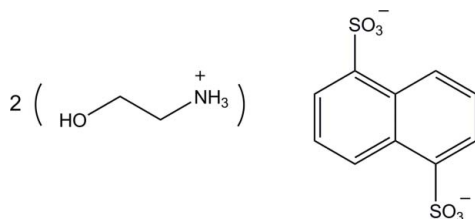
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.029; wR factor = 0.080; data-to-parameter ratio = 14.1.

In the crystal structure of the title compound, $2\text{C}_2\text{H}_8\text{NO}^+\cdot\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2^{2-}$, the anion lies on an inversion centre. The components are held together by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond, forming a 2:1 aggregate. The aggregates are further connected by $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For related structures, see: Gao *et al.* (2005); Li & Chai (2007); Russell *et al.* (1997); Sakwa & Wheeler (2003); Wang *et al.* (2008); Zhang *et al.* (2005).



Experimental

Crystal data

$2\text{C}_2\text{H}_8\text{NO}^+\cdot\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2^{2-}$
 $M_r = 410.48$
 Monoclinic, $P2_1/c$
 $a = 9.7946$ (14) Å
 $b = 8.9011$ (13) Å
 $c = 10.4050$ (16) Å
 $\beta = 104.334$ (2)°

$V = 878.9$ (2) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.35$ mm⁻¹
 $T = 293$ K
 $0.42 \times 0.34 \times 0.30$ mm

Data collection

Bruker APEX area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.855$, $T_{\max} = 0.898$

4721 measured reflections
 1718 independent reflections
 1625 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.080$
 $S = 1.04$
 1718 reflections
 122 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.43$ e Å⁻³
 $\Delta\rho_{\min} = -0.34$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O4}-\text{H4}^{\text{i}}\cdots\text{O2}$	0.835 (10)	2.041 (10)	2.840 (2)	160 (2)
$\text{N1}-\text{H1A}\cdots\text{O4}^{\text{i}}$	0.89	2.13	2.934 (2)	149
$\text{N1}-\text{H1B}\cdots\text{O1}^{\text{ii}}$	0.89	1.90	2.766 (2)	164
$\text{N1}-\text{H1C}\cdots\text{O3}^{\text{iii}}$	0.89	1.96	2.837 (2)	168
$\text{C3}-\text{H3}\cdots\text{O1}^{\text{iv}}$	0.93	2.41	3.270 (2)	154
$\text{C5}-\text{H5}\cdots\text{O2}^{\text{v}}$	0.93	2.55	3.459 (2)	167

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

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; 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.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2725).

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supporting information

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

Cong Wang and Sheng Li Yang

S1. Comment

Several supramolecular structures of naphthalene-1,5-disulfonate have been reported previously (Russell *et al.*, 1997; Zhang *et al.*, 2005; Gao *et al.*, 2005; Wang *et al.*, 2008). As an extension of research, we report here the synthesis and structure of the title compound, (I) (Table 1 & Fig. 1).

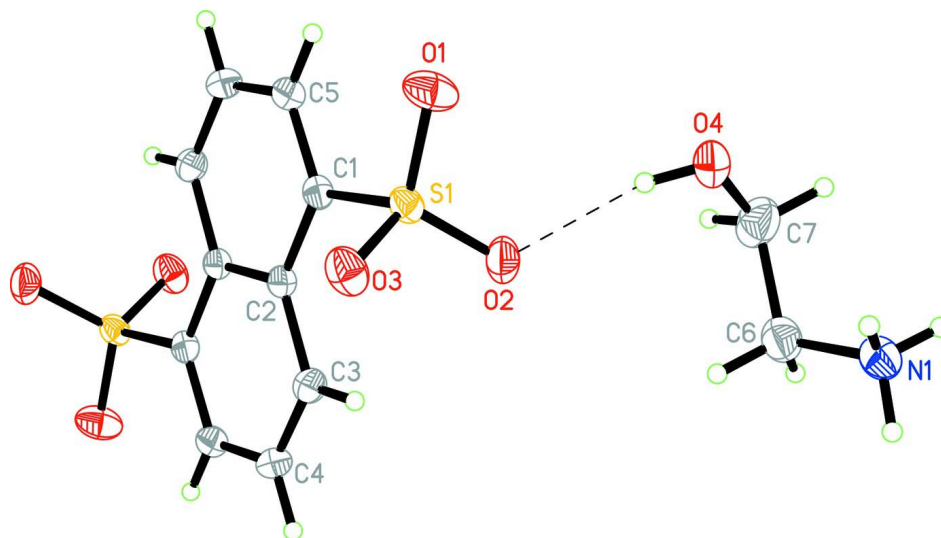
The naphthalene-1,5-disulfonate anion is linked to the ethanolaminium cation by O2—H4'···O4 hydrogen bond (Fig. 1 & Table 1). The crystal structure is further built by N—H···O and C—H···O hydrogen bonds. (Fig. 2 & Table 1). Due to steric hindrance of sulfonate, the nearest centroid separation between naphthalene rings is of 5.264 (3) Å, suggesting no π - π stacking. The arrangement of naphthalene rings is very similar to those observed in previous cases (Sakwa & Wheeler, 2003; Li & Chai, 2007; Wang *et al.*, 2008), but is different from that in bis(oxonium monohydrate) naphthalene-1,5-disulfonate, which shows a partial π - π stacking.

S2. Experimental

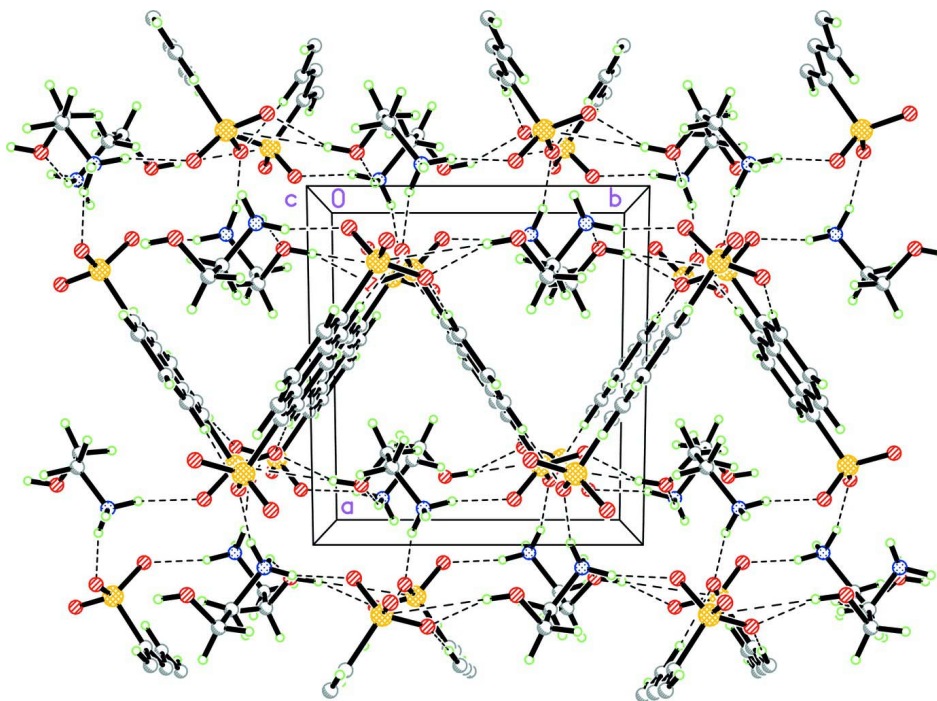
Naphthalene-1,5-disulfonic acid (5.1 g) and ethanolamine (1.2 g) in a molar ratio of 1:1 were mixed and dissolved in sufficient ethanol by heating to 373 K, at which point a clear solution resulted. The system was then cooled slowly to room temperature. Crystals of (I) (4.7 g) were formed, collected and washed with ethanol.

S3. Refinement

All H atoms except atom H4' were placed in calculated positions and allowed to ride on their parent atoms with distances of 0.93 Å for aromatic group, 0.97 Å for methylene and 0.89 Å for amino group, and with $U_{\text{iso}}(\text{H})$ set at 1.2 or 1.5 U_{eq} of the parent atoms. The positions of atom H4' were refined with a distance restraint O—H = 0.84 (2) Å, and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

**Figure 1**

The molecular structure of the title compound. The unlabeled half of naphthalene-1,5-disulfonate anion are generated from the labeled half by symmetry operation of $(1 - x, 2 - y, -z)$. The O—H \cdots O hydrogen bond is illustrated as a dashed line.

**Figure 2**

The crystal packing of the title compound, viewed down the c axis. Hydrogen bonds are drawn as dashed lines.

Bis(2-hydroxyethaniminium) naphthalene-1,5-disulfonate*Crystal data* $2\text{C}_2\text{H}_8\text{NO}^+\cdot\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2^{2-}$ $M_r = 410.48$ Monoclinic, $P2_1/c$ Hall symbol: $-P\ 2ybc$ $a = 9.7946\ (14)\ \text{\AA}$ $b = 8.9011\ (13)\ \text{\AA}$ $c = 10.4050\ (16)\ \text{\AA}$ $\beta = 104.334\ (2)^\circ$ $V = 878.9\ (2)\ \text{\AA}^3$ $Z = 2$ $F(000) = 432.0$ $D_x = 1.551\ \text{Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1567 reflections

 $\theta = 2.1\text{--}16.1^\circ$ $\mu = 0.35\ \text{mm}^{-1}$ $T = 293\ \text{K}$

Prism, colorless

 $0.42 \times 0.34 \times 0.30\ \text{mm}$ *Data collection*

Bruker APEX area-detector

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2001)

 $T_{\min} = 0.855$, $T_{\max} = 0.898$

4721 measured reflections

1718 independent reflections

1625 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.019$ $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 2.2^\circ$ $h = -12 \rightarrow 11$ $k = -7 \rightarrow 10$ $l = -11 \rightarrow 12$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.029$ $wR(F^2) = 0.080$ $S = 1.04$

1718 reflections

122 parameters

1 restraint

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

 $w = 1/[\sigma^2(F_o^2) + (0.0441P)^2 + 0.3961P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.43\ \text{e \AA}^{-3}$ $\Delta\rho_{\min} = -0.34\ \text{e \AA}^{-3}$ Extinction correction: *SHELXL97* (Sheldrick,2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.017 (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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.84543 (12)	0.76241 (16)	-0.12460 (11)	0.0408 (3)
O2	0.75476 (13)	0.65670 (12)	0.04999 (12)	0.0380 (3)
O3	0.89350 (12)	0.88364 (13)	0.09087 (11)	0.0368 (3)

O4	0.85710 (15)	0.38353 (14)	-0.03184 (13)	0.0449 (3)
H4'	0.843 (3)	0.4733 (14)	-0.015 (2)	0.067*
S1	0.79615 (4)	0.79324 (4)	-0.00698 (3)	0.02542 (15)
N1	0.91425 (15)	0.17298 (16)	0.20858 (14)	0.0359 (3)
H1A	0.9313	0.1641	0.2963	0.054*
H1B	0.9898	0.2108	0.1872	0.054*
H1C	0.8947	0.0830	0.1713	0.054*
C1	0.64066 (14)	0.90372 (15)	-0.06386 (14)	0.0232 (3)
C2	0.56077 (14)	0.95467 (15)	0.02605 (13)	0.0223 (3)
C3	0.59815 (15)	0.91921 (17)	0.16367 (14)	0.0266 (3)
H3	0.6775	0.8609	0.1983	0.032*
C4	0.51843 (16)	0.97004 (17)	0.24527 (14)	0.0292 (3)
H4	0.5440	0.9455	0.3349	0.035*
C5	0.60178 (16)	0.94103 (17)	-0.19575 (14)	0.0271 (3)
H5	0.6551	0.9074	-0.2526	0.033*
C6	0.79262 (19)	0.2745 (2)	0.16027 (18)	0.0394 (4)
H6A	0.8137	0.3716	0.2029	0.047*
H6B	0.7112	0.2338	0.1856	0.047*
C7	0.7569 (2)	0.2955 (2)	0.01220 (19)	0.0490 (5)
H7A	0.7502	0.1977	-0.0300	0.059*
H7B	0.6653	0.3433	-0.0157	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0307 (6)	0.0605 (8)	0.0332 (6)	0.0100 (5)	0.0116 (5)	-0.0071 (6)
O2	0.0458 (7)	0.0233 (6)	0.0464 (7)	0.0058 (5)	0.0145 (5)	0.0026 (5)
O3	0.0315 (6)	0.0343 (6)	0.0396 (6)	0.0016 (5)	-0.0006 (5)	-0.0078 (5)
O4	0.0649 (8)	0.0265 (6)	0.0475 (7)	0.0058 (6)	0.0219 (6)	0.0020 (5)
S1	0.0249 (2)	0.0249 (2)	0.0263 (2)	0.00367 (13)	0.00597 (15)	-0.00329 (13)
N1	0.0386 (7)	0.0330 (7)	0.0368 (7)	-0.0016 (6)	0.0102 (6)	-0.0055 (6)
C1	0.0238 (7)	0.0200 (7)	0.0262 (7)	-0.0003 (5)	0.0070 (5)	-0.0013 (5)
C2	0.0246 (7)	0.0194 (6)	0.0233 (7)	-0.0017 (5)	0.0063 (5)	-0.0007 (5)
C3	0.0274 (7)	0.0253 (7)	0.0261 (7)	0.0033 (6)	0.0050 (6)	0.0025 (6)
C4	0.0361 (8)	0.0305 (8)	0.0210 (7)	0.0014 (6)	0.0068 (6)	0.0026 (6)
C5	0.0325 (8)	0.0252 (7)	0.0256 (7)	0.0004 (6)	0.0109 (6)	-0.0017 (6)
C6	0.0420 (9)	0.0340 (9)	0.0454 (9)	0.0024 (7)	0.0168 (8)	-0.0010 (7)
C7	0.0594 (12)	0.0359 (10)	0.0457 (10)	-0.0083 (8)	0.0016 (9)	0.0037 (8)

Geometric parameters (Å, °)

O1—S1	1.4485 (11)	C2—C2 ⁱ	1.429 (3)
O2—S1	1.4533 (12)	C3—C4	1.365 (2)
O3—S1	1.4535 (11)	C3—H3	0.9300
O4—C7	1.417 (2)	C4—C5 ⁱ	1.406 (2)
O4—H4'	0.835 (10)	C4—H4	0.9300
S1—C1	1.7862 (14)	C5—C4 ⁱ	1.406 (2)
N1—C6	1.481 (2)	C5—H5	0.9300

N1—H1A	0.8900	C6—C7	1.505 (3)
N1—H1B	0.8900	C6—H6A	0.9700
N1—H1C	0.8900	C6—H6B	0.9700
C1—C5	1.371 (2)	C7—H7A	0.9700
C1—C2	1.4338 (19)	C7—H7B	0.9700
C2—C3	1.423 (2)		
C7—O4—H4'	107.6 (18)	C4—C3—H3	119.7
O1—S1—O2	111.79 (8)	C2—C3—H3	119.7
O1—S1—O3	113.50 (7)	C3—C4—C5 ⁱ	120.96 (13)
O2—S1—O3	112.22 (7)	C3—C4—H4	119.5
O1—S1—C1	105.04 (7)	C5 ⁱ —C4—H4	119.5
O2—S1—C1	107.12 (7)	C1—C5—C4 ⁱ	120.20 (13)
O3—S1—C1	106.56 (7)	C1—C5—H5	119.9
C6—N1—H1A	109.5	C4 ⁱ —C5—H5	119.9
C6—N1—H1B	109.5	N1—C6—C7	112.78 (15)
H1A—N1—H1B	109.5	N1—C6—H6A	109.0
C6—N1—H1C	109.5	C7—C6—H6A	109.0
H1A—N1—H1C	109.5	N1—C6—H6B	109.0
H1B—N1—H1C	109.5	C7—C6—H6B	109.0
C5—C1—C2	121.01 (13)	H6A—C6—H6B	107.8
C5—C1—S1	117.89 (11)	O4—C7—C6	113.31 (16)
C2—C1—S1	121.08 (10)	O4—C7—H7A	108.9
C3—C2—C2 ⁱ	119.32 (15)	C6—C7—H7A	108.9
C3—C2—C1	122.72 (13)	O4—C7—H7B	108.9
C2 ⁱ —C2—C1	117.97 (15)	C6—C7—H7B	108.9
C4—C3—C2	120.54 (13)	H7A—C7—H7B	107.7

Symmetry code: (i) $-x+1, -y+2, -z$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H4' \cdots O2	0.84 (1)	2.04 (1)	2.840 (2)	160 (2)
N1—H1A \cdots O4 ⁱⁱ	0.89	2.13	2.934 (2)	149
N1—H1B \cdots O1 ⁱⁱⁱ	0.89	1.90	2.766 (2)	164
N1—H1C \cdots O3 ^{iv}	0.89	1.96	2.837 (2)	168
C3—H3 \cdots O1 ^v	0.93	2.41	3.270 (2)	154
C5—H5 \cdots O2 ^{vi}	0.93	2.55	3.459 (2)	167

Symmetry codes: (ii) $x, -y+1/2, z+1/2$; (iii) $-x+2, -y+1, -z$; (iv) $x, y-1, z$; (v) $x, -y+3/2, z+1/2$; (vi) $x, -y+3/2, z-1/2$.