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1-Ammonionaphthalene-2-sulfonate

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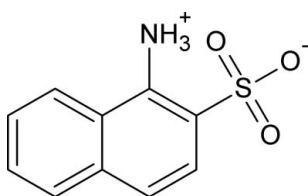
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.038; wR factor = 0.107; data-to-parameter ratio = 16.0.

In the molecule of the zwitterionic title compound, $\text{C}_{10}\text{H}_9\text{NO}_3\text{S}$, an intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond results in the formation of an almost planar six-membered ring (r.m.s deviation = 0.0150 Å), which is oriented at a dihedral angle of $1.63(3)^\circ$ with respect to the naphthalene ring system. In the crystal structure, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into a two-dimensional network.

Related literature

For general background to the use of amino naphthalene sulfonic acid derivatives as intermediates for the syntheses of azo dyes, see: O'Neil (2001). For related structures, see: Arshad *et al.* (2008a,b); Genter *et al.* (2007); Shafiq *et al.* (2008); Smith *et al.* (2004, 2009). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

 $\text{C}_{10}\text{H}_9\text{NO}_3\text{S}$ $M_r = 223.24$ Orthorhombic, *Pbca* $a = 9.4337(3)$ Å $b = 10.6359(4)$ Å $c = 18.6775(6)$ Å $V = 1874.02(11)$ Å³ $Z = 8$ Mo $K\alpha$ radiation $\mu = 0.33$ mm⁻¹ $T = 296$ K $0.29 \times 0.21 \times 0.18$ mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2007)

 $T_{\min} = 0.911$, $T_{\max} = 0.943$

10742 measured reflections

2326 independent reflections

1763 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.035$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.038$ $wR(F^2) = 0.107$ $S = 1.03$

2326 reflections

145 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.50$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^{\ddagger}$	0.86 (2)	1.94 (2)	2.762 (2)	160.4 (18)
$\text{N1}-\text{H2N}\cdots\text{O2}$	0.91 (2)	1.83 (2)	2.651 (2)	149.0 (19)
$\text{N1}-\text{H3N}\cdots\text{O3}^{\ddagger}$	0.87 (3)	2.14 (3)	2.982 (2)	162 (2)

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

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2764).

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supplementary materials

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1-Ammonionaphthalene-2-sulfonate

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Comment

The title compound is a zwitterion of 1-amino-2-naphthalene sulfonic acid (*o*-naphthionic acid). Amino naphthalene sulfonic acid derivatives have been used as an intermediate for the syntheses of azo dyes (O'Neil, 2001) and gained importance in complexation (Genther *et al.*, 2007). We purchased 1-amino-2-naphthalene sulfonic acid to use as a precursor for the syntheses of biologically active thiazine related heterocycles (Arshad *et al.*, 2008*a, b*; Shafiq *et al.*, 2008). The crystal structures of 5-aminonaphthalene-1-sulfonic acid (Genther *et al.*, 2007; Smith *et al.*, 2004) and 8-ammonionaphthalene-2-sulfonate monohydrate (Smith *et al.*, 2009) have already been published, which are position isomers of the title compound.

In the molecule of the title compound, (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (C1-C4/C9/C10) and B (C5-C10) are, of course, planar. The dihedral angles between them are A/B = 1.94 (3)°. The intramolecular N-H...O hydrogen bond (Table 1) results in the formation of a planar six-membered ring C (S1/O2/N1/C1/C2/H2N), which is oriented with respect to the other rings at dihedral angles of A/C = 0.74 (3) and B/C = 2.59 (3)°. So, the rings are almost coplanar.

In the crystal structure, intermolecular N-H...O hydrogen bonds (Table 1) link the molecules into a two-dimensional network (Fig. 2), in which they may be effective in the stabilization of the structure.

Experimental

The title compound was purchased from Sigma-Aldrich and recrystallized in methanol for X-ray analysis.

Refinement

Atoms H1N, H2N and H3N (for NH₃) are located in a difference Fourier map and constrained to ride on their parent atom, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. The remaining H atoms were positioned geometrically with C-H = 0.93 Å for aromatic H atoms and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

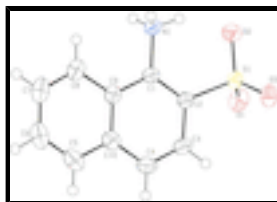


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

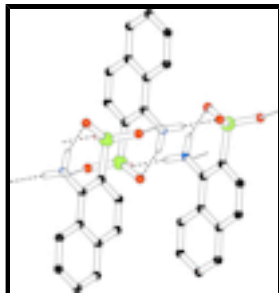


Fig. 2. A partial packing diagram. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

1-Ammonionaphthalene-2-sulfonate

Crystal data

$C_{10}H_9NO_3S$

$M_r = 223.24$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 9.4337$ (3) Å

$b = 10.6359$ (4) Å

$c = 18.6775$ (6) Å

$V = 1874.02$ (11) Å³

$Z = 8$

$F_{000} = 928$

$D_x = 1.582$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2779 reflections

$\theta = 3.1$ – 27.5°

$\mu = 0.33$ mm⁻¹

$T = 296$ K

Hexagonal, dark brown

$0.29 \times 0.21 \times 0.18$ mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 296$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2007)

$T_{\min} = 0.911$, $T_{\max} = 0.943$

10742 measured reflections

2326 independent reflections

1763 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 28.3^\circ$

$\theta_{\min} = 3.1^\circ$

$h = -12 \rightarrow 12$

$k = -14 \rightarrow 8$

$l = -24 \rightarrow 24$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.107$

$S = 1.03$

2326 reflections

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

$$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.6846P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.30$ e Å⁻³

145 parameters

$$\Delta\rho_{\min} = -0.50 \text{ e } \text{\AA}^{-3}$$

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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	0.48056 (5)	0.72906 (4)	1.02833 (2)	0.02659 (15)
O1	0.62861 (15)	0.69891 (13)	1.04059 (8)	0.0375 (4)
O2	0.38420 (16)	0.65642 (14)	1.07238 (7)	0.0401 (4)
O3	0.45788 (15)	0.86350 (13)	1.03188 (7)	0.0369 (3)
N1	0.25407 (18)	0.53879 (16)	0.96588 (9)	0.0282 (3)
H1N	0.270 (2)	0.459 (2)	0.9642 (10)	0.034*
H2N	0.273 (2)	0.565 (2)	1.0112 (12)	0.034*
H3N	0.166 (3)	0.558 (2)	0.9579 (11)	0.034*
C1	0.34654 (18)	0.60250 (16)	0.91466 (9)	0.0248 (4)
C2	0.44755 (18)	0.68602 (17)	0.93750 (9)	0.0253 (4)
C3	0.5354 (2)	0.7463 (2)	0.88661 (11)	0.0346 (4)
H3	0.6054	0.8017	0.9018	0.041*
C4	0.5178 (2)	0.7236 (2)	0.81550 (11)	0.0373 (5)
H4	0.5756	0.7646	0.7826	0.045*
C5	0.3947 (2)	0.6159 (2)	0.71689 (11)	0.0411 (5)
H5	0.4495	0.6590	0.6836	0.049*
C6	0.2971 (3)	0.5317 (2)	0.69409 (12)	0.0455 (6)
H6	0.2852	0.5175	0.6453	0.055*
C7	0.2144 (3)	0.4659 (2)	0.74345 (12)	0.0457 (5)
H7	0.1486	0.4074	0.7272	0.055*
C8	0.2287 (2)	0.48638 (19)	0.81515 (11)	0.0373 (5)
H8	0.1727	0.4418	0.8473	0.045*
C9	0.32805 (19)	0.57488 (17)	0.84084 (10)	0.0277 (4)
C10	0.4142 (2)	0.63928 (18)	0.79073 (10)	0.0317 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0286 (2)	0.0233 (2)	0.0278 (2)	-0.00027 (18)	-0.00188 (17)	0.00027 (17)

supplementary materials

O1	0.0327 (7)	0.0331 (7)	0.0467 (8)	0.0037 (6)	-0.0106 (6)	-0.0016 (6)
O2	0.0467 (9)	0.0439 (8)	0.0297 (7)	-0.0125 (7)	0.0029 (6)	0.0025 (6)
O3	0.0451 (8)	0.0254 (7)	0.0402 (8)	0.0056 (6)	-0.0033 (6)	-0.0043 (6)
N1	0.0271 (8)	0.0231 (8)	0.0343 (9)	-0.0033 (7)	0.0033 (7)	0.0014 (7)
C1	0.0224 (8)	0.0216 (8)	0.0303 (9)	0.0027 (7)	0.0034 (7)	0.0015 (7)
C2	0.0252 (8)	0.0231 (8)	0.0277 (9)	0.0012 (7)	0.0008 (7)	0.0000 (7)
C3	0.0301 (9)	0.0379 (11)	0.0357 (10)	-0.0108 (8)	0.0024 (8)	0.0019 (8)
C4	0.0364 (10)	0.0415 (12)	0.0339 (10)	-0.0053 (9)	0.0087 (8)	0.0057 (9)
C5	0.0490 (12)	0.0454 (12)	0.0290 (10)	0.0119 (11)	0.0036 (9)	0.0006 (9)
C6	0.0558 (13)	0.0454 (13)	0.0353 (11)	0.0151 (11)	-0.0103 (10)	-0.0111 (9)
C7	0.0502 (13)	0.0377 (12)	0.0493 (13)	0.0010 (10)	-0.0143 (10)	-0.0119 (10)
C8	0.0360 (11)	0.0323 (10)	0.0435 (11)	-0.0026 (9)	-0.0048 (8)	-0.0044 (9)
C9	0.0259 (8)	0.0244 (9)	0.0328 (10)	0.0051 (7)	-0.0016 (7)	-0.0023 (7)
C10	0.0322 (9)	0.0323 (10)	0.0305 (10)	0.0055 (8)	0.0005 (7)	0.0004 (8)

Geometric parameters (Å, °)

S1—O3	1.4473 (14)	C4—C10	1.405 (3)
S1—O2	1.4491 (14)	C4—H4	0.9300
S1—O1	1.4513 (14)	C5—C6	1.353 (3)
S1—C2	1.7845 (18)	C5—C10	1.413 (3)
N1—C1	1.461 (2)	C5—H5	0.9300
N1—H1N	0.86 (2)	C6—C7	1.395 (3)
N1—H2N	0.91 (2)	C6—H6	0.9300
N1—H3N	0.86 (2)	C7—C8	1.363 (3)
C1—C2	1.371 (2)	C7—H7	0.9300
C1—C9	1.420 (2)	C8—C9	1.412 (3)
C2—C3	1.415 (3)	C8—H8	0.9300
C3—C4	1.360 (3)	C9—C10	1.416 (3)
C3—H3	0.9300		
O3—S1—O2	114.07 (9)	C3—C4—C10	121.32 (18)
O3—S1—O1	110.69 (9)	C3—C4—H4	119.3
O2—S1—O1	113.34 (9)	C10—C4—H4	119.3
O3—S1—C2	105.74 (8)	C6—C5—C10	120.8 (2)
O2—S1—C2	107.07 (8)	C6—C5—H5	119.6
O1—S1—C2	105.15 (8)	C10—C5—H5	119.6
C1—N1—H1N	109.3 (14)	C5—C6—C7	120.3 (2)
C1—N1—H2N	110.2 (14)	C5—C6—H6	119.9
H1N—N1—H2N	107.7 (19)	C7—C6—H6	119.9
C1—N1—H3N	110.5 (14)	C8—C7—C6	120.9 (2)
H1N—N1—H3N	113 (2)	C8—C7—H7	119.5
H2N—N1—H3N	106.3 (19)	C6—C7—H7	119.5
C2—C1—C9	121.43 (16)	C7—C8—C9	120.4 (2)
C2—C1—N1	120.77 (16)	C7—C8—H8	119.8
C9—C1—N1	117.79 (16)	C9—C8—H8	119.8
C1—C2—C3	119.45 (17)	C8—C9—C10	118.59 (17)
C1—C2—S1	125.70 (14)	C8—C9—C1	123.31 (18)
C3—C2—S1	114.85 (14)	C10—C9—C1	118.10 (16)
C4—C3—C2	120.28 (18)	C4—C10—C5	121.61 (19)

C4—C3—H3	119.9	C4—C10—C9	119.36 (17)
C2—C3—H3	119.9	C5—C10—C9	119.03 (19)
C9—C1—C2—C3	0.1 (3)	C6—C7—C8—C9	0.0 (3)
N1—C1—C2—C3	-179.95 (17)	C7—C8—C9—C10	-1.4 (3)
C9—C1—C2—S1	179.69 (14)	C7—C8—C9—C1	178.78 (19)
N1—C1—C2—S1	-0.3 (3)	C2—C1—C9—C8	177.77 (18)
O3—S1—C2—C1	-120.22 (16)	N1—C1—C9—C8	-2.2 (3)
O2—S1—C2—C1	1.78 (19)	C2—C1—C9—C10	-2.0 (3)
O1—S1—C2—C1	122.61 (16)	N1—C1—C9—C10	178.00 (16)
O3—S1—C2—C3	59.42 (16)	C3—C4—C10—C5	179.5 (2)
O2—S1—C2—C3	-178.58 (14)	C3—C4—C10—C9	-1.2 (3)
O1—S1—C2—C3	-57.75 (16)	C6—C5—C10—C4	178.0 (2)
C1—C2—C3—C4	1.4 (3)	C6—C5—C10—C9	-1.2 (3)
S1—C2—C3—C4	-178.30 (17)	C8—C9—C10—C4	-177.23 (18)
C2—C3—C4—C10	-0.8 (3)	C1—C9—C10—C4	2.6 (3)
C10—C5—C6—C7	-0.3 (3)	C8—C9—C10—C5	2.0 (3)
C5—C6—C7—C8	0.9 (3)	C1—C9—C10—C5	-178.18 (17)

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots O1 ⁱ	0.86 (2)	1.94 (2)	2.762 (2)	160.4 (18)
N1—H2N \cdots O2	0.91 (2)	1.83 (2)	2.651 (2)	149.0 (19)
N1—H3N \cdots O3 ⁱⁱ	0.87 (3)	2.14 (3)	2.982 (2)	162 (2)

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

Fig. 1

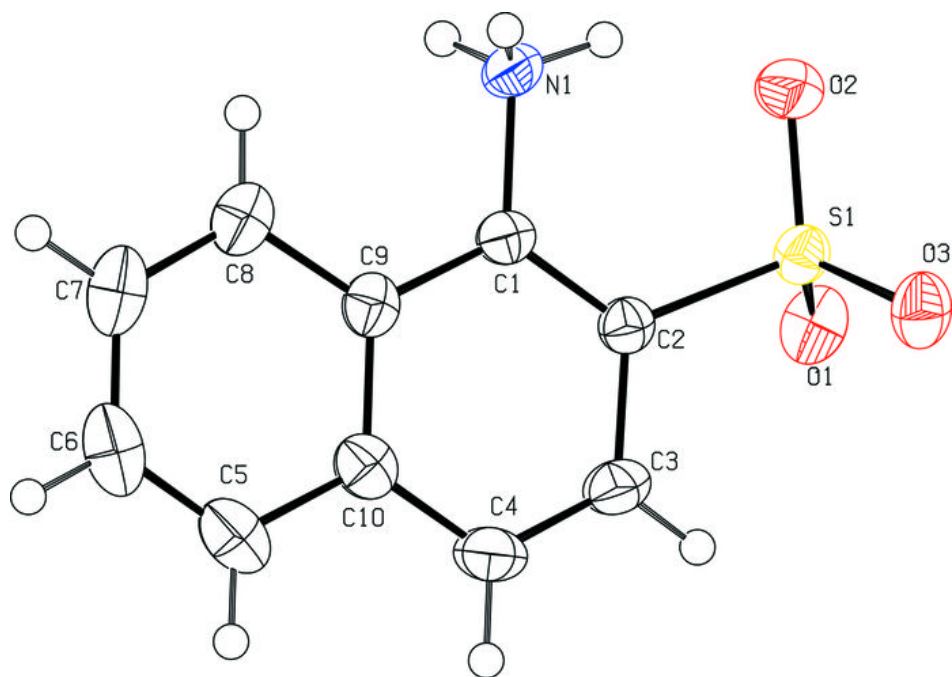


Fig. 2

