

4-Amino-3-ammoniobenzenesulfonate

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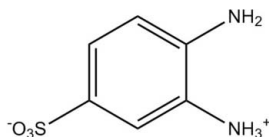
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.031; wR factor = 0.087; data-to-parameter ratio = 12.2.

The title compound, $\text{C}_6\text{H}_8\text{N}_2\text{O}_3\text{S}$, crystallized as a sulfonate–ammonium zwitterion. In the crystal, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds generate an extensive three-dimensional network, which consolidates the packing.

Related literature

For the crystal structures of isomers of the title compound, see: Rubin-Preminger & Bernstein (2003). For details of the synthesis, see: Miranda *et al.* (2008).



Experimental

Crystal data

 $\text{C}_6\text{H}_8\text{N}_2\text{O}_3\text{S}$
 $M_r = 188.20$

 Monoclinic, $P2_1/c$
 $a = 5.602$ (1) Å

 $b = 8.4135$ (15) Å

 $c = 16.221$ (3) Å

 $\beta = 95.613$ (2)°

 $V = 760.9$ (2) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.39$ mm⁻¹
 $T = 295$ K

 $0.35 \times 0.25 \times 0.15$ mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.875$, $T_{\max} = 0.944$

4039 measured reflections
 1490 independent reflections
 1351 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.087$
 $S = 1.09$
 1490 reflections
 122 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1B}\cdots\text{O1}^{\text{i}}$	0.92 (3)	1.86 (3)	2.778 (2)	178 (2)
$\text{N1}-\text{H1C}\cdots\text{O1}^{\text{ii}}$	0.92 (3)	1.89 (3)	2.792 (2)	165 (2)
$\text{N1}-\text{H1A}\cdots\text{O2}^{\text{iii}}$	0.90 (2)	1.88 (3)	2.759 (2)	165 (2)
$\text{N2}-\text{H2B}\cdots\text{O3}^{\text{iv}}$	0.86	2.46	3.047 (2)	126

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2799).

References

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supporting information

Acta Cryst. (2010). E66, o3281 [https://doi.org/10.1107/S1600536810048063]

4-Amino-3-ammoniobenzenesulfonate

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S1. Comment

The title compound (I) (Fig. 1) is a zwitterion of 4-amino-3-ammoniobenzenesulfonate. The bond lengths and angles in (I) are normal and comparable with those observed in the related compounds (Rubin-Preminger & Bernstein, 2003). In the crystal structure, intermolecular N—H···O hydrogen bonds generate an extensive three-dimensional network which consolidate the crystal packing.

S2. Experimental

The title compound was synthesized according to the method reported in the literature (Miranda *et al.*, 2008). Orange single crystals suitable for X-ray diffraction were obtained by slow evaporation of a water solution of the compound.

S3. Refinement

C-bound H atoms and *N*(amino)-bound H atoms were geometrically positioned (C—H = 0.93 Å, N—H = 0.86 Å) and included in the riding model approximation, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C}, \text{N})$. H atoms attached to *N*(ammonio) were located from an electron density map, and isotropically refined with the N—H bond length restrained to 0.91 (3) Å.

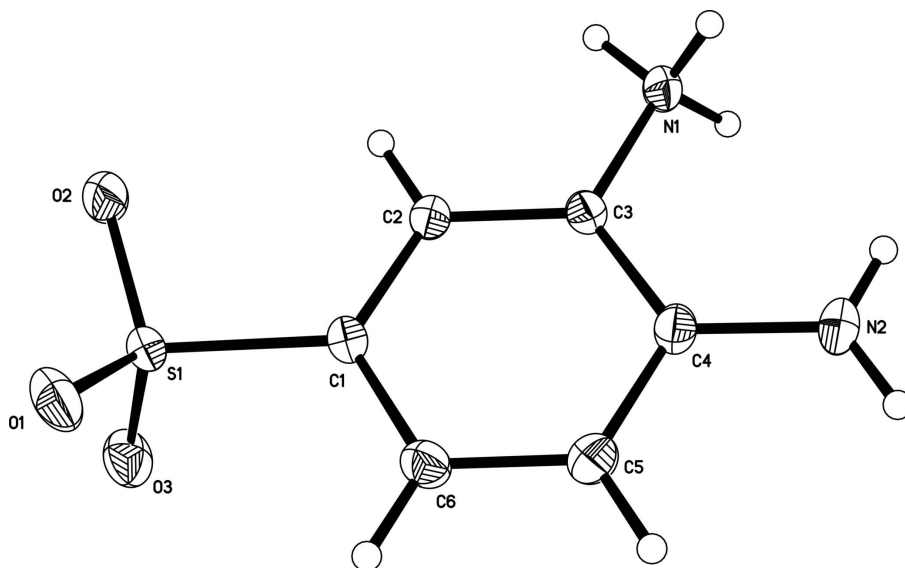


Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

4-Amino-3-ammoniobenzenesulfonate

Crystal data

C₆H₈N₂O₃S $M_r = 188.20$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 5.602 (1) \text{ \AA}$ $b = 8.4135 (15) \text{ \AA}$ $c = 16.221 (3) \text{ \AA}$ $\beta = 95.613 (2)^\circ$ $V = 760.9 (2) \text{ \AA}^3$ $Z = 4$ $F(000) = 392$ $D_x = 1.643 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2332 reflections

 $\theta = 2.4\text{--}27.7^\circ$ $\mu = 0.39 \text{ mm}^{-1}$ $T = 295 \text{ K}$

Block, orange

 $0.35 \times 0.25 \times 0.15 \text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ϕ and ω scansAbsorption correction: multi-scan
(SADABS; Bruker, 2005) $T_{\min} = 0.875$, $T_{\max} = 0.944$

4039 measured reflections

1490 independent reflections

1351 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.019$ $\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 2.5^\circ$ $h = -6 \rightarrow 6$ $k = -9 \rightarrow 10$ $l = -17 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.031$ $wR(F^2) = 0.087$ $S = 1.09$

1490 reflections

122 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.3348P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.34 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.33 \text{ e \AA}^{-3}$ Extinction correction: *SHELXTL* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.022 (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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1329 (3)	0.2272 (2)	1.04906 (10)	0.0265 (4)
C2	0.1963 (3)	0.15996 (19)	0.97641 (10)	0.0263 (4)
H2	0.3348	0.0988	0.9769	0.032*

C3	0.0532 (3)	0.18407 (19)	0.90339 (10)	0.0263 (4)
C4	-0.1567 (3)	0.2754 (2)	0.90017 (10)	0.0300 (4)
C5	-0.2178 (3)	0.3403 (2)	0.97466 (11)	0.0341 (4)
H5	-0.3570	0.4005	0.9747	0.041*
C6	-0.0764 (3)	0.3168 (2)	1.04811 (11)	0.0314 (4)
H6	-0.1207	0.3608	1.0970	0.038*
H1A	0.266 (4)	0.065 (3)	0.8377 (14)	0.052 (7)*
H1B	0.132 (4)	0.185 (3)	0.7858 (16)	0.045 (6)*
H1C	0.012 (4)	0.033 (3)	0.8111 (15)	0.055 (7)*
N1	0.1219 (3)	0.1112 (2)	0.82725 (9)	0.0304 (3)
N2	-0.2985 (3)	0.3022 (2)	0.82732 (11)	0.0502 (5)
H2B	-0.4260	0.3592	0.8274	0.060*
H2A	-0.2597	0.2619	0.7818	0.060*
O1	0.1572 (3)	0.16078 (16)	1.20518 (8)	0.0413 (4)
O2	0.4779 (3)	0.07161 (18)	1.12735 (8)	0.0518 (4)
O3	0.4413 (2)	0.34942 (17)	1.16250 (8)	0.0424 (4)
S1	0.31916 (8)	0.20155 (5)	1.14202 (2)	0.02874 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0281 (9)	0.0290 (8)	0.0218 (8)	0.0004 (6)	-0.0011 (6)	-0.0005 (6)
C2	0.0269 (8)	0.0274 (8)	0.0243 (8)	0.0028 (6)	0.0004 (6)	0.0009 (6)
C3	0.0302 (9)	0.0259 (8)	0.0222 (8)	0.0004 (6)	0.0005 (6)	-0.0006 (6)
C4	0.0303 (9)	0.0298 (8)	0.0285 (9)	0.0012 (7)	-0.0045 (7)	0.0012 (7)
C5	0.0277 (9)	0.0369 (9)	0.0368 (10)	0.0073 (7)	-0.0004 (7)	-0.0027 (7)
C6	0.0309 (9)	0.0363 (9)	0.0275 (9)	0.0036 (7)	0.0045 (7)	-0.0054 (7)
N1	0.0352 (9)	0.0334 (8)	0.0217 (7)	0.0054 (7)	-0.0010 (6)	-0.0001 (6)
N2	0.0538 (11)	0.0559 (11)	0.0364 (9)	0.0241 (9)	-0.0188 (8)	-0.0073 (8)
O1	0.0556 (9)	0.0457 (8)	0.0228 (6)	-0.0086 (6)	0.0054 (6)	-0.0029 (5)
O2	0.0625 (9)	0.0574 (9)	0.0324 (7)	0.0326 (8)	-0.0104 (6)	-0.0080 (6)
O3	0.0452 (8)	0.0432 (8)	0.0366 (7)	-0.0086 (6)	-0.0072 (6)	-0.0032 (6)
S1	0.0343 (3)	0.0307 (3)	0.0203 (2)	0.00447 (16)	-0.00195 (17)	-0.00273 (15)

Geometric parameters (Å, °)

C1—C2	1.385 (2)	C5—H5	0.9300
C1—C6	1.393 (2)	C6—H6	0.9300
C1—S1	1.7611 (17)	N1—H1A	0.90 (2)
C2—C3	1.379 (2)	N1—H1B	0.92 (3)
C2—H2	0.9300	N1—H1C	0.92 (3)
C3—C4	1.401 (2)	N2—H2B	0.8600
C3—N1	1.464 (2)	N2—H2A	0.8600
C4—N2	1.376 (2)	O1—S1	1.4739 (14)
C4—C5	1.399 (2)	O2—S1	1.4435 (14)
C5—C6	1.379 (2)	O3—S1	1.4428 (14)
C2—C1—C6	119.85 (15)	C1—C6—H6	120.0

C2—C1—S1	119.92 (13)	C3—N1—H1A	109.1 (15)
C6—C1—S1	120.23 (13)	C3—N1—H1B	111.8 (14)
C3—C2—C1	119.66 (15)	H1A—N1—H1B	108 (2)
C3—C2—H2	120.2	C3—N1—H1C	108.5 (14)
C1—C2—H2	120.2	H1A—N1—H1C	108 (2)
C2—C3—C4	121.88 (15)	H1B—N1—H1C	111 (2)
C2—C3—N1	118.96 (15)	C4—N2—H2B	120.0
C4—C3—N1	119.16 (15)	C4—N2—H2A	120.0
N2—C4—C5	120.55 (16)	H2B—N2—H2A	120.0
N2—C4—C3	122.25 (16)	O3—S1—O2	113.91 (10)
C5—C4—C3	117.20 (15)	O3—S1—O1	110.55 (8)
C6—C5—C4	121.48 (16)	O2—S1—O1	111.67 (9)
C6—C5—H5	119.3	O3—S1—C1	108.61 (8)
C4—C5—H5	119.3	O2—S1—C1	105.94 (8)
C5—C6—C1	119.93 (16)	O1—S1—C1	105.68 (8)
C5—C6—H6	120.0		
C6—C1—C2—C3	0.9 (3)	C4—C5—C6—C1	0.2 (3)
S1—C1—C2—C3	-178.25 (13)	C2—C1—C6—C5	-1.0 (3)
C1—C2—C3—C4	0.0 (3)	S1—C1—C6—C5	178.15 (14)
C1—C2—C3—N1	-179.58 (15)	C2—C1—S1—O3	106.55 (15)
C2—C3—C4—N2	178.93 (17)	C6—C1—S1—O3	-72.59 (16)
N1—C3—C4—N2	-1.5 (3)	C2—C1—S1—O2	-16.19 (17)
C2—C3—C4—C5	-0.8 (3)	C6—C1—S1—O2	164.66 (15)
N1—C3—C4—C5	178.80 (16)	C2—C1—S1—O1	-134.81 (14)
N2—C4—C5—C6	-179.04 (18)	C6—C1—S1—O1	46.04 (17)
C3—C4—C5—C6	0.7 (3)		

Hydrogen-bond geometry (Å, °)

<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—H1B \cdots O1 ⁱ	0.92 (3)	1.86 (3)	2.778 (2)	178 (2)
N1—H1C \cdots O1 ⁱⁱ	0.92 (3)	1.89 (3)	2.792 (2)	165 (2)
N1—H1A \cdots O2 ⁱⁱⁱ	0.90 (2)	1.88 (3)	2.759 (2)	165 (2)
N2—H2B \cdots O3 ^{iv}	0.86	2.46	3.047 (2)	126

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