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Diammonium biphenyl-4,4′-disulfonate

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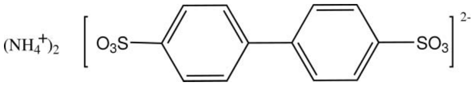
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.006 Å; R factor = 0.045; wR factor = 0.172; data-to-parameter ratio = 16.5.

In the title salt, $2NH_4^+\cdot C_{12}H_8O_6S_2^{2-}$, the dianion has crystallographic inversion symmetry. A three-dimensional framework is formed from primary hydrogen-bonded sheet structures comprising ammonium N—H⋯$O_{sulfonate}$ interactions and is linked peripherally through the biphenyl residues of the anions. This open framework has 43 Å^3 solvent-accessible voids.

Related literature

Biphenyl-4,4′-disulfonate clathrate structures may be found in: Russell *et al.* (1997); Swift, Pivovar *et al.* (1998); Swift, Reynolds & Ward (1998); Swift & Ward (2000); Pivovar *et al.* (2001).



Experimental

Crystal data

$2NH_4^+\cdot C_{12}H_8O_6S_2^{2-}$
M_r = 348.39
Monoclinic, $P2_1/c$
a = 14.778 (2) Å
b = 7.4138 (12) Å
c = 7.6647 (13) Å
β = 96.667 (13)°
V = 834.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 297 (2) K
0.40 × 0.30 × 0.11 mm

Data collection

Rigaku AFC 7R four-circle diffractometer
Absorption correction: ψ scan (*TEXSAN for Windows*; Molecular Structure Corporation, 1999)
T_{min} = 0.874, T_{max} = 0.963
2129 measured reflections
1909 independent reflections
1030 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
3 standard reflections frequency: 150 min intensity decay: 1.6%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.172
S = 0.86
1909 reflections
116 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 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
N1—H11⋯O11^{i}	0.91 (5)	1.96 (4)	2.861 (5)	172 (4)
N1—H12⋯O11^{ii}	0.80 (5)	2.18 (5)	2.922 (5)	153 (5)
N1—H12⋯O13^{iii}	0.80 (5)	2.45 (5)	2.955 (4)	122 (4)
N1—H13⋯O13	0.91 (6)	1.96 (6)	2.841 (5)	162 (4)
N1—H14⋯O12^{iv}	0.88 (5)	1.97 (5)	2.848 (5)	174 (4)
C2—H2⋯O11	0.95	2.48	2.873 (6)	105
C6—H6⋯O12^{v}	0.95	2.33	3.243 (6)	161

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

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2396).

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Graham Smith, Urs D. Wermuth and Peter C. Healy

S1. Comment

The guanidinium salts of biphenyl-4,4′-disulfonic acid ($BPDSH_2$) form two-dimensional hydrogen-bonded open-framework structures in which the guanidinium cations form sheets connected by BPDS pillars. (Russell *et al.*, 1997). These structures may accommodate various molecular guest species, commonly aromatic hydrocarbons, giving crystalline clathrates of the type $(Gu^+)_2$ $BPDS^{2-}$. *n*G (where G = the guest species) (Swift, Pivovar *et al.*, 1998; Swift, Reynolds & Ward, 1998; Swift & Ward, 2000; Pivovar *et al.*, 2001). Because it was considered that the ammonium salt of $BPDSH_2$ might also have an open framework structure, we prepared crystals of anhydrous $(NH_4^+)_2$ $C_{12}H_8O_6S_2^{2-}$ (I) from an aqueous ammoniacal solution of the acid and the structure is reported here.

In (I), the planar anions have inversion symmetry coincident with crystallographic symmetry (Fig. 1). Each ammonium cation gives a total of five associations with sulfonate-O acceptors of the cation (Table 1) resulting in sheet structures which extend across the *bc* planes in the unit cell at *a* = 0. These sheets are linked across the *a* cell direction through the biphenyl residues of the BPDS anions, giving a three- dimensional framework structure (Fig. 2). There are 43 Å^3 solvent accessible voids within the structure.

S2. Experimental

Compound (I) was prepared by the room temperature interaction in a 2:1 stoichiometric ratio of ammonia as an aqueous solution with biphenyl-4,4′-disulfonic acid. Colourless crystal plates (m. p. >573 K) were obtained from the partial room temperature evaporation of this solution.

S3. Refinement

The ammonium hydrogen atoms were located by difference methods and their positional and isotropic displacement parameters were refined. The aromatic H atoms were included in the refinement in calculated positions (C–H = 0.95 Å) using a riding model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

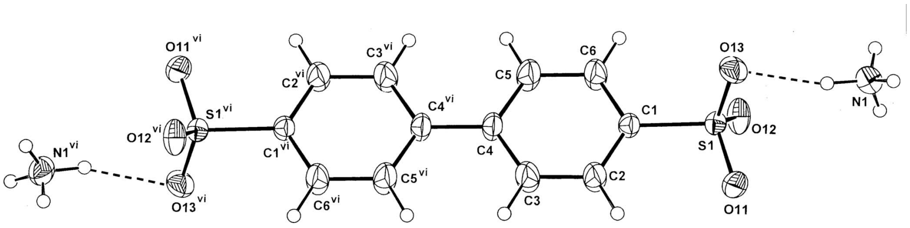

Figure 1

Molecular configuration and atom naming scheme for the BPDS anion in (I). Atoms of the inversion-related half of the compound are indicated by symmetry code (vi) (-*x* + 1, -*y*, -*z* + 1). The dashed lines represent the hydrogen bonds between the ammonium protons and the sulfonate-O acceptors.

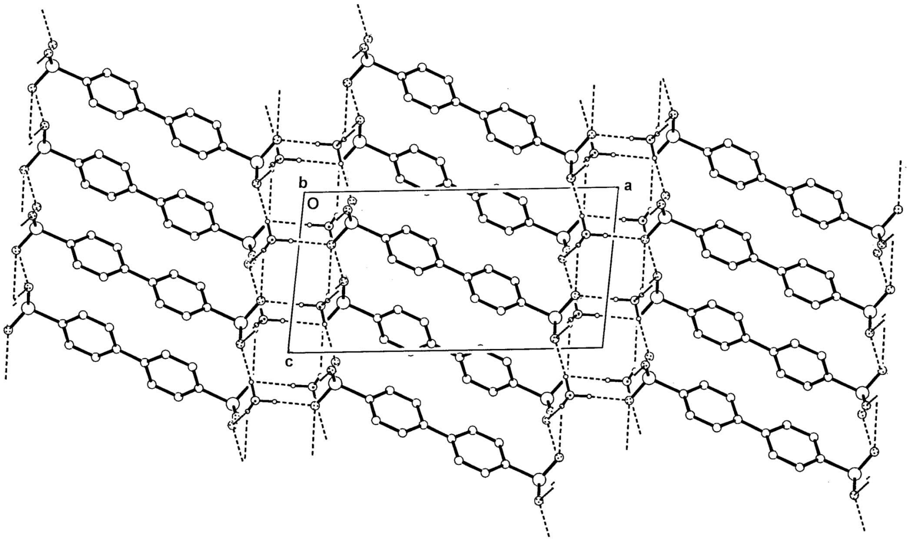

Figure 2

A perspective view of the three-dimensional hydrogen-bonded framework structure of (I) with ammonium *N*–*H*–$O_{sulfonate}$ sheets interlinked by the biphenyl residues of the BPDS anions.

Diammonium biphenyl-4,4′-disulfonate

Crystal data

$2NH_4^+\cdot C_{12}H_8O_6S_2^{2-}$
$M_r = 348.39$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.778$ (2) Å
$b = 7.4138$ (12) Å
$c = 7.6647$ (13) Å
$\beta = 96.667$ (13)°
$V = 834.1$ (2) Å^3
$Z = 2$

$F(000) = 364$
$D_x = 1.387$ Mg m^{-3}
Melting point > 573 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 13.7$–17.1°
$\mu = 0.35$ mm^{-1}
$T = 297$ K
Plate, colourless
0.40 × 0.30 × 0.11 mm

Data collection

Rigaku AFC 7R four-circle diffractometer
Radiation source: Rigaku rotating anode
Graphite monochromator
ω–2θ scans
Absorption correction: ψ scan (*TEXSAN* for Windows; Molecular Structure Corporation,1999)
$T_{min} = 0.874$, $T_{max} = 0.963$
2129 measured reflections

1909 independent reflections
1030 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.8°$
$h = -8 \rightarrow 19$
$k = 0 \rightarrow 9$
$l = -9 \rightarrow 9$
3 standard reflections every 150 min
intensity decay: 1.6%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.172$

$S = 0.86$
1909 reflections
116 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.8639P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.15898 (5)	0.00272 (12)	0.19165 (11)	0.0282 (2)
O11	0.15361 (18)	−0.1393 (4)	0.0601 (3)	0.0415 (8)
O12	0.14295 (19)	0.1783 (4)	0.1126 (4)	0.0458 (9)
O13	0.10228 (17)	−0.0332 (4)	0.3285 (3)	0.0442 (9)
C1	0.2732 (2)	0.0001 (5)	0.2931 (4)	0.0313 (9)
C2	0.3348 (3)	−0.1181 (8)	0.2409 (8)	0.079 (2)
C3	0.4233 (3)	−0.1161 (8)	0.3218 (8)	0.086 (2)
C4	0.4528 (2)	0.0004 (6)	0.4560 (5)	0.0383 (11)
C5	0.3888 (3)	0.1184 (8)	0.5045 (6)	0.0660 (18)
C6	0.3001 (3)	0.1182 (8)	0.4249 (7)	0.0664 (18)
N1	−0.0887 (3)	0.0292 (5)	0.2899 (5)	0.0376 (11)
H2	0.31710	−0.20180	0.14940	0.0940*
H3	0.46580	−0.19870	0.28270	0.1020*
H5	0.40600	0.20300	0.59550	0.0790*
H6	0.25740	0.20130	0.46240	0.0800*
H11	−0.107 (3)	0.055 (5)	0.175 (6)	0.039 (11)*
H12	−0.110 (3)	0.096 (7)	0.357 (6)	0.055 (15)*
H13	−0.027 (4)	0.035 (6)	0.299 (5)	0.055 (14)*
H14	−0.109 (3)	−0.076 (7)	0.323 (6)	0.053 (14)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0262 (4)	0.0305 (4)	0.0273 (4)	−0.0015 (4)	0.0011 (3)	0.0018 (4)
O11	0.0439 (16)	0.0432 (15)	0.0345 (13)	0.0003 (13)	−0.0073 (11)	−0.0066 (12)
O12	0.0426 (16)	0.0346 (15)	0.0595 (17)	0.0035 (12)	0.0030 (13)	0.0109 (13)
O13	0.0284 (13)	0.073 (2)	0.0313 (13)	−0.0046 (13)	0.0043 (10)	0.0096 (13)
C1	0.0245 (15)	0.0370 (17)	0.0324 (16)	−0.0007 (17)	0.0030 (12)	−0.0043 (18)
C2	0.042 (3)	0.089 (4)	0.097 (4)	0.024 (3)	−0.026 (3)	−0.067 (3)
C3	0.041 (3)	0.098 (4)	0.110 (4)	0.028 (3)	−0.023 (3)	−0.073 (4)

C4	0.0226 (17)	0.051 (2)	0.0404 (19)	−0.0011 (19)	0.0003 (14)	−0.010 (2)
C5	0.034 (2)	0.090 (4)	0.071 (3)	0.010 (2)	−0.006 (2)	−0.053 (3)
C6	0.031 (2)	0.090 (4)	0.075 (3)	0.016 (2)	−0.007 (2)	−0.054 (3)
N1	0.039 (2)	0.046 (2)	0.0285 (17)	0.0049 (16)	0.0073 (14)	0.0010 (16)

Geometric parameters (Å, º)

S1—O11	1.454 (3)	C2—C3	1.381 (7)
S1—O12	1.444 (3)	C3—C4	1.375 (7)
S1—O13	1.441 (3)	C4—C5	1.371 (6)
S1—C1	1.775 (3)	C4—C4^{i}	1.477 (4)
N1—H14	0.88 (5)	C5—C6	1.380 (6)
N1—H11	0.91 (5)	C2—H2	0.9500
N1—H12	0.80 (5)	C3—H3	0.9500
N1—H13	0.91 (6)	C5—H5	0.9500
C1—C6	1.361 (6)	C6—H6	0.9500
C1—C2	1.357 (6)		

O11—S1—O12	111.65 (17)	C1—C2—C3	119.6 (5)
O11—S1—O13	112.42 (16)	C2—C3—C4	123.0 (5)
O11—S1—C1	105.58 (16)	C3—C4—C5	115.8 (4)
O12—S1—O13	113.02 (17)	C4^{i}—C4—C5	121.6 (4)
O12—S1—C1	107.17 (17)	C3—C4—C4^{i}	122.7 (4)
O13—S1—C1	106.43 (15)	C4—C5—C6	122.0 (5)
H12—N1—H14	101 (5)	C1—C6—C5	120.6 (5)
H13—N1—H14	113 (4)	C1—C2—H2	120.00
H11—N1—H14	113 (4)	C3—C2—H2	120.00
H11—N1—H12	113 (4)	C4—C3—H3	118.00
H11—N1—H13	104 (4)	C2—C3—H3	119.00
H12—N1—H13	113 (4)	C4—C5—H5	119.00
C2—C1—C6	119.1 (4)	C6—C5—H5	119.00
S1—C1—C2	121.0 (3)	C5—C6—H6	120.00
S1—C1—C6	120.0 (3)	C1—C6—H6	120.00

O11—S1—C1—C2	0.7 (4)	C1—C2—C3—C4	0.6 (9)
O11—S1—C1—C6	−179.5 (3)	C2—C3—C4—C5	−0.9 (8)
O12—S1—C1—C2	−118.4 (4)	C2—C3—C4—C4^{i}	178.9 (5)
O12—S1—C1—C6	61.4 (4)	C3—C4—C5—C6	0.8 (7)
O13—S1—C1—C2	120.4 (4)	C4^{i}—C4—C5—C6	−179.0 (5)
O13—S1—C1—C6	−59.8 (4)	C3—C4—C4^{i}—C3^{i}	−180.0 (5)
S1—C1—C2—C3	179.6 (4)	C3—C4—C4^{i}—C5^{i}	0.2 (7)
C6—C1—C2—C3	−0.2 (8)	C5—C4—C4^{i}—C3^{i}	−0.2 (7)
S1—C1—C6—C5	−179.7 (4)	C5—C4—C4^{i}—C5^{i}	180.0 (5)
C2—C1—C6—C5	0.1 (7)	C4—C5—C6—C1	−0.5 (8)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H11···O11^{ii}	0.91 (5)	1.96 (4)	2.861 (5)	172 (4)
N1—H12···O11^{iii}	0.80 (5)	2.18 (5)	2.922 (5)	153 (5)
N1—H12···O13^{iv}	0.80 (5)	2.45 (5)	2.955 (4)	122 (4)
N1—H13···O13	0.91 (6)	1.96 (6)	2.841 (5)	162 (4)
N1—H14···O12^{v}	0.88 (5)	1.97 (5)	2.848 (5)	174 (4)
C2—H2···O11	0.95	2.48	2.873 (6)	105
C6—H6···O12^{vi}	0.95	2.33	3.243 (6)	161

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