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Bis(dimethylammonium) 2,5-dihydroxybenzene-1,4-disulfonate

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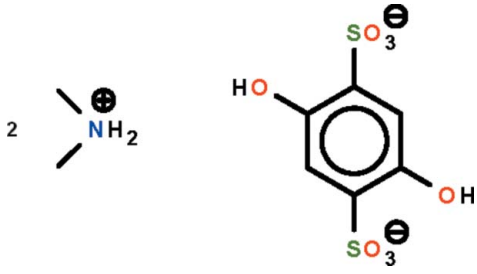
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.115; data-to-parameter ratio = 16.5.

In the crystal of the title salt, $2C_2H_8N^+\cdot C_6H_4O_8S_2^{2-}$, the anion lies on a center of inversion. The dimethylammonium cation forms one N—H···O hydrogen bond and another bifurcated N—H···O hydrogen bond. The hydroxy group links with the sulfonyl group *via* an intermolecular O—H···O hydrogen bond. These N—H···O and O—H···O hydrogen bonds generate a three-dimensional network.

Related literature

For the diethylammonium salt, see: Solans *et al.* (1982).



Experimental

Crystal data

$2C_2H_8N^+\cdot C_6H_4O_8S_2^{2-}$
M_r = 360.40
Monoclinic, $P2_1/c$
a = 8.0136 (12) Å
b = 12.2741 (19) Å
c = 9.2061 (16) Å
β = 115.268 (5)°
V = 818.9 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 293 K
0.25 × 0.20 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.770, T_{max} = 1.000
7785 measured reflections
1849 independent reflections
1675 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.115
S = 1.07
1849 reflections
112 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O3^{i}	0.83 (1)	1.85 (1)	2.670 (2)	175 (2)
N1—H1···O1	0.88 (1)	2.13 (2)	2.866 (2)	140 (2)
N1—H1···O1^{ii}	0.88 (1)	2.21 (2)	2.921 (2)	138 (2)
N1—H2···O2^{iii}	0.89 (1)	2.07 (2)	2.837 (2)	143 (3)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5456).

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supplementary materials

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Bis(dimethylammonium) 2,5-dihydroxybenzene-1,4-disulfonate

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Comment

Bis(diethylammonium) 2,5-dihydroxy-1,4-benzenedisulfonate is a commercial pharmacological chemical whose crystal structure has been described (Solans *et al.*, 1982). The title dimethyammonium salt (Scheme I) is an unexpected product of a hydrothermal synthesis involving 2,5-dihydroxy-1,4-benzenesulfonate in DMS solvent; the dimethylammonium cation probably resulted from the decomposition of DMF. The anion lies on a center-of-inversion (Fig. 1). The dimethylammonium cation forms one N–H···O hydrogen bond and another bifurcated hydrogen bond. These N–H···O and O–H···O hydrogen bonds generate a three-dimensional network (Table 1).

Experimental

DMF (8 ml), magnesium hydroxide (1 mmol) and 1,4-dihydroxy-2,5-benzenedisulfonic acid (1 mmol) were heated in a 23-ml, Teflon-lined, stainless-stell Parr bomb at 413 K for 3 days. Colorless crystals were isolated from the cool vessel.

Refinement

The carbon-bound H-atoms were placed in a calculated position (C–H 0.93 and 0.96 Å) and were included in the refinement in the riding model approximation, *U*(H) set to 1.2*U*(C). The amino and hydroxy H-atoms were located in a difference Fourier map, and were refined with distance restraints of N–H 0.88±0.01 Å, O–H 0.84±0.01 Å; their temperature factors were refined.

Figures

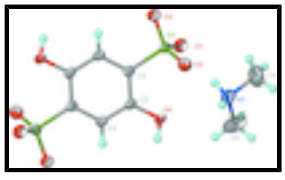

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $2(CH_3)_2NH_2{\cdot}C_6H_2(OH)_2(SO_3)_2$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Bis(dimethylammonium) 2,5-dihydroxybenzene-1,4-disulfonate

Crystal data

$2C_2H_8N^+{\cdot}C_6H_4O_8S_2^{2-}$	$F(000) = 380$
$M_r = 360.40$	$D_x = 1.462$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 5427 reflections
$a = 8.0136$ (12) Å	$\theta = 3.3$–$27.4°$

b = 12.2741 (19) Å	μ = 0.36 mm^{-1}
c = 9.2061 (16) Å	T = 293 K
β = 115.268 (5)°	Prism, colorless
V = 818.9 (2) Å^3	0.25 × 0.20 × 0.15 mm
Z = 2	

Data collection

Rigaku R-AXIS RAPID IP diffractometer	1849 independent reflections
Radiation source: fine-focus sealed tube	1675 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.037
ω scan	θ_{max} = 27.4°, θ_{min} = 3.3°
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	h = −9→10
T_{min} = 0.770, T_{max} = 1.000	k = −15→15
7785 measured reflections	l = −11→11

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.042	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.115	H atoms treated by a mixture of independent and constrained refinement
S = 1.07	$w = 1/[\sigma^2(F_o^2) + (0.0776P)^2 + 0.1341P]$ where $P = (F_o^2 + 2F_c^2)/3$
1849 reflections	$(\Delta/\sigma)_{max}$ = 0.001
112 parameters	$\Delta\rho_{max}$ = 0.78 e Å^{-3}
3 restraints	$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.31946 (5)	0.62757 (3)	0.66986 (4)	0.02663 (17)
O1	0.14241 (15)	0.57550 (9)	0.57815 (14)	0.0388 (3)
O2	0.44282 (17)	0.61906 (10)	0.59274 (16)	0.0405 (3)
O3	0.29844 (16)	0.73891 (8)	0.71461 (14)	0.0358 (3)
O4	0.4472 (2)	0.39499 (10)	0.71737 (15)	0.0439 (3)
H4	0.523 (2)	0.3448 (13)	0.742 (3)	0.047 (6)*
N1	0.1913 (2)	0.41948 (15)	0.3680 (2)	0.0466 (4)
H1	0.132 (3)	0.445 (2)	0.422 (3)	0.076 (8)*
H2	0.3121 (16)	0.432 (2)	0.410 (3)	0.085 (9)*
C1	0.4220 (2)	0.55462 (11)	0.85428 (17)	0.0274 (3)
C2	0.4752 (2)	0.44621 (12)	0.85763 (18)	0.0305 (3)
C3	0.5534 (2)	0.39234 (12)	1.00446 (19)	0.0308 (3)

H3	0.5897	0.3200	1.0083	0.037*
C4	0.1193 (3)	0.4894 (2)	0.2257 (3)	0.0727 (7)
H4A	0.1489	0.5640	0.2578	0.109*
H4B	−0.0122	0.4812	0.1710	0.109*
H4C	0.1741	0.4689	0.1551	0.109*
C5	0.1563 (3)	0.3028 (2)	0.3375 (4)	0.0769 (8)
H5A	0.2122	0.2637	0.4372	0.115*
H5B	0.2081	0.2784	0.2665	0.115*
H5C	0.0257	0.2898	0.2890	0.115*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0296 (2)	0.0240 (2)	0.0307 (3)	−0.00098 (12)	0.01698 (18)	0.00029 (12)
O1	0.0337 (6)	0.0388 (6)	0.0430 (7)	−0.0054 (5)	0.0156 (5)	−0.0072 (5)
O2	0.0418 (7)	0.0465 (7)	0.0445 (7)	0.0048 (5)	0.0293 (6)	0.0084 (5)
O3	0.0463 (6)	0.0230 (5)	0.0402 (6)	0.0005 (4)	0.0205 (5)	0.0012 (4)
O4	0.0667 (9)	0.0330 (6)	0.0303 (6)	0.0166 (6)	0.0192 (6)	−0.0040 (5)
N1	0.0400 (8)	0.0577 (10)	0.0451 (9)	0.0031 (7)	0.0211 (7)	−0.0086 (7)
C1	0.0334 (7)	0.0236 (7)	0.0302 (7)	−0.0008 (5)	0.0183 (6)	0.0001 (5)
C2	0.0409 (8)	0.0251 (7)	0.0300 (8)	0.0011 (6)	0.0194 (6)	−0.0040 (5)
C3	0.0423 (8)	0.0205 (6)	0.0346 (8)	0.0029 (6)	0.0211 (7)	−0.0017 (5)
C4	0.0601 (13)	0.110 (2)	0.0519 (13)	−0.0003 (14)	0.0279 (11)	0.0132 (13)
C5	0.0589 (13)	0.0636 (15)	0.120 (2)	−0.0071 (11)	0.0497 (15)	−0.0276 (14)

Geometric parameters (Å, °)

S1—O2	1.4462 (12)	C1—C2	1.394 (2)
S1—O1	1.4531 (11)	C2—C3	1.391 (2)
S1—O3	1.4577 (11)	C3—C1^{i}	1.391 (2)
S1—C1	1.7799 (15)	C3—H3	0.9300
O4—C2	1.3657 (18)	C4—H4A	0.9600
O4—H4	0.827 (9)	C4—H4B	0.9600
N1—C5	1.463 (3)	C4—H4C	0.9600
N1—C4	1.462 (3)	C5—H5A	0.9600
N1—H1	0.879 (10)	C5—H5B	0.9600
N1—H2	0.890 (10)	C5—H5C	0.9600
C1—C3^{i}	1.391 (2)		
O2—S1—O1	112.67 (8)	O4—C2—C1	119.56 (14)
O2—S1—O3	113.09 (7)	C3—C2—C1	118.83 (13)
O1—S1—O3	112.00 (7)	C2—C3—C1^{i}	120.76 (13)
O2—S1—C1	107.34 (7)	C2—C3—H3	119.6
O1—S1—C1	105.76 (7)	C1^{i}—C3—H3	119.6
O3—S1—C1	105.31 (7)	N1—C4—H4A	109.5
C2—O4—H4	106.1 (15)	N1—C4—H4B	109.5
C5—N1—C4	115.6 (2)	H4A—C4—H4B	109.5
C5—N1—H1	110.4 (19)	N1—C4—H4C	109.5
C4—N1—H1	101.2 (19)	H4A—C4—H4C	109.5

C5—N1—H2	110 (2)	H4B—C4—H4C	109.5
C4—N1—H2	103.1 (19)	N1—C5—H5A	109.5
H1—N1—H2	116 (3)	N1—C5—H5B	109.5
C3^{i}—C1—C2	120.41 (13)	H5A—C5—H5B	109.5
C3^{i}—C1—S1	118.67 (11)	N1—C5—H5C	109.5
C2—C1—S1	120.91 (11)	H5A—C5—H5C	109.5
O4—C2—C3	121.61 (13)	H5B—C5—H5C	109.5
O2—S1—C1—C3^{i}	126.33 (13)	C3^{i}—C1—C2—O4	178.89 (14)
O1—S1—C1—C3^{i}	−113.16 (13)	S1—C1—C2—O4	−0.5 (2)
O3—S1—C1—C3^{i}	5.58 (14)	C3^{i}—C1—C2—C3	−0.1 (3)
O2—S1—C1—C2	−54.28 (14)	S1—C1—C2—C3	−179.49 (11)
O1—S1—C1—C2	66.23 (14)	O4—C2—C3—C1^{i}	−178.87 (14)
O3—S1—C1—C2	−175.03 (12)	C1—C2—C3—C1^{i}	0.1 (3)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3^{ii}	0.83 (1)	1.85 (1)	2.670 (2)	175 (2)
N1—H1···O1	0.88 (1)	2.13 (2)	2.866 (2)	140 (2)
N1—H1···O1^{iii}	0.88 (1)	2.21 (2)	2.921 (2)	138 (2)
N1—H2···O2^{iv}	0.89 (1)	2.07 (2)	2.837 (2)	143 (3)

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

Fig. 1