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Redetermination of ethylenediammonium bis(*p*-methylbenzenesulfonate) monohydrate

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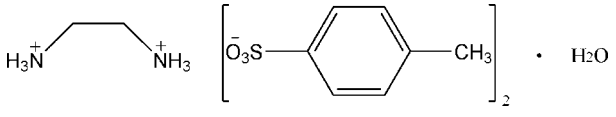
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.052; wR factor = 0.151; data-to-parameter ratio = 9.9.

In the asymmetric unit of the title compound, $C_2H_{10}N_2^{2+}\cdot 2C_7H_7O_3S^-\cdot H_2O$, there are two independent 4-methylbenzenesulfonate anions, one ethylenediammonium cation and a water molecule. The present redetermination was carried out to improve the treatment of disorder, which was not refined in the previous study [Ahn & Kim (1985). *J. Korean Chem. Soc.* **29**, 335–340]. One of the sulfonate groups is disordered over two positions, with site-occupancy factors of 0.588 (14) and 0.412 (14). Intermolecular N—H···O and O—H···O hydrogen bonds hold the three components together, affording a layer structure extending parallel to the (001) plane.

Related literature

The crystal structure of the title compound has been reported previously by Ahn & Kim (1985). For related compounds, see: Edwards *et al.* (2001); Bryant *et al.* (1993); Nakamura & Iitaka (1978); Nethaji *et al.* (1992).



Experimental

Crystal data

$C_2H_{10}N_2^{2+}\cdot 2C_7H_7O_3S^-\cdot H_2O$
M_r = 422.53
Monoclinic, $P2_1$
a = 11.302 (2) Å
b = 7.724 (1) Å
c = 12.648 (2) Å
β = 111.77 (1)°
V = 1025.4 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 293 (2) K
0.56 × 0.44 × 0.44 mm

Data collection

Siemens $P4$ diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.865, T_{max} = 0.877
2942 measured reflections
2693 independent reflections
2293 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
3 standard reflections
every 97 reflections
intensity decay: 5.0%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.151
S = 1.06
2693 reflections
273 parameters
7 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}
Absolute structure: Flack (1983), with 631 Friedel pairs
Flack parameter: −0.13 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O7^{i}	0.89	1.87	2.736 (7)	165
N1—H1B···O5^{ii}	0.89	2.14	2.942 (8)	149
N1—H1B···O6^{ii}	0.89	2.57	3.365 (5)	148
N1—H1B···O6′^{ii}	0.89	1.88	2.735 (8)	162
N1—H1C···O2	0.89	1.87	2.761 (6)	176
N2—H2A···O6^{iii}	0.89	2.03	2.780 (7)	142
N2—H2B···O4	0.89	1.78	2.665 (5)	177
N2—H2C···O1^{iv}	0.89	2.05	2.893 (6)	157
O7—H7D···O5	0.82	2.15	2.793 (5)	134
O7—H7E···O1^{iv}	0.82	2.15	2.938 (9)	160

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

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); 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: IS2252).

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supplementary materials

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Redetermination of ethylenediammonium bis(*p*-methylbenzenesulfonate) monohydrate

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Comment

Previously, Ahn and Kim (1985) have reported X-ray diffraction study of the title compound, (I), with $R = 0.060$. The present redetermination of (I) gives improvement in the treatment of disorder.

In compound (I), one of sulfonate groups of 4-methylbenzenesulfonate anions is disordered (Fig. 1). In the previous paper (Ahn & Kim, 1985), these occupancy factors were not refined and for each disordered O atom a different occupancy factor was given, which could not be accepted from the chemical point of view. The shortest S—O bond lengths in the disorder sulfonate group is of [1.410 (8) Å], it deviates greatly from the shortest one observed in the previous study [1.380 (16) Å]. The present C—N bond lengths [1.468 (8) and 1.480 (7) Å] are smaller obviously than the previous result [1.502 (12) and 1.527 (14) Å]. All C—C bond lengths by our redetermination are consistent with those observed in the Ahn & Kim's result, correspondingly. The present bond lengths and angles of (I) are coherent with those observed in tosylate (Bryant *et al.*, 1993; Nakamura & Iitaka, 1978) and ethylenediammonium (Edwards *et al.*, 2001; Nethaji *et al.*, 1992).

Experimental

4-Methylbenzenesulfonic acid (0.02 mol, 3.12 g), ethylenediamine (0.01 mol, 0.60 g) and sufficient water were added together at 373 K with stirring. The resulting solution was allowed to stand for 5 days at room temperature to give single crystals of (I).

Refinement

The sulfonate O atoms in one of 4-methylbenzenesulfonates are disordered over two sites, with occupancies of 0.412 (14) and 0.588 (14). S2—O4 and S2—O4', S2—O5 and S2—O5', and S2—O6 and S2—O6' are restrained to be identical with 0.01 Å deviation. The water H atoms were located in a difference Fourier map and the positions were fiexed, with U_{iso}(H) = 1.2U_{eq}(O). Other H atoms were placed in calculated positions (C—H = 0.93–0.96 Å and N—H = 0.89 Å), and allowed to ride on their parent atoms, with U_{iso}(H) values 1.2–1.5 times U_{eq} of the parent atoms.

Figures

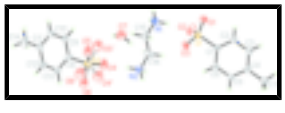


Fig. 1. A view of (I), showing 40% probability displacement ellipsoids.

ethylenediammonium bis(*p*-methylbenzenesulfonate) monohydrate

Crystal data

$C_2H_{10}N_2^{2+}\cdot 2C_7H_7O_3S^-\cdot H_2O$	$F_{000} = 448$
$M_r = 422.53$	$D_x = 1.368$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 31 reflections
$a = 11.302$ (2) Å	$\theta = 3.3$–18.3º
$b = 7.724$ (1) Å	$\mu = 0.30$ mm^{-1}
$c = 12.648$ (2) Å	$T = 293$ (2) K
$\beta = 111.77$ (1)º	Rod, colorless
$V = 1025.4$ (3) Å^3	0.56 × 0.44 × 0.44 mm
$Z = 2$	

Data collection

Siemens P4 diffractometer	$R_{int} = 0.015$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.5$º
Monochromator: graphite	$\theta_{min} = 1.7$º
$T = 293(2)$ K	$h = -13\rightarrow13$
ω scans	$k = -8\rightarrow9$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = -15\rightarrow14$
$T_{min} = 0.865$, $T_{max} = 0.877$	3 standard reflections
2942 measured reflections	every 97 reflections
2693 independent reflections	intensity decay: 5.0%
2293 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.053$	$w = 1/[\sigma^2(F_o^2) + (0.0975P)^2 + 0.2733P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.151$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.46$ e Å^{-3}
2693 reflections	$\Delta\rho_{min} = -0.45$ e Å^{-3}
273 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
7 restraints	Extinction coefficient: 0.041 (7)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 631 Friedel pairs

Secondary atom site location: difference Fourier map Flack parameter: −0.13 (14)

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
S1	0.39487 (9)	1.37382 (17)	0.69865 (8)	0.0449 (3)	
S2	0.23545 (10)	0.3854 (2)	0.30145 (8)	0.0486 (3)	
N1	0.1694 (3)	1.0595 (6)	0.4993 (3)	0.0493 (9)	
H1A	0.0876	1.0493	0.4898	0.074*	
H1B	0.1759	1.1189	0.4415	0.074*	
H1C	0.2108	1.1151	0.5642	0.074*	
N2	0.4215 (4)	0.7319 (6)	0.5269 (4)	0.0572 (10)	
H2A	0.5031	0.7456	0.5365	0.086*	
H2B	0.3816	0.6734	0.4629	0.086*	
H2C	0.4163	0.6734	0.5856	0.086*	
O3	0.4599 (3)	1.3428 (6)	0.6225 (3)	0.0651 (11)	
O1	0.3297 (4)	1.5388 (6)	0.6770 (4)	0.0644 (11)	
O2	0.3079 (4)	1.2347 (7)	0.6970 (4)	0.0806 (14)	
O4	0.3036 (16)	0.5455 (15)	0.3399 (9)	0.074 (4)	0.412 (14)
O5	0.1256 (9)	0.3402 (16)	0.3317 (8)	0.073 (4)	0.412 (14)
O6	0.3193 (14)	0.2405 (15)	0.3414 (10)	0.074 (4)	0.412 (14)
O4'	0.3607 (7)	0.4619 (15)	0.3379 (6)	0.072 (3)	0.588 (14)
O5'	0.1431 (8)	0.4885 (14)	0.3320 (7)	0.090 (4)	0.588 (14)
O6'	0.2406 (12)	0.2116 (11)	0.3368 (8)	0.091 (3)	0.588 (14)
O7	0.0703 (3)	0.4716 (7)	0.5140 (3)	0.0804 (14)	
H7D	0.0782	0.4933	0.4533	0.097*	
H7E	0.1349	0.4841	0.5713	0.097*	
C1	0.5101 (3)	1.3818 (7)	0.8381 (3)	0.0373 (7)	
C2	0.4835 (4)	1.4629 (7)	0.9233 (4)	0.0482 (10)	
H2	0.4066	1.5206	0.9070	0.058*	
C3	0.5726 (4)	1.4581 (7)	1.0342 (4)	0.0531 (11)	
H3	0.5544	1.5137	1.0916	0.064*	
C4	0.6870 (4)	1.3731 (8)	1.0608 (3)	0.0462 (9)	
C5	0.7114 (4)	1.2905 (7)	0.9742 (4)	0.0496 (11)	
H5	0.7879	1.2313	0.9911	0.060*	
C6	0.6248 (4)	1.2935 (7)	0.8628 (4)	0.0463 (10)	
H6	0.6429	1.2375	0.8055	0.056*	

C7	0.7803 (5)	1.3664 (10)	1.1808 (4)	0.0645 (13)
H7A	0.7475	1.4305	1.2290	0.097*
H7B	0.8597	1.4161	1.1848	0.097*
H7C	0.7937	1.2481	1.2058	0.097*
C8	0.1792 (3)	0.3870 (7)	0.1514 (3)	0.0392 (8)
C9	0.0644 (4)	0.4651 (7)	0.0900 (4)	0.0467 (10)
H9	0.0167	0.5167	0.1275	0.056*
C10	0.0205 (4)	0.4663 (8)	−0.0284 (4)	0.0529 (12)
H10	−0.0564	0.5204	−0.0696	0.063*
C11	0.0887 (4)	0.3890 (8)	−0.0854 (3)	0.0514 (10)
C12	0.2040 (4)	0.3120 (7)	−0.0222 (4)	0.0515 (11)
H12	0.2520	0.2607	−0.0595	0.062*
C13	0.2487 (4)	0.3101 (7)	0.0948 (4)	0.0484 (10)
H13	0.3259	0.2568	0.1358	0.058*
C14	0.0403 (7)	0.3881 (13)	−0.2143 (4)	0.0838 (18)
H14A	−0.0405	0.4460	−0.2442	0.126*
H14B	0.0307	0.2707	−0.2411	0.126*
H14C	0.1002	0.4470	−0.2391	0.126*
C15	0.2250 (4)	0.8868 (9)	0.5035 (4)	0.0552 (11)
H15A	0.1797	0.8254	0.4332	0.066*
H15B	0.2170	0.8209	0.5659	0.066*
C16	0.3611 (5)	0.9038 (9)	0.5199 (6)	0.0727 (16)
H16A	0.4054	0.9679	0.5893	0.087*
H16B	0.3684	0.9683	0.4568	0.087*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0487 (5)	0.0482 (6)	0.0368 (5)	0.0021 (6)	0.0147 (4)	0.0040 (6)
S2	0.0539 (5)	0.0557 (7)	0.0343 (5)	−0.0051 (6)	0.0141 (4)	0.0083 (6)
N1	0.0506 (19)	0.048 (2)	0.051 (2)	0.0041 (17)	0.0214 (17)	0.0042 (19)
N2	0.060 (2)	0.051 (2)	0.067 (3)	0.011 (2)	0.032 (2)	0.008 (2)
O3	0.0771 (19)	0.080 (3)	0.0425 (16)	0.011 (2)	0.0271 (15)	−0.0027 (19)
O1	0.073 (2)	0.060 (2)	0.056 (2)	0.0253 (19)	0.0182 (18)	0.0121 (19)
O2	0.081 (2)	0.088 (3)	0.056 (2)	−0.035 (3)	0.0054 (19)	0.010 (2)
O4	0.106 (10)	0.064 (8)	0.039 (6)	−0.012 (7)	0.013 (6)	0.002 (5)
O5	0.099 (7)	0.069 (10)	0.039 (5)	−0.010 (6)	0.014 (5)	0.004 (5)
O6	0.102 (9)	0.067 (7)	0.040 (6)	−0.006 (7)	0.012 (6)	0.003 (5)
O4'	0.076 (4)	0.091 (7)	0.053 (4)	0.003 (4)	0.030 (3)	0.013 (4)
O5'	0.109 (6)	0.111 (10)	0.053 (4)	0.010 (6)	0.032 (4)	0.012 (5)
O6'	0.112 (8)	0.108 (8)	0.054 (5)	0.007 (7)	0.033 (5)	0.015 (5)
O7	0.0564 (18)	0.118 (4)	0.066 (2)	0.011 (2)	0.0218 (17)	0.002 (3)
C1	0.0422 (16)	0.0346 (19)	0.0365 (17)	0.003 (2)	0.0164 (14)	0.001 (2)
C2	0.046 (2)	0.051 (3)	0.051 (2)	0.009 (2)	0.0222 (18)	−0.003 (2)
C3	0.063 (3)	0.053 (3)	0.049 (2)	−0.001 (2)	0.028 (2)	−0.012 (2)
C4	0.0514 (19)	0.041 (2)	0.045 (2)	−0.005 (3)	0.0171 (16)	−0.001 (3)
C5	0.047 (2)	0.047 (3)	0.055 (3)	0.006 (2)	0.0192 (19)	0.003 (2)
C6	0.048 (2)	0.050 (3)	0.045 (2)	0.006 (2)	0.0227 (18)	−0.005 (2)

C7	0.069 (3)	0.065 (3)	0.051 (3)	−0.003 (3)	0.013 (2)	0.000 (3)
C8	0.0416 (16)	0.036 (2)	0.0402 (19)	−0.006 (2)	0.0154 (14)	0.004 (2)
C9	0.0431 (19)	0.051 (3)	0.048 (2)	0.0043 (19)	0.0192 (17)	0.004 (2)
C10	0.041 (2)	0.063 (3)	0.046 (2)	0.001 (2)	0.0061 (17)	0.011 (2)
C11	0.065 (2)	0.046 (3)	0.041 (2)	−0.007 (3)	0.0169 (18)	−0.004 (3)
C12	0.059 (2)	0.054 (3)	0.047 (2)	0.002 (2)	0.026 (2)	−0.002 (2)
C13	0.0444 (19)	0.050 (3)	0.050 (2)	0.0083 (19)	0.0172 (18)	0.005 (2)
C14	0.108 (4)	0.096 (5)	0.040 (2)	−0.016 (5)	0.018 (2)	0.006 (4)
C15	0.058 (2)	0.051 (3)	0.057 (3)	0.000 (3)	0.0215 (19)	0.005 (3)
C16	0.070 (3)	0.054 (4)	0.107 (4)	0.009 (3)	0.048 (3)	0.011 (3)

Geometric parameters (Å, °)

S1—O3	1.432 (3)	C4—C5	1.381 (7)
S1—O1	1.446 (4)	C4—C7	1.494 (6)
S1—O2	1.451 (4)	C5—C6	1.386 (7)
S1—C1	1.762 (4)	C5—H5	0.9300
S2—O6'	1.410 (8)	C6—H6	0.9300
S2—O6	1.432 (9)	C7—H7A	0.9600
S2—O4	1.442 (8)	C7—H7B	0.9600
S2—O4'	1.442 (7)	C7—H7C	0.9600
S2—O5	1.471 (8)	C8—C13	1.379 (6)
S2—O5'	1.473 (7)	C8—C9	1.379 (6)
S2—C8	1.764 (4)	C9—C10	1.392 (6)
N1—C15	1.468 (8)	C9—H9	0.9300
N1—H1A	0.8900	C10—C11	1.372 (7)
N1—H1B	0.8900	C10—H10	0.9300
N1—H1C	0.8900	C11—C12	1.385 (7)
N2—C16	1.480 (7)	C11—C14	1.515 (6)
N2—H2A	0.8900	C12—C13	1.374 (7)
N2—H2B	0.8900	C12—H12	0.9300
N2—H2C	0.8900	C13—H13	0.9300
O7—H7D	0.8228	C14—H14A	0.9600
O7—H7E	0.8218	C14—H14B	0.9600
C1—C2	1.372 (6)	C14—H14C	0.9600
C1—C6	1.394 (6)	C15—C16	1.480 (7)
C2—C3	1.391 (7)	C15—H15A	0.9700
C2—H2	0.9300	C15—H15B	0.9700
C3—C4	1.376 (7)	C16—H16A	0.9700
C3—H3	0.9300	C16—H16B	0.9700
O3—S1—O1	111.9 (2)	C5—C6—C1	118.9 (4)
O3—S1—O2	112.4 (3)	C5—C6—H6	120.5
O1—S1—O2	110.8 (3)	C1—C6—H6	120.5
O3—S1—C1	107.82 (18)	C4—C7—H7A	109.5
O1—S1—C1	107.1 (2)	C4—C7—H7B	109.5
O2—S1—C1	106.6 (2)	H7A—C7—H7B	109.5
O6—S2—O4	110.5 (8)	C4—C7—H7C	109.5
O6—S2—O5	103.5 (8)	H7A—C7—H7C	109.5
O4—S2—O5	120.8 (8)	H7B—C7—H7C	109.5

O6'—S2—O4'	111.6 (6)	C13—C8—C9	119.6 (4)
O6'—S2—O5'	112.4 (6)	C13—C8—S2	120.6 (3)
O4'—S2—O5'	113.7 (6)	C9—C8—S2	119.8 (3)
O6'—S2—C8	107.7 (4)	C8—C9—C10	119.6 (4)
O6—S2—C8	108.2 (5)	C8—C9—H9	120.2
O4—S2—C8	107.0 (5)	C10—C9—H9	120.2
O4'—S2—C8	105.1 (3)	C11—C10—C9	121.1 (4)
O5—S2—C8	106.2 (4)	C11—C10—H10	119.4
O5'—S2—C8	105.7 (3)	C9—C10—H10	119.4
C15—N1—H1A	109.5	C10—C11—C12	118.4 (4)
C15—N1—H1B	109.5	C10—C11—C14	121.2 (5)
H1A—N1—H1B	109.5	C12—C11—C14	120.4 (5)
C15—N1—H1C	109.5	C13—C12—C11	121.2 (4)
H1A—N1—H1C	109.5	C13—C12—H12	119.4
H1B—N1—H1C	109.5	C11—C12—H12	119.4
C16—N2—H2A	109.5	C12—C13—C8	120.1 (4)
C16—N2—H2B	109.5	C12—C13—H13	119.9
H2A—N2—H2B	109.5	C8—C13—H13	119.9
C16—N2—H2C	109.5	C11—C14—H14A	109.5
H2A—N2—H2C	109.5	C11—C14—H14B	109.5
H2B—N2—H2C	109.5	H14A—C14—H14B	109.5
H7D—O7—H7E	115.5	C11—C14—H14C	109.5
C2—C1—C6	120.2 (4)	H14A—C14—H14C	109.5
C2—C1—S1	120.4 (3)	H14B—C14—H14C	109.5
C6—C1—S1	119.2 (3)	N1—C15—C16	109.5 (5)
C1—C2—C3	119.5 (4)	N1—C15—H15A	109.8
C1—C2—H2	120.2	C16—C15—H15A	109.8
C3—C2—H2	120.2	N1—C15—H15B	109.8
C4—C3—C2	121.5 (4)	C16—C15—H15B	109.8
C4—C3—H3	119.2	H15A—C15—H15B	108.2
C2—C3—H3	119.2	C15—C16—N2	111.2 (5)
C3—C4—C5	118.2 (4)	C15—C16—H16A	109.4
C3—C4—C7	120.7 (4)	N2—C16—H16A	109.4
C5—C4—C7	121.1 (4)	C15—C16—H16B	109.4
C4—C5—C6	121.7 (4)	N2—C16—H16B	109.4
C4—C5—H5	119.2	H16A—C16—H16B	108.0
C6—C5—H5	119.2		
O3—S1—C1—C2	158.9 (4)	O5—S2—C8—C13	−135.5 (7)
O1—S1—C1—C2	38.3 (5)	O5'—S2—C8—C13	176.0 (6)
O2—S1—C1—C2	−80.3 (5)	O6'—S2—C8—C9	116.2 (7)
O3—S1—C1—C6	−26.0 (5)	O6—S2—C8—C9	154.9 (8)
O1—S1—C1—C6	−146.6 (4)	O4—S2—C8—C9	−86.0 (9)
O2—S1—C1—C6	94.8 (4)	O4'—S2—C8—C9	−124.7 (6)
C6—C1—C2—C3	0.8 (7)	O5—S2—C8—C9	44.3 (7)
S1—C1—C2—C3	175.9 (4)	O5'—S2—C8—C9	−4.2 (6)
C1—C2—C3—C4	−0.3 (8)	C13—C8—C9—C10	−0.4 (7)
C2—C3—C4—C5	−0.5 (8)	S2—C8—C9—C10	179.9 (4)
C2—C3—C4—C7	−178.8 (5)	C8—C9—C10—C11	0.8 (8)
C3—C4—C5—C6	0.7 (8)	C9—C10—C11—C12	−1.2 (9)

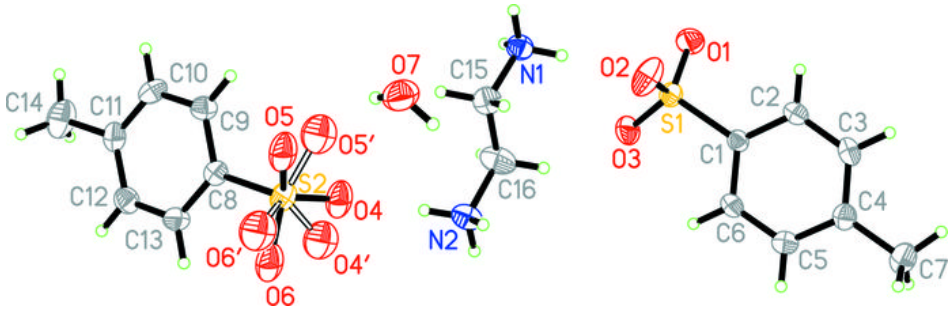
C7—C4—C5—C6	179.0 (5)	C9—C10—C11—C14	179.2 (6)
C4—C5—C6—C1	−0.2 (8)	C10—C11—C12—C13	1.0 (9)
C2—C1—C6—C5	−0.6 (7)	C14—C11—C12—C13	−179.4 (6)
S1—C1—C6—C5	−175.7 (4)	C11—C12—C13—C8	−0.6 (8)
O6'—S2—C8—C13	−63.6 (7)	C9—C8—C13—C12	0.2 (7)
O6—S2—C8—C13	−24.9 (9)	S2—C8—C13—C12	180.0 (4)
O4—S2—C8—C13	94.2 (9)	N1—C15—C16—N2	178.9 (4)
O4'—S2—C8—C13	55.5 (7)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O7^{i}	0.89	1.87	2.736 (7)	165
N1—H1B···O5^{ii}	0.89	2.14	2.942 (8)	149
N1—H1B···O6^{ii}	0.89	2.57	3.365 (5)	148
N1—H1B···O6^{ii}	0.89	1.88	2.735 (8)	162
N1—H1C···O2	0.89	1.87	2.761 (6)	176
N2—H2A···O6^{iii}	0.89	2.03	2.780 (7)	142
N2—H2B···O4	0.89	1.78	2.665 (5)	177
N2—H2C···O1^{iv}	0.89	2.05	2.893 (6)	157
O7—H7D···O5	0.82	2.15	2.793 (5)	134
O7—H7E···O1^{iv}	0.82	2.15	2.938 (9)	160

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

Fig. 1


C14
C11
C10
C9
C12
C8
C13
O5
O5′
S2
O4
O4′
O6′
O6
O7
C15
N1
C16
N2
O2
O1
S1
O3
C2
C3
C1
C4
C6
C5
C7