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4-Methylanilinium 4-hydroxybenzenesulfonate

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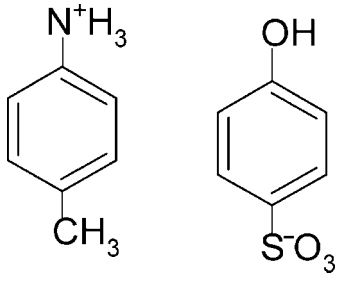
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.091; data-to-parameter ratio = 13.1.

In the crystal of the title molecular salt, $C_7H_{10}N^+\cdot C_6H_5O_4S^-$, the benzenesulfonate units are linked through phenol–sulfonate O—H···O hydrogen bonds, forming chains along the c-axis direction. These chains are linked *via* N—H···O hydrogen bonds involving two of the three H atoms of the ammonium group of the 4-methylanilium cation, giving rise to two-dimensional networks parallel to the bc plane which are further connected through an additional N—H···O interaction in which the third ammonium H atom is involved, generating a three-dimensional network.

Related literature

For the biological activity of related compounds, see: Fukami *et al.* (2000). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_7H_{10}N^+\cdot C_6H_5O_4S^-$
M_r = 281.32
Monoclinic, $P2_1/c$
a = 11.6450 (2) Å
b = 7.1670 (1) Å
c = 16.3080 (3) Å
β = 107.654 (1)°
V = 1296.96 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 293 K
0.30 × 0.30 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.926, T_{max} = 0.950
11047 measured reflections
2285 independent reflections
2045 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.091
S = 1.06
2285 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1C···O1^{i}	0.89	1.96	2.8367 (19)	169
N1—H1B···O4^{ii}	0.89	1.96	2.839 (2)	170
N1—H1A···O2^{iii}	0.89	1.94	2.8091 (19)	166
O4—H4···O3^{iii}	0.82	1.82	2.6343 (17)	173

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LR2102).

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supporting information

Acta Cryst. (2013). E**69**, o725 [https://doi.org/10.1107/S1600536813009410]

4-Methylanilinium 4-hydroxybenzenesulfonate

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S1. Comment

The stucture of the title compound, (I), is shown in Figure 1. Bond lengh and angles are within the standard values (Allen *et al.*, 1987). In the crystal the phenolsulfonate units are joined together through O4-H4···O3 hydrogen bonds, forming chains along the *c* crystallographic axis. These chains are subsequently linked together through N-H···O interactions involving the 4-methylanilium units. First, giving rise to a two-dimensional network parallel to *bc* plane, through interactions involving two of the three hydrogens of the ammonium moiety: N1-H1A···O2 and N1-H1C···O1 and finally generating a three-dimensional network through the use of the third hydrogen atom bonded to the nitrogen: N1-H1B···O4 (Table 1 and Figure 2).

S2. Experimental

The 4-MAPS compound was synthesized by the reaction of equimolar mixture of 4-methyl aniline and phenolsulfonic acid. To a saturated solution of 4-methyl aniline in acetone, phenolsulfonic acid was slowly added at room temperature. The solution was stirred for six hours to get an homogeneous solution, filtered and kept for slow evaporation at room temperature. After that a saturated solution of 4-MAPS was prepared by using methanol at room temperature. The prepared solution was kept to constant temperature in water bath at 30° C to avoid the effect of fluctuation in room temperature. An slow evaporation process was allowed for a period of 15 days. The grown crystals of an approximate size of 12 *x* 9 *x* 2 mm3 were harvested and re-crystallized to grow pure crystals for further studies.

S3. Refinement

H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H distance of 0.93 - 0.96 Å, N—H distance of 0.89 Å, O—H distance of 0.82 Å and U_{iso}(H) = 1.2Ueq(N and $C_{aromatic}$) and U_{iso}(H) = 1.5Ueq(C_{methyl})

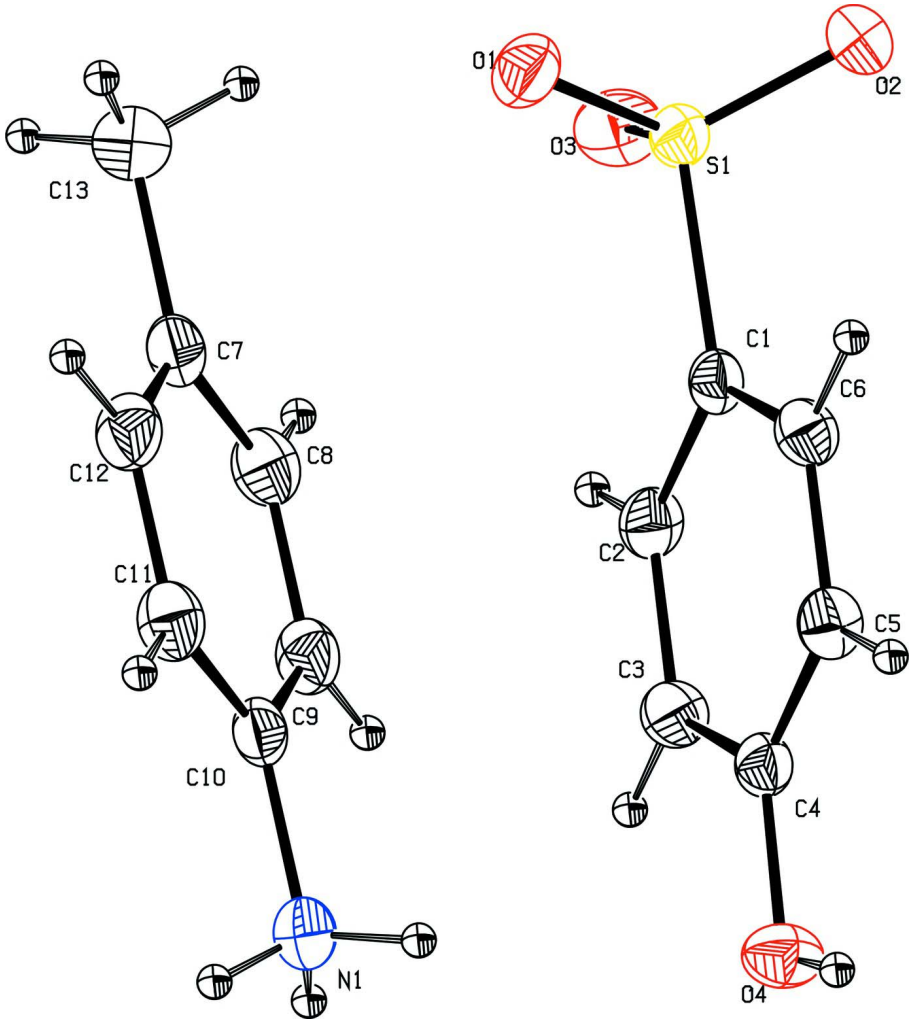

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

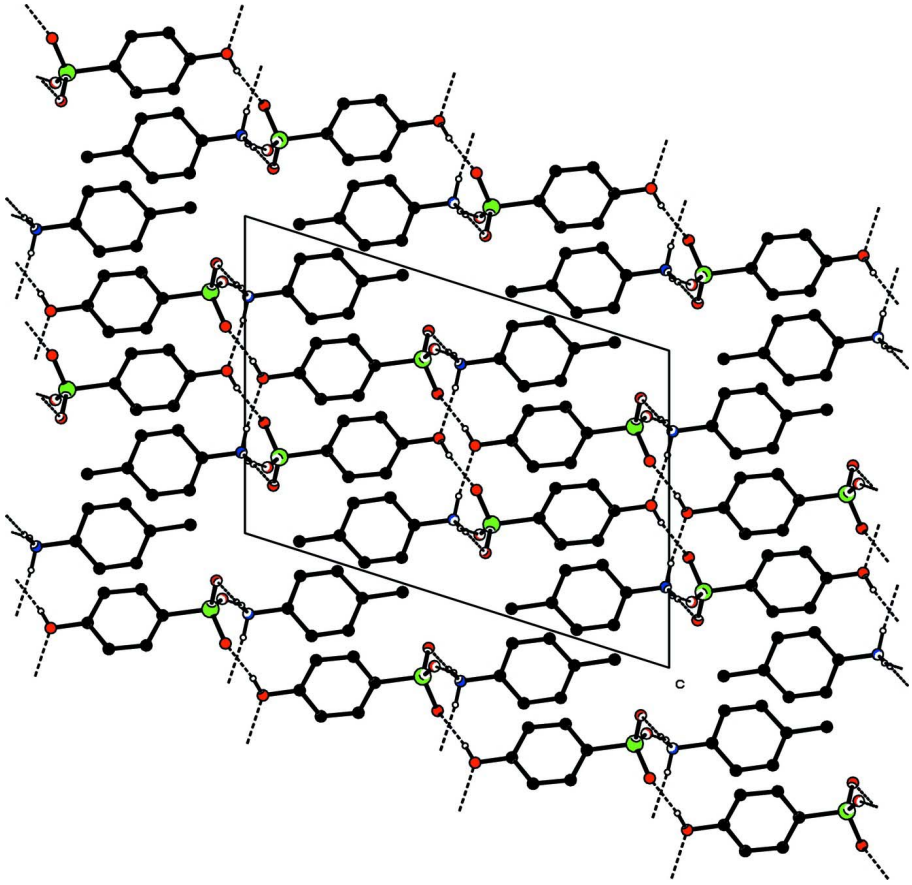

Figure 2

A view of the the packing of the the title compound. Dashed lines indicate hydrogen bonds.

4-Methylanilinium 4-hydroxybenzenesulfonate

Crystal data

$C_7H_{10}N^+·C_6H_5O_4S^-$
M_r = 281.32
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 11.6450 (2) Å
b = 7.1670 (1) Å
c = 16.3080 (3) Å
β = 107.654 (1)°
V = 1296.96 (4) Å^3
Z = 4
$F(000)$ = 592
D_x = 1.441 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5614 reflections
θ = 2.8–29.4°
μ = 0.26 mm^{-1}
T = 293 K
Block, colourless
0.30 × 0.30 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.926, T_{max} = 0.950
11047 measured reflections
2285 independent reflections
2045 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.0°, θ_{min} = 2.6°
h = −13→13
k = −8→8
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.091$
$S = 1.06$
2285 reflections
175 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.4532P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0070 (12)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.65813 (14)	0.2000 (2)	0.30612 (10)	0.0311 (4)
C2	0.54731 (14)	0.1192 (2)	0.26762 (10)	0.0375 (4)
H2	0.5057	0.0616	0.3011	0.045*
C3	0.49842 (14)	0.1242 (2)	0.17924 (10)	0.0391 (4)
H3	0.4240	0.0688	0.1532	0.047*
C4	0.55951 (14)	0.2109 (2)	0.12920 (10)	0.0328 (4)
C5	0.67033 (15)	0.2933 (2)	0.16791 (11)	0.0365 (4)
H5	0.7116	0.3521	0.1345	0.044*
C6	0.71913 (14)	0.2878 (2)	0.25594 (10)	0.0355 (4)
H6	0.7935	0.3432	0.2820	0.043*
C7	0.91246 (16)	−0.3025 (2)	0.27651 (11)	0.0420 (4)
C8	0.79646 (16)	−0.2324 (3)	0.24420 (12)	0.0455 (4)
H8	0.7542	−0.2001	0.2822	0.055*
C9	0.74260 (15)	−0.2096 (2)	0.15731 (12)	0.0423 (4)
H9	0.6646	−0.1628	0.1367	0.051*
C10	0.80527 (14)	−0.2568 (2)	0.10108 (11)	0.0353 (4)
C11	0.92034 (15)	−0.3268 (2)	0.13049 (12)	0.0432 (4)
H11	0.9623	−0.3581	0.0922	0.052*
C12	0.97244 (15)	−0.3499 (3)	0.21799 (12)	0.0450 (4)
H12	1.0500	−0.3986	0.2382	0.054*
C13	0.9715 (2)	−0.3262 (3)	0.37143 (13)	0.0614 (6)
H13A	0.9581	−0.2166	0.4011	0.092*
H13B	1.0565	−0.3446	0.3825	0.092*

H13C	0.9377	−0.4327	0.3914	0.092*
N1	0.74899 (13)	−0.2321 (2)	0.00829 (9)	0.0424 (4)
H1A	0.7692	−0.1211	−0.0076	0.064*
H1B	0.6692	−0.2387	−0.0039	0.064*
H1C	0.7743	−0.3214	−0.0200	0.064*
O1	0.82148 (11)	0.05029 (17)	0.43307 (7)	0.0458 (3)
O2	0.77002 (13)	0.37156 (18)	0.44678 (8)	0.0559 (4)
O3	0.63246 (11)	0.1221 (2)	0.45415 (8)	0.0526 (4)
O4	0.50641 (11)	0.21139 (18)	0.04252 (7)	0.0450 (3)
H4	0.5499	0.2661	0.0192	0.068*
S1	0.72587 (4)	0.18720 (6)	0.41863 (2)	0.03519 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0357 (8)	0.0251 (8)	0.0341 (8)	0.0030 (6)	0.0128 (7)	−0.0003 (6)
C2	0.0401 (9)	0.0344 (9)	0.0402 (9)	−0.0053 (7)	0.0156 (7)	0.0051 (7)
C3	0.0354 (8)	0.0381 (9)	0.0416 (9)	−0.0091 (7)	0.0084 (7)	0.0018 (7)
C4	0.0377 (8)	0.0283 (8)	0.0332 (8)	0.0027 (6)	0.0119 (7)	−0.0009 (6)
C5	0.0384 (9)	0.0356 (9)	0.0404 (9)	−0.0032 (7)	0.0193 (7)	0.0013 (7)
C6	0.0329 (8)	0.0336 (9)	0.0405 (9)	−0.0048 (7)	0.0119 (7)	−0.0026 (7)
C7	0.0459 (9)	0.0283 (9)	0.0484 (10)	−0.0016 (7)	0.0093 (8)	−0.0028 (7)
C8	0.0492 (10)	0.0398 (10)	0.0502 (10)	0.0040 (8)	0.0192 (8)	−0.0065 (8)
C9	0.0358 (9)	0.0345 (9)	0.0548 (11)	0.0065 (7)	0.0108 (8)	−0.0039 (8)
C10	0.0376 (8)	0.0243 (8)	0.0429 (9)	−0.0023 (7)	0.0105 (7)	−0.0014 (7)
C11	0.0404 (9)	0.0381 (9)	0.0542 (11)	0.0022 (7)	0.0191 (8)	−0.0033 (8)
C12	0.0349 (9)	0.0372 (9)	0.0589 (11)	0.0045 (7)	0.0079 (8)	0.0003 (8)
C13	0.0728 (14)	0.0525 (12)	0.0495 (12)	0.0006 (10)	0.0045 (10)	−0.0017 (9)
N1	0.0475 (8)	0.0328 (8)	0.0455 (8)	−0.0036 (6)	0.0119 (7)	0.0014 (6)
O1	0.0507 (7)	0.0420 (7)	0.0437 (7)	0.0128 (6)	0.0126 (5)	0.0063 (5)
O2	0.0760 (9)	0.0356 (7)	0.0452 (7)	−0.0002 (7)	0.0020 (6)	−0.0077 (6)
O3	0.0587 (8)	0.0666 (9)	0.0394 (7)	0.0015 (7)	0.0252 (6)	0.0000 (6)
O4	0.0464 (7)	0.0555 (8)	0.0325 (6)	−0.0069 (6)	0.0110 (5)	−0.0001 (5)
S1	0.0436 (3)	0.0304 (2)	0.0315 (2)	0.00412 (16)	0.01122 (17)	−0.00084 (15)

Geometric parameters (Å, º)

C1—C2	1.380 (2)	C9—C10	1.376 (2)
C1—C6	1.386 (2)	C9—H9	0.9300
C1—S1	1.7662 (16)	C10—C11	1.374 (2)
C2—C3	1.380 (2)	C10—N1	1.466 (2)
C2—H2	0.9300	C11—C12	1.381 (3)
C3—C4	1.382 (2)	C11—H11	0.9300
C3—H3	0.9300	C12—H12	0.9300
C4—O4	1.3603 (19)	C13—H13A	0.9600
C4—C5	1.385 (2)	C13—H13B	0.9600
C5—C6	1.375 (2)	C13—H13C	0.9600
C5—H5	0.9300	N1—H1A	0.8900

C6—H6	0.9300	N1—H1B	0.8900
C7—C12	1.385 (3)	N1—H1C	0.8900
C7—C8	1.387 (2)	O1—S1	1.4487 (12)
C7—C13	1.501 (3)	O2—S1	1.4414 (13)
C8—C9	1.374 (3)	O3—S1	1.4556 (13)
C8—H8	0.9300	O4—H4	0.8200

C2—C1—C6	119.85 (15)	C11—C10—N1	119.19 (15)
C2—C1—S1	120.82 (12)	C9—C10—N1	119.79 (15)
C6—C1—S1	119.30 (12)	C10—C11—C12	118.74 (16)
C1—C2—C3	119.81 (15)	C10—C11—H11	120.6
C1—C2—H2	120.1	C12—C11—H11	120.6
C3—C2—H2	120.1	C11—C12—C7	121.83 (16)
C2—C3—C4	120.32 (15)	C11—C12—H12	119.1
C2—C3—H3	119.8	C7—C12—H12	119.1
C4—C3—H3	119.8	C7—C13—H13A	109.5
O4—C4—C3	117.48 (14)	C7—C13—H13B	109.5
O4—C4—C5	122.66 (14)	H13A—C13—H13B	109.5
C3—C4—C5	119.85 (15)	C7—C13—H13C	109.5
C6—C5—C4	119.78 (15)	H13A—C13—H13C	109.5
C6—C5—H5	120.1	H13B—C13—H13C	109.5
C4—C5—H5	120.1	C10—N1—H1A	109.5
C5—C6—C1	120.38 (14)	C10—N1—H1B	109.5
C5—C6—H6	119.8	H1A—N1—H1B	109.5
C1—C6—H6	119.8	C10—N1—H1C	109.5
C12—C7—C8	117.63 (17)	H1A—N1—H1C	109.5
C12—C7—C13	120.91 (17)	H1B—N1—H1C	109.5
C8—C7—C13	121.46 (17)	C4—O4—H4	109.5
C9—C8—C7	121.47 (17)	O2—S1—O1	112.76 (8)
C9—C8—H8	119.3	O2—S1—O3	113.86 (8)
C7—C8—H8	119.3	O1—S1—O3	110.38 (8)
C8—C9—C10	119.31 (16)	O2—S1—C1	106.67 (7)
C8—C9—H9	120.3	O1—S1—C1	106.46 (7)
C10—C9—H9	120.3	O3—S1—C1	106.13 (7)
C11—C10—C9	121.02 (16)		

C6—C1—C2—C3	0.8 (2)	C8—C9—C10—C11	0.3 (3)
S1—C1—C2—C3	−177.26 (13)	C8—C9—C10—N1	−179.65 (15)
C1—C2—C3—C4	−0.5 (2)	C9—C10—C11—C12	0.2 (3)
C2—C3—C4—O4	−179.85 (15)	N1—C10—C11—C12	−179.88 (15)
C2—C3—C4—C5	0.0 (2)	C10—C11—C12—C7	−0.7 (3)
O4—C4—C5—C6	−179.95 (14)	C8—C7—C12—C11	0.8 (3)
C3—C4—C5—C6	0.2 (2)	C13—C7—C12—C11	−179.14 (17)
C4—C5—C6—C1	0.1 (2)	C2—C1—S1—O2	−134.51 (14)
C2—C1—C6—C5	−0.6 (2)	C6—C1—S1—O2	47.42 (14)
S1—C1—C6—C5	177.49 (12)	C2—C1—S1—O1	104.85 (14)
C12—C7—C8—C9	−0.3 (3)	C6—C1—S1—O1	−73.21 (14)
C13—C7—C8—C9	179.63 (17)	C2—C1—S1—O3	−12.76 (15)

C7—C8—C9—C10	−0.2 (3)	C6—C1—S1—O3	169.17 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···O1^{i}	0.89	1.96	2.8367 (19)	169
N1—H1*B*···O4^{ii}	0.89	1.96	2.839 (2)	170
N1—H1*A*···O2^{iii}	0.89	1.94	2.8091 (19)	166
O4—H4···O3^{iii}	0.82	1.82	2.6343 (17)	173

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