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Piperidinium 3-carboxy-4-hydroxybenzenesulfonate monohydrate

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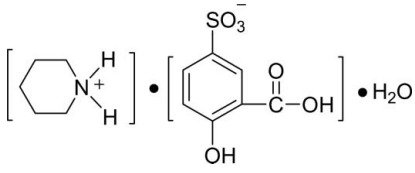
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.091; data-to-parameter ratio = 12.4.

The asymmetric unit of the title compound, $C_5H_{12}N^+\cdot C_7H_5O_6S^-\cdot H_2O$, contains a piperidinium cation, one 3-carboxy-4-hydroxybenzenesulfonate anion and one water molecule. Intermolecular O—H···O, O—H···S and N—H···O hydrogen bonds generate a three-dimensional hydrogen-bonded framework.

Related literature

For related literature, see: Smith *et al.* (2007).



Experimental

Crystal data

$C_5H_{12}N^+\cdot C_7H_5O_6S^-\cdot H_2O$
M_r = 321.34
Monoclinic, $P2_1/n$
a = 6.8895 (14) Å
b = 13.202 (3) Å
c = 16.255 (3) Å
β = 93.739 (3)°
V = 1475.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 294 (2) K
0.24 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.942, T_{max} = 0.961
7480 measured reflections
2602 independent reflections
1966 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.091
S = 1.04
2602 reflections
209 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7B···O1	0.855 (9)	1.933 (10)	2.786 (2)	176 (2)
O7—H7A···O2^{i}	0.859 (9)	1.912 (10)	2.770 (2)	177 (2)
N1—H1B···O5	0.88 (3)	2.55 (2)	2.983 (3)	110.6 (18)
N1—H1B···O7^{ii}	0.88 (3)	2.16 (3)	2.996 (3)	157 (2)
N1—H1A···O2^{iii}	0.92 (3)	1.90 (3)	2.807 (3)	170 (2)
O6—H6···O5	0.84 (3)	1.82 (3)	2.597 (2)	153 (3)
O4—H4···O7^{ii}	0.85 (3)	1.75 (3)	2.601 (2)	179 (3)

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2042).

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Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
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supporting information

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Piperidinium 3-carboxy-4-hydroxybenzenesulfonate monohydrate

Zhenhuan Li, Bowen Cheng and Su Kunmei

S1. Comment

5-Sulfosalicylic acid (SSA) has six potential donor sites in the three substituent groups (the sulfonic acid, the carboxylic acid and the phenolic groups), and it gives mono-, di- and trianionic ligand species through deprotonation. The presence of numerous oxygen atoms in the substituent groups usually results in hydrogen-bonding associations, and the self-assembly process of crystallization often requires the incorporation of water molecules in the structures (Smith *et al.* 2007). We report here the crystal structure of the title compound.

The asymmetric unit of the title compound contains one piperidium cation cation, one 3-carboxyl-4-hydroxyl-benzene-sulfonate anion and one water molecule (Fig. 1). The bond distances and angles in the cationic and anionic species are normal. An intramolecular O6—H6···O5 hydrogen bond is observed. The molecular packing (Fig. 2) is stabilized by intermolecular O—H···O, O—H···S and N—H···O hydrogen bonds (Table 1), These interactions generate a three-dimensional hydrogen-bonded framework structure.

S2. Experimental

2-Hydroxy-5-sulfobenzoic acid (2.18 g, 10 mmol), piperidine (0.85 g, 10 mmol) and H_2O (20 ml) were loaded into a 50 ml roundbottom flask, and heated to dissolve the solid. Crystals of the title compound were obtained by slow evaporation of deionic H_2O solution.

S3. Refinement

The H atoms of the water molecule, and the N-bound H atom H atom were located in a difference Fourier map, and refined with the O—H and N—H distance restraints of 0.86 (1) and 0.90 (1) Å, respectively. All other H atoms were positioned geometrically [O—H = 0.82 Å (hydroxyl), C—H = 0.93 Å (aromatic) and 0.96 Å (methyl)] and refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(carrier) for hydroxyl and methyl H atoms and 1.2U_{eq}(C) for other H atoms.

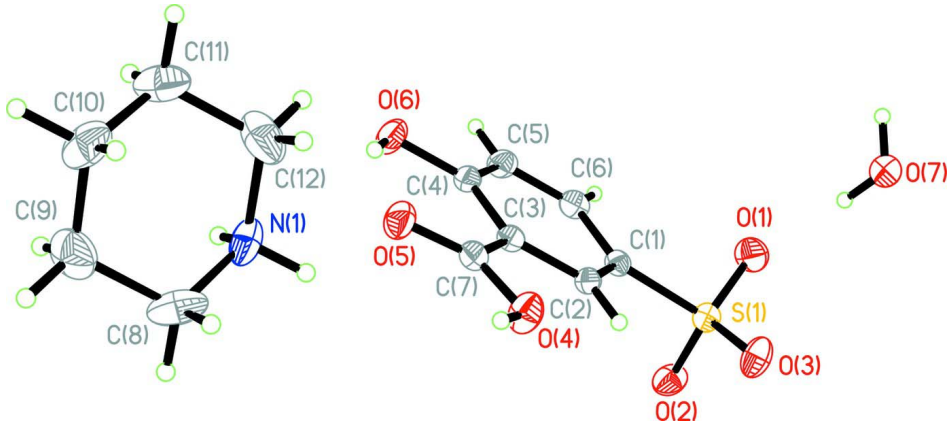

Figure 1

The asymmteric unit of title compound. Displacement ellipsoids are drawn at the 30% probability level.

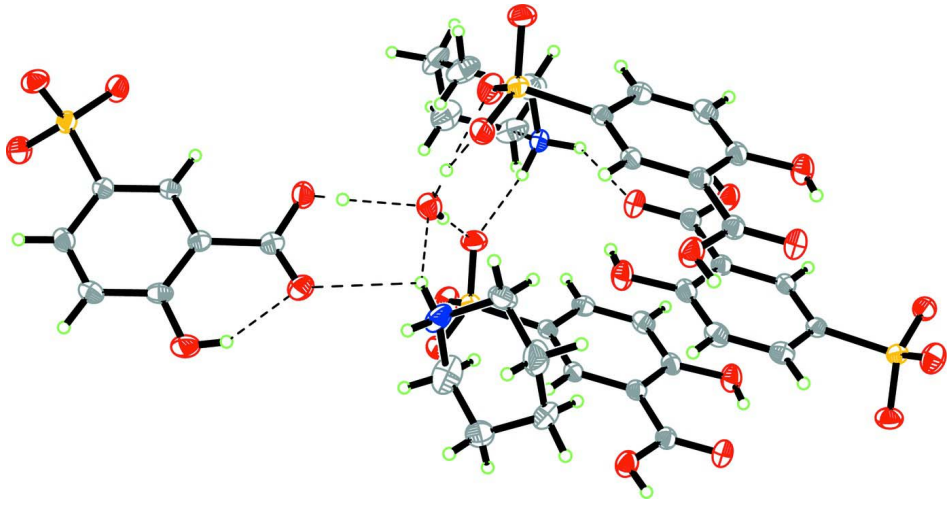

Figure 2

Part of the crystal packing of the title compound. O—H···O and N—H···O hydrogen bonds are shown as dashed lines.

Piperidinium 3-carboxy-4-hydroxybenzenesulfonate monohydrate

Crystal data

$C_5H_{12}N^+·C_7H_5O_6S^-·H_2O$
$M_r = 321.34$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.8895\ (14)$ Å
$b = 13.202\ (3)$ Å
$c = 16.255\ (3)$ Å
$\beta = 93.739\ (3)°$
$V = 1475.3\ (5)$ Å^3
$Z = 4$

$F(000) = 680$
$D_x = 1.447$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2608 reflections
$\theta = 3.0–25.1°$
$\mu = 0.25$ mm^{-1}
$T = 294$ K
Stick, colourless
0.24 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.942, T_{max} = 0.961
7480 measured reflections
2602 independent reflections
1966 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −8→8
k = −15→15
l = −19→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.091
S = 1.04
2602 reflections
209 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.0392P)^2 + 0.5098P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0191 (15)

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}
S1	0.16322 (7)	0.89594 (4)	0.24082 (3)	0.03289 (18)
O1	0.3120 (2)	0.93486 (13)	0.19039 (9)	0.0516 (4)
O2	−0.0297 (2)	0.93311 (11)	0.21241 (10)	0.0483 (4)
O3	0.1658 (2)	0.78755 (11)	0.25065 (9)	0.0479 (4)
O4	0.1660 (2)	0.78250 (12)	0.55005 (9)	0.0464 (4)
H4	0.160 (4)	0.752 (2)	0.5956 (17)	0.070*
O5	0.2197 (2)	0.91602 (11)	0.63192 (9)	0.0462 (4)
O6	0.2977 (2)	1.08950 (12)	0.56636 (10)	0.0487 (4)
H6	0.279 (4)	1.044 (2)	0.6018 (17)	0.073*
N1	0.1423 (3)	0.86332 (17)	0.80518 (12)	0.0481 (5)
H1A	0.100 (3)	0.928 (2)	0.7936 (15)	0.058*
H1B	0.124 (4)	0.8238 (18)	0.7615 (16)	0.058*
C1	0.2116 (3)	0.95050 (14)	0.33929 (12)	0.0294 (4)
C2	0.1907 (3)	0.89450 (15)	0.40946 (12)	0.0296 (4)
H2	0.1565	0.8265	0.4050	0.036*

C3	0.2204 (3)	0.93869 (15)	0.48727 (12)	0.0310 (5)
C4	0.2711 (3)	1.04151 (15)	0.49318 (13)	0.0349 (5)
C5	0.2947 (3)	1.09718 (16)	0.42200 (14)	0.0387 (5)
H5	0.3302	1.1651	0.4258	0.046*
C6	0.2656 (3)	1.05223 (15)	0.34626 (13)	0.0365 (5)
H6A	0.2819	1.0899	0.2989	0.044*
C7	0.2020 (3)	0.87879 (16)	0.56285 (13)	0.0345 (5)
C8	0.0182 (4)	0.81764 (19)	0.86596 (17)	0.0630 (8)
H8A	0.0510	0.7466	0.8732	0.076*
H8B	−0.1172	0.8221	0.8458	0.076*
C9	0.0467 (4)	0.8713 (2)	0.94665 (16)	0.0691 (8)
H9A	−0.0299	0.8381	0.9868	0.083*
H9B	0.0008	0.9405	0.9403	0.083*
C10	0.2573 (4)	0.8719 (2)	0.97778 (15)	0.0629 (7)
H10A	0.2722	0.9105	1.0286	0.075*
H10B	0.3002	0.8031	0.9895	0.075*
C11	0.3795 (4)	0.9178 (2)	0.91490 (17)	0.0648 (8)
H11A	0.3461	0.9888	0.9081	0.078*
H11B	0.5154	0.9136	0.9343	0.078*
C12	0.3510 (4)	0.8657 (3)	0.83399 (17)	0.0726 (9)
H12A	0.4242	0.9008	0.7937	0.087*
H12B	0.4002	0.7970	0.8390	0.087*
O7	0.6418 (2)	0.81286 (11)	0.18899 (10)	0.0456 (4)
H7A	0.7460 (19)	0.8483 (15)	0.1972 (16)	0.068*
H7B	0.5375 (18)	0.8480 (15)	0.1880 (17)	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0357 (3)	0.0317 (3)	0.0308 (3)	0.0030 (2)	−0.0010 (2)	0.0020 (2)
O1	0.0537 (10)	0.0640 (11)	0.0382 (9)	−0.0077 (8)	0.0121 (7)	0.0017 (8)
O2	0.0434 (9)	0.0428 (9)	0.0561 (10)	0.0062 (7)	−0.0162 (7)	−0.0029 (7)
O3	0.0720 (11)	0.0301 (8)	0.0405 (9)	0.0082 (7)	−0.0032 (8)	−0.0010 (7)
O4	0.0677 (11)	0.0385 (9)	0.0334 (9)	−0.0039 (8)	0.0054 (8)	0.0020 (7)
O5	0.0549 (10)	0.0521 (10)	0.0317 (9)	−0.0034 (8)	0.0037 (7)	−0.0078 (7)
O6	0.0595 (10)	0.0418 (10)	0.0445 (10)	−0.0072 (8)	0.0004 (8)	−0.0151 (7)
N1	0.0664 (14)	0.0453 (12)	0.0314 (10)	0.0145 (10)	−0.0064 (9)	−0.0087 (9)
C1	0.0253 (10)	0.0302 (11)	0.0327 (11)	0.0025 (8)	0.0022 (8)	−0.0003 (8)
C2	0.0268 (10)	0.0274 (10)	0.0347 (11)	0.0009 (8)	0.0017 (8)	−0.0035 (9)
C3	0.0243 (10)	0.0347 (11)	0.0340 (11)	0.0017 (8)	0.0014 (8)	−0.0026 (9)
C4	0.0263 (10)	0.0359 (12)	0.0422 (13)	0.0010 (9)	−0.0001 (9)	−0.0102 (10)
C5	0.0363 (12)	0.0291 (11)	0.0507 (14)	−0.0026 (9)	0.0032 (10)	−0.0027 (10)
C6	0.0332 (11)	0.0333 (12)	0.0430 (13)	0.0007 (9)	0.0034 (9)	0.0043 (10)
C7	0.0276 (11)	0.0398 (13)	0.0361 (12)	0.0019 (9)	0.0030 (9)	−0.0046 (10)
C8	0.0621 (17)	0.0474 (15)	0.0756 (19)	−0.0215 (13)	−0.0251 (14)	0.0167 (13)
C9	0.0687 (19)	0.092 (2)	0.0485 (16)	−0.0086 (16)	0.0178 (14)	0.0167 (15)
C10	0.082 (2)	0.0681 (18)	0.0357 (14)	−0.0133 (15)	−0.0139 (13)	0.0039 (12)
C11	0.0533 (16)	0.0748 (19)	0.0635 (18)	−0.0191 (14)	−0.0169 (13)	0.0147 (14)

C12	0.0524 (17)	0.107 (2)	0.0592 (18)	0.0259 (16)	0.0114 (13)	0.0104 (16)
O7	0.0447 (9)	0.0437 (9)	0.0486 (10)	−0.0015 (7)	0.0040 (8)	−0.0046 (7)

Geometric parameters (Å, º)

S1—O3	1.4398 (15)	C5—C6	1.370 (3)
S1—O1	1.4478 (16)	C5—H5	0.9300
S1—O2	1.4628 (15)	C6—H6A	0.9300
S1—C1	1.767 (2)	C8—C9	1.492 (4)
O4—C7	1.309 (3)	C8—H8A	0.9700
O4—H4	0.85 (3)	C8—H8B	0.9700
O5—C7	1.224 (2)	C9—C10	1.505 (4)
O6—C4	1.350 (2)	C9—H9A	0.9700
O6—H6	0.84 (3)	C9—H9B	0.9700
N1—C8	1.477 (3)	C10—C11	1.494 (4)
N1—C12	1.483 (3)	C10—H10A	0.9700
N1—H1A	0.92 (3)	C10—H10B	0.9700
N1—H1B	0.88 (3)	C11—C12	1.485 (4)
C1—C2	1.375 (3)	C11—H11A	0.9700
C1—C6	1.396 (3)	C11—H11B	0.9700
C2—C3	1.396 (3)	C12—H12A	0.9700
C2—H2	0.9300	C12—H12B	0.9700
C3—C4	1.403 (3)	O7—H7A	0.859 (9)
C3—C7	1.473 (3)	O7—H7B	0.855 (9)
C4—C5	1.389 (3)		
O3—S1—O1	114.30 (10)	O5—C7—C3	122.69 (19)
O3—S1—O2	111.89 (9)	O4—C7—C3	114.48 (18)
O1—S1—O2	111.36 (10)	N1—C8—C9	110.2 (2)
O3—S1—C1	107.72 (9)	N1—C8—H8A	109.6
O1—S1—C1	105.64 (9)	C9—C8—H8A	109.6
O2—S1—C1	105.25 (9)	N1—C8—H8B	109.6
C7—O4—H4	109.9 (19)	C9—C8—H8B	109.6
C4—O6—H6	105 (2)	H8A—C8—H8B	108.1
C8—N1—C12	112.9 (2)	C8—C9—C10	111.5 (2)
C8—N1—H1A	109.1 (15)	C8—C9—H9A	109.3
C12—N1—H1A	109.4 (16)	C10—C9—H9A	109.3
C8—N1—H1B	103.9 (16)	C8—C9—H9B	109.3
C12—N1—H1B	110.4 (16)	C10—C9—H9B	109.3
H1A—N1—H1B	111 (2)	H9A—C9—H9B	108.0
C2—C1—C6	119.45 (18)	C11—C10—C9	110.2 (2)
C2—C1—S1	120.57 (15)	C11—C10—H10A	109.6
C6—C1—S1	119.95 (15)	C9—C10—H10A	109.6
C1—C2—C3	120.67 (18)	C11—C10—H10B	109.6
C1—C2—H2	119.7	C9—C10—H10B	109.6
C3—C2—H2	119.7	H10A—C10—H10B	108.1
C2—C3—C4	119.16 (18)	C12—C11—C10	111.7 (2)
C2—C3—C7	121.05 (18)	C12—C11—H11A	109.3

C4—C3—C7	119.78 (18)	C10—C11—H11A	109.3
O6—C4—C5	117.99 (18)	C12—C11—H11B	109.3
O6—C4—C3	122.22 (19)	C10—C11—H11B	109.3
C5—C4—C3	119.79 (19)	H11A—C11—H11B	107.9
C6—C5—C4	120.09 (19)	N1—C12—C11	111.0 (2)
C6—C5—H5	120.0	N1—C12—H12A	109.4
C4—C5—H5	120.0	C11—C12—H12A	109.4
C5—C6—C1	120.8 (2)	N1—C12—H12B	109.4
C5—C6—H6A	119.6	C11—C12—H12B	109.4
C1—C6—H6A	119.6	H12A—C12—H12B	108.0
O5—C7—O4	122.83 (19)	H7A—O7—H7B	113.6 (15)
O3—S1—C1—C2	−17.95 (18)	C3—C4—C5—C6	1.0 (3)
O1—S1—C1—C2	−140.49 (16)	C4—C5—C6—C1	0.2 (3)
O2—S1—C1—C2	101.57 (16)	C2—C1—C6—C5	−1.1 (3)
O3—S1—C1—C6	164.09 (15)	S1—C1—C6—C5	176.88 (15)
O1—S1—C1—C6	41.55 (18)	C2—C3—C7—O5	−177.13 (18)
O2—S1—C1—C6	−76.39 (17)	C4—C3—C7—O5	3.7 (3)
C6—C1—C2—C3	0.9 (3)	C2—C3—C7—O4	3.2 (3)
S1—C1—C2—C3	−177.12 (14)	C4—C3—C7—O4	−175.97 (18)
C1—C2—C3—C4	0.3 (3)	C12—N1—C8—C9	55.3 (3)
C1—C2—C3—C7	−178.85 (17)	N1—C8—C9—C10	−55.7 (3)
C2—C3—C4—O6	178.52 (17)	C8—C9—C10—C11	56.0 (3)
C7—C3—C4—O6	−2.3 (3)	C9—C10—C11—C12	−55.3 (3)
C2—C3—C4—C5	−1.2 (3)	C8—N1—C12—C11	−54.9 (3)
C7—C3—C4—C5	177.93 (17)	C10—C11—C12—N1	54.5 (3)
O6—C4—C5—C6	−178.76 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H7*B*···O1	0.86 (1)	1.93 (1)	2.786 (2)	176 (2)
O7—H7*A*···O2^{i}	0.86 (1)	1.91 (1)	2.770 (2)	177 (2)
N1—H1*B*···O5	0.88 (3)	2.55 (2)	2.983 (3)	110.6 (18)
N1—H1*B*···O7^{ii}	0.88 (3)	2.16 (3)	2.996 (3)	157 (2)
N1—H1*A*···O2^{iii}	0.92 (3)	1.90 (3)	2.807 (3)	170 (2)
O6—H6···O5	0.84 (3)	1.82 (3)	2.597 (2)	153 (3)
O4—H4···O7^{ii}	0.85 (3)	1.75 (3)	2.601 (2)	179 (3)

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