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3-Aminopyridinium 4-hydroxy-3-iodonaphthalene-1-sulfonate dihydrate

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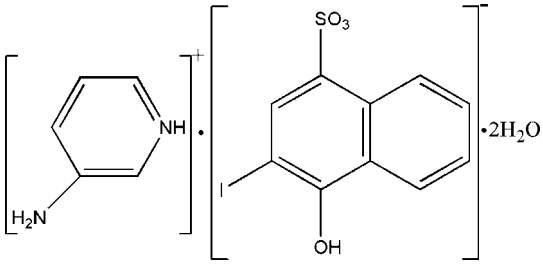
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.025; wR factor = 0.069; data-to-parameter ratio = 13.5.

In the hydrated title salt, $C_5H_7N_2^+\cdot C_{10}H_6IO_4S^-\cdot 2H_2O$, the component species are linked by O—H···O and N—H···O hydrogen bonds, forming an infinite three-dimensional framework.

Related literature

For background, see: Li (2007).



Experimental

Crystal data

$C_5H_7N_2^+\cdot C_{10}H_6IO_4S^-\cdot 2H_2O$
M_r = 480.27
Monoclinic, $P2_1/n$
a = 15.0219 (6) Å
b = 6.9917 (3) Å
c = 18.0729 (7) Å
β = 110.868 (1)°
V = 1773.66 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.96 mm^{-1}
T = 296 (2) K
0.18 × 0.12 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.720, T_{max} = 0.828
17926 measured reflections
3484 independent reflections
3128 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.069
S = 1.08
3484 reflections
258 parameters
34 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.96 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2W	0.882 (10)	1.853 (13)	2.725 (4)	170 (4)
N2—H2B···O1	0.886 (10)	2.232 (16)	3.085 (3)	162 (4)
N2—H2C···O2^{i}	0.888 (10)	2.179 (11)	3.064 (4)	175 (3)
O4—H4···O1W	0.843 (10)	1.89 (2)	2.655 (3)	150 (4)
O1W—H1WB···O3^{ii}	0.847 (10)	1.983 (12)	2.822 (3)	171 (4)
O1W—H1WA···O4^{iii}	0.851 (10)	2.158 (15)	2.944 (3)	153 (3)
O2W—H2WA···O1^{iv}	0.853 (10)	1.981 (12)	2.820 (3)	167 (3)
O2W—H2WB···O2^{v}	0.856 (10)	1.944 (11)	2.796 (3)	173 (3)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2719).

References

Bruker (2001). *SADABS*, *SAINT-Plus* and *SMART*. Bruker AXS, Inc., Madison, Wisconsin, USA.
Li, J. (2007). *Acta Cryst.* E**63**, o4171.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supporting information

Acta Cryst. (2008). E**64**, o1109 [doi:10.1107/S1600536808014098]

3-Aminopyridinium 4-hydroxy-3-iodonaphthalene-1-sulfonate dihydrate

Yun Liu and Jie Li

S1. Comment

This work continues our previous synthetic and structural studies of the hydrogen bonding interactions between various organic acids and substituted pyridines (Li, 2007).

The asymmetric unit of the title salt, (I), is composed of one 3-aminopyridinium cation, one 8-hydroxy-7-iodo-5-quinolinesulfonate anion and two water molecules (Fig. 1). One water molecule (O1W), acting as a hydrogen bonding donor interacts with the hydroxy oxygen (O4) acting as hydrogen bonding acceptor (Table 1). The other water molecule (O2W) and the 3-aminopyridinium cation are linked by a N1—H1A···O2W hydrogen bond. Moreover, the cation and anion are linked together by a N2—H2B···O1 hydrogen bond. Overall, an infinite three-dimensional framework results (Fig. 2).

S2. Experimental

A 5-ml ethanol solution of 3-aminopyridine (1.0 mmol, 0.094 g) was added to a 20-ml hot aqueous solution of 8-hydroxy-7-iodo-5-quinolinesulfonic acid (1.0 mmol, 0.351 g) and the mixture was stirred for 20 minutes at 373 K. Then the solution was filtered, and the filtrate was kept at the room temperature. After two weeks, yellow blocks of (I) were obtained.

S3. Refinement

The N- and O-bonded H atoms bonded were located in a difference synthesis and refined isotropically with N—H = 0.89 (1), O—H = 0.85 (1) and H···H = 1.34 (1) Å, respectively. All the remaining H atoms were placed in calculated positions, with C—H = 0.93Å and were refined as riding with U_{iso} = 1.2U_{eq}(C). This refinement scheme results in a short intramolecular H4···H6 contact of 1.71Å, although H4 participates in a plausible intermolecular hydrogen bond: thus the location of H4 should be regarded as less certain. Other placement schemes for H4 appear to lead to disordered hydrogen bond arrangements.

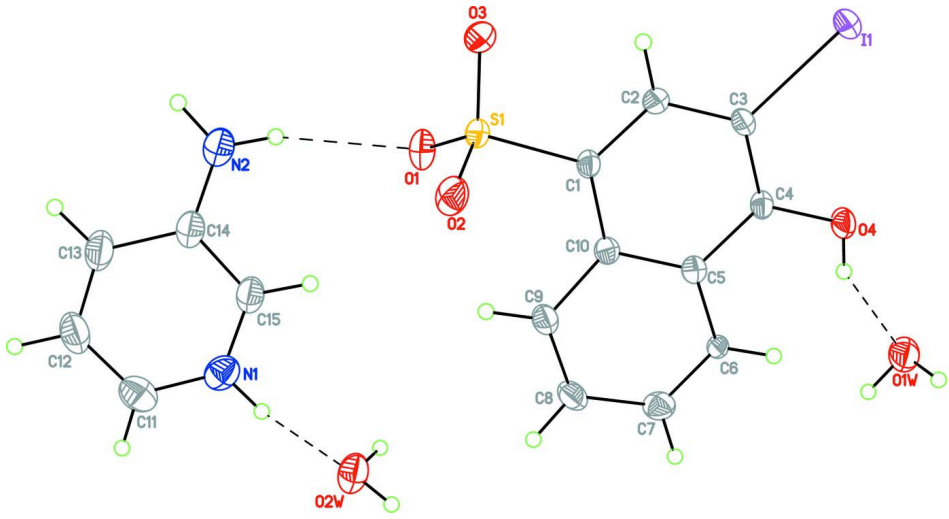


Figure 1

The molecular structure of (I). Displacement ellipsoids for the non-H atoms are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

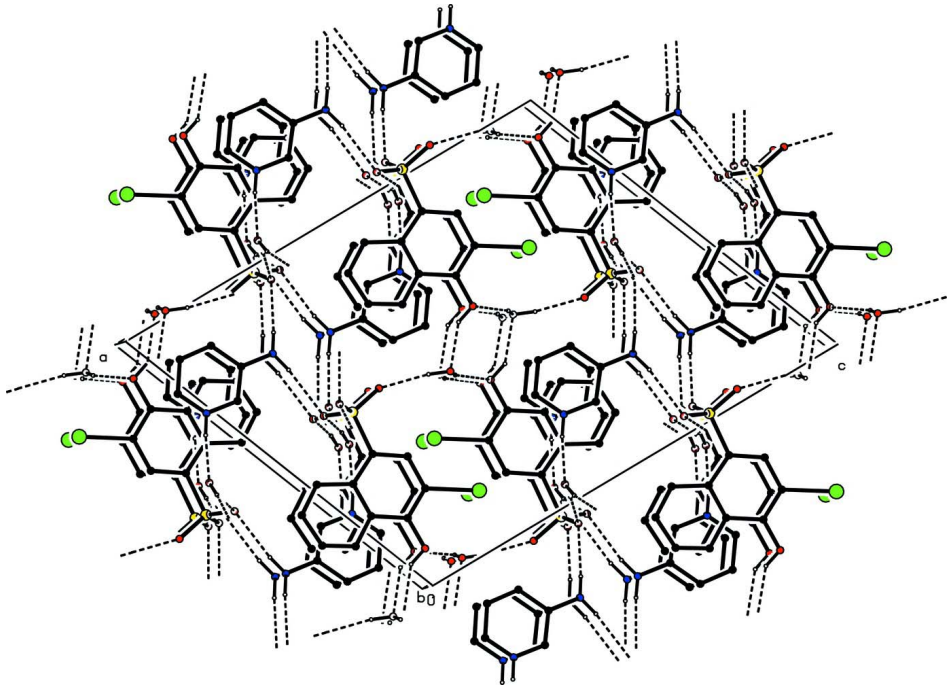


Figure 2

Part of the packing of (I) viewed along the direction [010]. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

3-Aminopyridinium 4-hydroxy-3-iodonaphthalene-1-sulfonate dihydrate

Crystal data

$C_5H_7N_2^+·C_{10}H_6IO_4S^-·2H_2O$
M_r = 480.27

Monoclinic, $P2_1/n$
Hall symbol: -P 2yn

a = 15.0219 (6) Å
b = 6.9917 (3) Å
c = 18.0729 (7) Å
β = 110.868 (1)°
V = 1773.66 (12) Å^3
Z = 4
F(000) = 952
D_x = 1.799 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9093 reflections
θ = 2.4–27.7°
μ = 1.96 mm^{-1}
T = 296 K
Block, yellow
0.18 × 0.12 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.720, T_{max} = 0.828

17926 measured reflections
3484 independent reflections
3128 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.0°, θ_{min} = 2.2°
h = −18→18
k = −8→8
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.069
S = 1.08
3484 reflections
258 parameters
34 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
$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 1.4355P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.96 e Å^{-3}

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}
N1	0.7208 (2)	0.1965 (4)	−0.01736 (18)	0.0486 (7)
H1A	0.6708 (18)	0.233 (5)	−0.0582 (16)	0.056 (11)*
N2	0.7670 (2)	0.0577 (5)	0.18478 (18)	0.0614 (8)
H2B	0.7069 (11)	0.034 (6)	0.179 (2)	0.065 (11)*
H2C	0.8150 (18)	0.008 (5)	0.2246 (15)	0.059 (10)*
C11	0.8061 (3)	0.1948 (5)	−0.0234 (2)	0.0564 (9)
H11A	0.8135	0.2277	−0.0708	0.068*
C12	0.8834 (2)	0.1434 (5)	0.0416 (2)	0.0577 (9)

H12A	0.9438	0.1405	0.0384	0.069*
C13	0.8718 (2)	0.0965 (5)	0.1112 (2)	0.0541 (8)
H13A	0.9245	0.0618	0.1550	0.065*
C14	0.7811 (2)	0.1006 (4)	0.11680 (19)	0.0433 (7)
C15	0.7058 (2)	0.1516 (4)	0.0493 (2)	0.0447 (7)
H15A	0.6443	0.1547	0.0502	0.054*
S1	0.51967 (4)	0.21097 (9)	0.19020 (4)	0.03081 (15)
I1	0.159514 (12)	0.20253 (3)	0.230170 (10)	0.03822 (8)
O1	0.54976 (13)	0.0659 (3)	0.14622 (13)	0.0469 (5)
O2	0.55842 (13)	0.3982 (3)	0.18257 (13)	0.0469 (5)
O3	0.53822 (15)	0.1580 (4)	0.27109 (13)	0.0570 (6)
O4	0.10004 (13)	0.2998 (3)	0.04865 (12)	0.0393 (5)
H4	0.080 (3)	0.315 (6)	−0.0008 (7)	0.071 (13)*
C1	0.39401 (17)	0.2327 (4)	0.14385 (15)	0.0273 (5)
C2	0.33925 (18)	0.2092 (3)	0.18943 (15)	0.0285 (5)
H2A	0.3682	0.1784	0.2427	0.034*
C3	0.24002 (18)	0.2308 (4)	0.15712 (15)	0.0281 (5)
C4	0.19533 (17)	0.2770 (3)	0.07873 (15)	0.0275 (5)
C5	0.25099 (18)	0.3043 (3)	0.03008 (15)	0.0275 (5)
C6	0.20225 (16)	0.3533 (3)	−0.04661 (13)	0.0230 (5)
H6	0.1364	0.3671	−0.0655	0.028*
C7	0.2508 (2)	0.3803 (4)	−0.09292 (16)	0.0399 (6)
H7	0.2179	0.4140	−0.1454	0.048*
C8	0.3502 (2)	0.3611 (5)	−0.06744 (17)	0.0423 (7)
H8	0.3818	0.3819	−0.1027	0.051*
C9	0.4003 (2)	0.3120 (4)	0.00897 (17)	0.0371 (6)
H9	0.4661	0.2985	0.0262	0.045*
C10	0.35128 (18)	0.2815 (3)	0.06225 (15)	0.0275 (5)
O1W	−0.02157 (17)	0.3572 (4)	−0.09765 (13)	0.0532 (6)
H1WA	−0.038 (2)	0.473 (2)	−0.095 (2)	0.060 (5)*
H1WB	0.002 (2)	0.359 (4)	−0.1337 (16)	0.056 (5)*
O2W	0.55451 (16)	0.2736 (3)	−0.13855 (17)	0.0555 (6)
H2WA	0.5162 (18)	0.179 (3)	−0.147 (2)	0.059 (11)*
H2WB	0.5167 (18)	0.370 (3)	−0.151 (2)	0.072 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0482 (16)	0.0385 (14)	0.0518 (16)	−0.0003 (12)	0.0091 (13)	0.0011 (12)
N2	0.0386 (16)	0.090 (2)	0.0548 (17)	0.0127 (16)	0.0150 (14)	0.0124 (16)
C11	0.063 (2)	0.0483 (19)	0.064 (2)	−0.0091 (16)	0.0302 (19)	0.0008 (16)
C12	0.0395 (17)	0.060 (2)	0.080 (3)	−0.0068 (15)	0.0282 (18)	0.0007 (19)
C13	0.0307 (15)	0.058 (2)	0.068 (2)	0.0012 (14)	0.0112 (15)	0.0044 (17)
C14	0.0321 (14)	0.0431 (16)	0.0531 (17)	0.0027 (12)	0.0129 (13)	−0.0010 (14)
C15	0.0316 (14)	0.0430 (16)	0.0580 (19)	0.0040 (12)	0.0142 (14)	0.0016 (14)
S1	0.0198 (3)	0.0380 (3)	0.0329 (3)	0.0003 (2)	0.0073 (2)	0.0048 (3)
I1	0.03151 (12)	0.05175 (14)	0.03694 (12)	0.00166 (7)	0.01898 (8)	0.00622 (8)
O1	0.0299 (10)	0.0489 (12)	0.0630 (13)	0.0051 (9)	0.0180 (9)	−0.0058 (10)

O2	0.0289 (10)	0.0424 (11)	0.0616 (13)	−0.0083 (9)	0.0066 (9)	0.0054 (10)
O3	0.0303 (11)	0.0986 (19)	0.0378 (11)	0.0063 (11)	0.0069 (9)	0.0226 (12)
O4	0.0214 (9)	0.0645 (14)	0.0313 (11)	0.0048 (8)	0.0084 (8)	0.0037 (9)
C1	0.0206 (11)	0.0297 (12)	0.0318 (13)	0.0004 (9)	0.0095 (10)	0.0015 (10)
C2	0.0258 (12)	0.0323 (13)	0.0261 (12)	0.0011 (10)	0.0078 (10)	0.0036 (10)
C3	0.0249 (12)	0.0336 (13)	0.0297 (12)	−0.0003 (10)	0.0144 (10)	0.0007 (10)
C4	0.0214 (12)	0.0318 (12)	0.0297 (12)	0.0013 (10)	0.0095 (10)	−0.0014 (10)
C5	0.0255 (12)	0.0278 (12)	0.0296 (12)	−0.0006 (9)	0.0101 (10)	−0.0019 (9)
C6	0.0162 (10)	0.0326 (12)	0.0178 (10)	0.0005 (9)	0.0030 (8)	0.0016 (9)
C7	0.0390 (15)	0.0479 (16)	0.0289 (13)	−0.0033 (13)	0.0075 (11)	0.0030 (12)
C8	0.0375 (15)	0.0600 (18)	0.0342 (14)	−0.0037 (14)	0.0188 (12)	0.0049 (13)
C9	0.0274 (13)	0.0495 (16)	0.0362 (14)	−0.0016 (12)	0.0135 (12)	0.0028 (12)
C10	0.0248 (12)	0.0285 (12)	0.0288 (12)	−0.0015 (9)	0.0092 (10)	−0.0006 (10)
O1W	0.0462 (12)	0.0741 (16)	0.0393 (12)	0.0129 (12)	0.0151 (10)	0.0012 (11)
O2W	0.0377 (12)	0.0424 (13)	0.0792 (18)	0.0006 (10)	0.0122 (12)	−0.0025 (12)

Geometric parameters (Å, º)

N1—C11	1.325 (5)	C1—C2	1.366 (4)
N1—C15	1.339 (4)	C1—C10	1.424 (4)
N1—H1A	0.882 (10)	C2—C3	1.402 (4)
N2—C14	1.353 (4)	C2—H2A	0.9300
N2—H2B	0.886 (10)	C3—C4	1.373 (4)
N2—H2C	0.888 (10)	C4—C5	1.425 (4)
C11—C12	1.374 (5)	C5—C6	1.361 (3)
C11—H11A	0.9300	C5—C10	1.417 (4)
C12—C13	1.369 (5)	C6—C7	1.305 (4)
C12—H12A	0.9300	C6—H6	0.9300
C13—C14	1.403 (4)	C7—C8	1.404 (4)
C13—H13A	0.9300	C7—H7	0.9300
C14—C15	1.382 (4)	C8—C9	1.360 (4)
C15—H15A	0.9300	C8—H8	0.9300
S1—O3	1.436 (2)	C9—C10	1.421 (4)
S1—O1	1.456 (2)	C9—H9	0.9300
S1—O2	1.459 (2)	O1W—H1WA	0.851 (10)
S1—C1	1.778 (2)	O1W—H1WB	0.847 (10)
I1—C3	2.093 (2)	O2W—H2WA	0.853 (10)
O4—C4	1.347 (3)	O2W—H2WB	0.856 (10)
O4—H4	0.843 (10)		
C11—N1—C15	123.4 (3)	C10—C1—S1	121.29 (19)
C11—N1—H1A	120 (3)	C1—C2—C3	121.0 (2)
C15—N1—H1A	117 (3)	C1—C2—H2A	119.5
C14—N2—H2B	115 (2)	C3—C2—H2A	119.5
C14—N2—H2C	119 (2)	C4—C3—C2	120.8 (2)
H2B—N2—H2C	122 (4)	C4—C3—I1	119.55 (18)
N1—C11—C12	118.6 (4)	C2—C3—I1	119.64 (19)
N1—C11—H11A	120.7	O4—C4—C3	120.2 (2)

C12—C11—H11A	120.7	O4—C4—C5	120.5 (2)
C13—C12—C11	120.3 (3)	C3—C4—C5	119.3 (2)
C13—C12—H12A	119.9	C6—C5—C10	123.6 (2)
C11—C12—H12A	119.9	C6—C5—C4	116.2 (2)
C12—C13—C14	120.4 (3)	C10—C5—C4	120.2 (2)
C12—C13—H13A	119.8	C7—C6—C5	118.0 (2)
C14—C13—H13A	119.8	C7—C6—H6	121.0
N2—C14—C15	121.0 (3)	C5—C6—H6	121.0
N2—C14—C13	122.1 (3)	C6—C7—C8	123.2 (3)
C15—C14—C13	116.9 (3)	C6—C7—H7	118.4
N1—C15—C14	120.5 (3)	C8—C7—H7	118.4
N1—C15—H15A	119.8	C9—C8—C7	119.7 (3)
C14—C15—H15A	119.8	C9—C8—H8	120.1
O3—S1—O1	113.00 (15)	C7—C8—H8	120.1
O3—S1—O2	112.85 (15)	C8—C9—C10	119.6 (3)
O1—S1—O2	111.19 (13)	C8—C9—H9	120.2
O3—S1—C1	106.86 (12)	C10—C9—H9	120.2
O1—S1—C1	106.64 (12)	C5—C10—C9	115.9 (2)
O2—S1—C1	105.73 (12)	C5—C10—C1	118.2 (2)
C4—O4—H4	111 (3)	C9—C10—C1	125.9 (2)
C2—C1—C10	120.5 (2)	H1WA—O1W—H1WB	103.8 (15)
C2—C1—S1	118.17 (19)	H2WA—O2W—H2WB	102.6 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2*W*	0.88 (1)	1.85 (1)	2.725 (4)	170 (4)
N2—H2*B*···O1	0.89 (1)	2.23 (2)	3.085 (3)	162 (4)
N2—H2*C*···O2^{i}	0.89 (1)	2.18 (1)	3.064 (4)	175 (3)
O4—H4···O1*W*	0.84 (1)	1.89 (2)	2.655 (3)	150 (4)
O1*W*—H1*WB*···O3^{ii}	0.85 (1)	1.98 (1)	2.822 (3)	171 (4)
O1*W*—H1*WA*···O4^{iii}	0.85 (1)	2.16 (2)	2.944 (3)	153 (3)
O2*W*—H2*WA*···O1^{iv}	0.85 (1)	1.98 (1)	2.820 (3)	167 (3)
O2*W*—H2*WB*···O2^{v}	0.86 (1)	1.94 (1)	2.796 (3)	173 (3)

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