Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1*H*-1,2,4-Triazol-4-ium 4-nitrobenzenesulfonate monohydrate

Madhukar Hemamalini, Ibrahim Abdul Razak‡ and Hoong-Kun Fun*§

X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: hkfun@usm.my

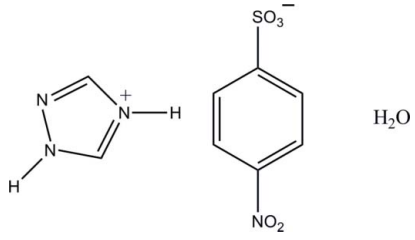
Received 6 September 2011; accepted 10 September 2011

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.144; data-to-parameter ratio = 14.3.

In the 4-nitrobenzene sulfonate anion of the title compound, $C_2H_4N_3^+\cdot C_6H_4NO_5S^-\cdot H_2O$, the nitro group is slightly twisted from the plane of the benzene ring [dihedral angle = 2.8 (3)°]. In the crystal, the three components are linked *via* N—H···O, O—H···N, O—H···O and C—H···O hydrogen bonds, forming a two-dimensional network parallel to the *bc* plane. A short intermolecular O···N contact of 2.872 (3) Å is also observed between the nitro and sulfonate groups.

Related literature

For details and applications of aromatic sulfonates, see: Yachi *et al.* (1989); Spungin *et al.* (1992); Jiang *et al.* (1990); Narayanan & Krakow (1983).



Experimental

Crystal data

$C_2H_4N_3^+\cdot C_6H_4NO_5S^-\cdot H_2O$
M_r = 290.26
Monoclinic, $P2_1/c$
a = 14.0931 (13) Å
b = 6.4859 (6) Å
c = 14.5707 (14) Å
β = 117.182 (2)°
V = 1184.77 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 296 K
0.41 × 0.28 × 0.05 mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.885, T_{max} = 0.986
10925 measured reflections
2692 independent reflections
2136 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.144
S = 1.07
2692 reflections
188 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H1*NA*···O5^{i}	0.88 (4)	1.88 (4)	2.744 (3)	169 (2)
O1*W*—H1*W*···N3	0.91 (4)	2.17 (4)	3.041 (3)	160 (4)
N2—H1*NB*···O1*W*^{ii}	0.86 (4)	1.84 (4)	2.692 (3)	171 (3)
O1*W*—H2*W*···O3^{iii}	0.95 (4)	1.86 (4)	2.774 (3)	161 (5)
C7—H7*A*···O4^{iv}	0.93	2.36	3.063 (3)	132
C8—H8*A*···O1	0.93	2.54	3.186 (4)	126

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

MH, HKF and IAR thank the Ministry of Higher Education, Malaysia and Universiti Sains Malaysia for the Fundamental Research Grant Scheme (FRGS) grant No. 203/PFIZIK/6711171. MH also thanks Universiti Sains Malaysia for a post-doctoral research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2774).

References

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Jiang, F. N., Jiang, S., Liu, D., Richter, A. & Levy, J. G. (1990). *J. Immunol. Methods*, **134**, 139–149.
Narayanan, C. S. & Krakow, J. S. (1983). *Nucleic Acids Res.* **11**, 2701–2716.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Spungin, B., Levinshal, T., Rubenstein, S. & Breitbart, H. (1992). *FEBS Lett.* **311**, 155–160.
Yachi, K., Sugiyama, Y., Sawada, Y., Iga, T., Ikeda, Y., Toda, G. & Hanano, M. (1989). *Biochim. Biophys. Acta*, **978**, 1–7.

‡ Thomson Reuters ResearcherID: A-5599-2009.
§ Thomson Reuters ResearcherID: A-3561-2009.

supporting information

Acta Cryst. (2011). E**67**, o2655 [https://doi.org/10.1107/S1600536811036774]

1*H*-1,2,4-Triazol-4-ium 4-nitrobenzenesulfonate monohydrate

Madhukar Hemamalini, Ibrahim Abdul Razak and Hoong-Kun Fun

S1. Comment

In recent years, there has been of great interest in the design and utilization of 1,2,4-triazole and its derivatives in coordination and biological chemistry for they represent the simple small molecular ligands. Aromatic sulfonates are used in monitoring the merging of lipids (Yachi *et al.*, 1989) and in many other fields (Spungin *et al.*, 1992; Jiang *et al.*, 1990; Narayanan & Krakow, 1983). An X-ray study of the title compound was undertaken in order to determine its crystal and molecular structure owing to the biological importance of its analogues. The molecular structure of the title compound (I).

The asymmetric unit of the title compound, (Fig. 1), contains a protonated 1,2,4-triazolinium cation, a 4-nitrobenzenesulfonate anion and a water molecule. In the 4-nitrobenzenesulfonate anion, the nitro and sulfonate groups are twisted slightly from the ring to which they are attached with the dihedral angles between the O1/O2/N1 and C1–C6 planes, and the S1/O3/O5 and C1–C6 planes being 2.8 (3) and 88.85 (13)°, respectively.

In the crystal structure, (Fig. 2), the ion pairs and water molecules are linked *via* intermolecular N—H···O, O—H···N, O—H···O and C—H···O hydrogen bonds (Table 1), forming two-dimensional networks parallel to (100). A short O···N contact of 2.87 Å is also observed.

S2. Experimental

A methanol solution (20 ml) of 1-(*p*-Nitrobenzenesulfonayl)-1*H*-1,2,4-triazole (63.55 mg, Aldrich) was warmed over a heating magnetic stirrer for 15 minutes. The resulting solution was allowed to cool slowly at room temperature. Crystals of the title compound appeared from the mother liquor after a few days.

S3. Refinement

Atoms H1NA and H1NB were located in a difference Fourier map and refined freely [N—H = 0.86 (3)–0.87 (3) Å]. Atoms H1W and H2W were also located in a difference map and were refined with restraints of bond lengths and angles [O—H = 0.917 (18)–0.950 (18) Å and H2W—O1W—H1W = 110 (3)°]. The remaining H atoms were positioned geometrically (C—H = 0.93 Å) and were refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C).

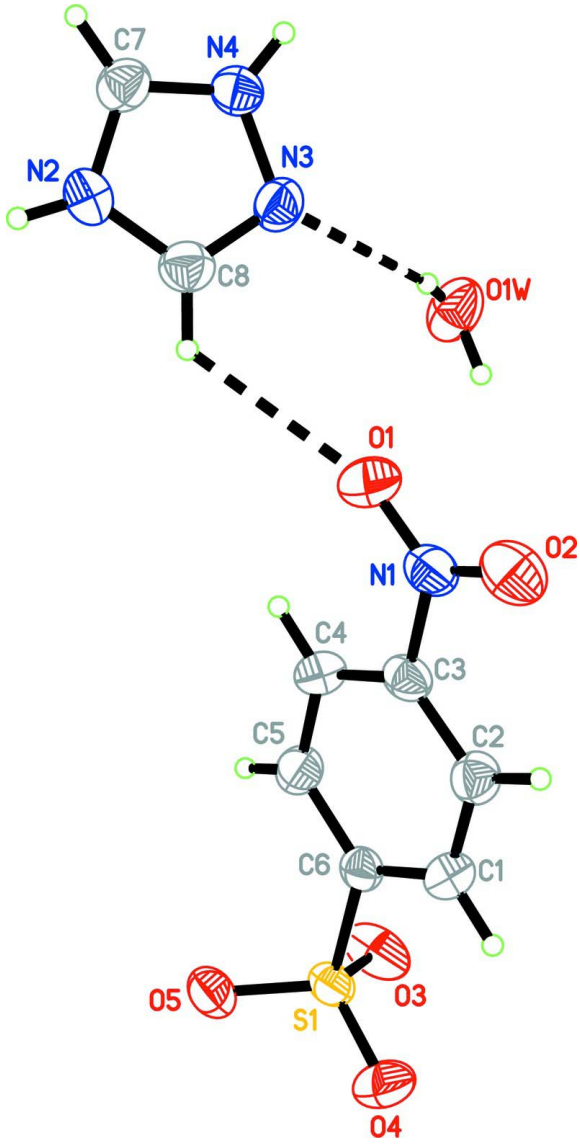


Figure 1

The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids. Intermolecular O—H···N and C—H···O hydrogen bonds are shown by dashed lines.

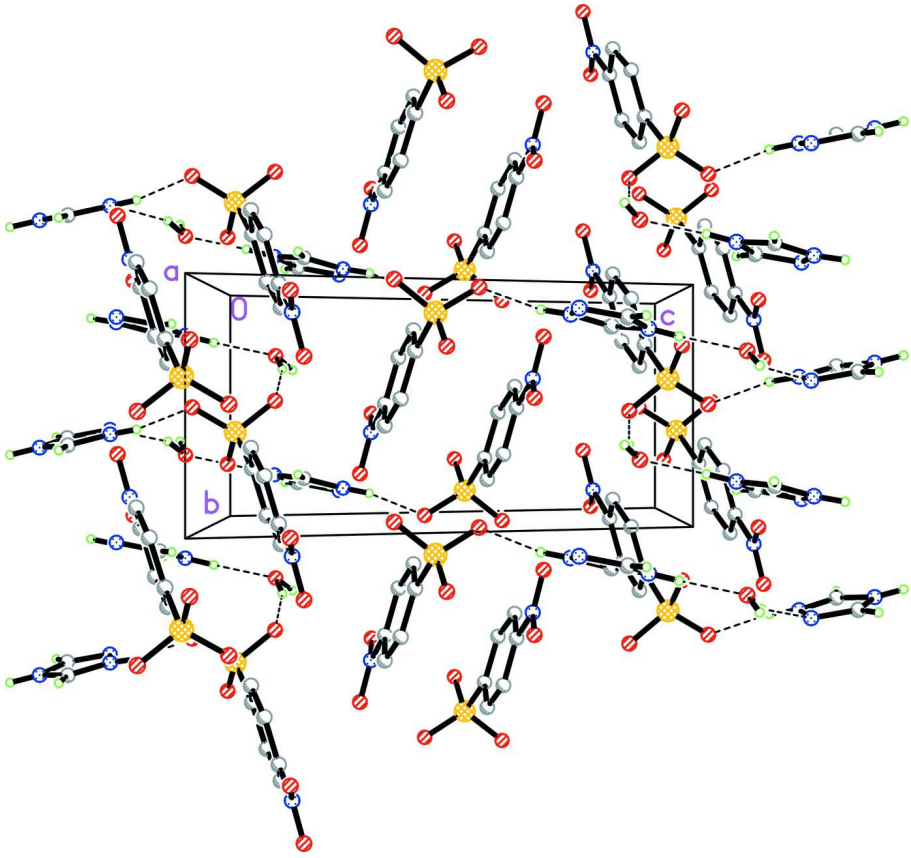


Figure 2

The crystal packing of the title compound. Dashed lines represent hydrogen bonds.

1*H*-1,2,4-Triazol-4-ium 4-nitrobenzenesulfonate monohydrate

Crystal data

$C_2H_4N_3^+·C_6H_4NO_5S^-·H_2O$
M_r = 290.26
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 14.0931 (13) Å
b = 6.4859 (6) Å
c = 14.5707 (14) Å
β = 117.182 (2)°
V = 1184.77 (19) Å^3
Z = 4
$F(000)$ = 600
D_x = 1.627 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3747 reflections
θ = 2.8–30.9°
μ = 0.31 mm^{-1}
T = 296 K
Block, colourless
0.41 × 0.28 × 0.05 mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.885, T_{max} = 0.986
10925 measured reflections
2692 independent reflections
2136 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.5°, θ_{min} = 1.6°
h = −18→16
k = −8→8
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.144$
$S = 1.07$
2692 reflections
188 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
$w = 1/[\sigma^2(F_o^2) + (0.0862P)^2 + 0.2559P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
O1W	0.09777 (16)	0.2748 (3)	0.10622 (14)	0.0521 (5)
H2W	0.165 (2)	0.336 (7)	0.125 (4)	0.126 (16)*
H1W	0.066 (3)	0.338 (7)	0.141 (3)	0.130 (18)*
O3	0.72455 (14)	−0.0217 (3)	0.38351 (14)	0.0535 (5)
O4	0.83055 (14)	0.2569 (3)	0.49177 (16)	0.0520 (5)
O5	0.75198 (13)	0.0065 (3)	0.55875 (13)	0.0441 (4)
N1	0.34545 (15)	0.6163 (3)	0.32475 (15)	0.0384 (5)
C1	0.62491 (17)	0.4703 (4)	0.39475 (18)	0.0357 (5)
H1A	0.6865	0.5296	0.3982	0.043*
C2	0.53180 (18)	0.5842 (3)	0.35933 (18)	0.0360 (5)
H2A	0.5293	0.7195	0.3373	0.043*
C3	0.44280 (16)	0.4921 (3)	0.35755 (16)	0.0313 (5)
C4	0.44120 (17)	0.2906 (4)	0.38632 (18)	0.0365 (5)
H4A	0.3795	0.2324	0.3832	0.044*
C5	0.53449 (17)	0.1771 (4)	0.42016 (18)	0.0355 (5)
H5A	0.5359	0.0402	0.4397	0.043*
C6	0.62590 (16)	0.2680 (3)	0.42492 (15)	0.0293 (4)
S1	0.74353 (4)	0.11570 (9)	0.46843 (4)	0.03281 (19)
O1	0.26690 (14)	0.5340 (3)	0.32361 (16)	0.0553 (5)
O2	0.34762 (14)	0.7968 (3)	0.30119 (15)	0.0513 (5)
C7	−0.08790 (19)	0.3289 (4)	0.33222 (18)	0.0377 (5)
H7A	−0.1481	0.3087	0.3418	0.045*
C8	0.07316 (18)	0.3574 (4)	0.35568 (19)	0.0386 (5)
H8A	0.1473	0.3593	0.3885	0.046*

N2	0.01283 (16)	0.3160 (3)	0.40391 (16)	0.0372 (4)
H1NA	−0.141 (3)	0.397 (4)	0.185 (3)	0.057 (9)*
N3	0.01514 (15)	0.3939 (3)	0.25866 (15)	0.0378 (5)
N4	−0.08644 (15)	0.3754 (3)	0.24581 (16)	0.0356 (4)
H1NB	0.033 (2)	0.284 (4)	0.467 (3)	0.056 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1W	0.0560 (12)	0.0645 (12)	0.0418 (10)	−0.0220 (9)	0.0277 (9)	−0.0133 (9)
O3	0.0481 (10)	0.0691 (12)	0.0361 (9)	0.0226 (9)	0.0128 (8)	−0.0110 (9)
O4	0.0309 (9)	0.0637 (12)	0.0627 (12)	−0.0006 (7)	0.0225 (8)	0.0041 (9)
O5	0.0381 (9)	0.0573 (10)	0.0332 (9)	0.0099 (7)	0.0131 (7)	0.0110 (8)
N1	0.0338 (10)	0.0511 (12)	0.0294 (10)	0.0095 (8)	0.0136 (8)	0.0016 (9)
C1	0.0303 (10)	0.0394 (11)	0.0397 (12)	−0.0027 (9)	0.0179 (9)	0.0001 (10)
C2	0.0378 (12)	0.0345 (11)	0.0376 (12)	0.0023 (9)	0.0188 (9)	0.0036 (9)
C3	0.0289 (10)	0.0382 (11)	0.0250 (10)	0.0054 (8)	0.0108 (8)	−0.0016 (9)
C4	0.0273 (10)	0.0432 (12)	0.0400 (13)	−0.0006 (9)	0.0163 (9)	0.0011 (10)
C5	0.0345 (11)	0.0352 (11)	0.0397 (12)	0.0022 (9)	0.0195 (10)	0.0049 (10)
C6	0.0284 (10)	0.0374 (11)	0.0229 (10)	0.0026 (8)	0.0126 (8)	−0.0001 (8)
S1	0.0276 (3)	0.0439 (3)	0.0256 (3)	0.0065 (2)	0.0110 (2)	0.0010 (2)
O1	0.0336 (9)	0.0730 (12)	0.0644 (13)	0.0111 (8)	0.0267 (8)	0.0157 (10)
O2	0.0509 (11)	0.0436 (10)	0.0545 (12)	0.0141 (8)	0.0199 (9)	0.0063 (9)
C7	0.0367 (12)	0.0416 (12)	0.0370 (12)	−0.0036 (10)	0.0188 (10)	−0.0047 (10)
C8	0.0335 (11)	0.0392 (12)	0.0407 (13)	−0.0020 (9)	0.0149 (10)	−0.0004 (10)
N2	0.0414 (11)	0.0373 (10)	0.0298 (11)	0.0010 (8)	0.0136 (8)	0.0021 (8)
N3	0.0375 (10)	0.0408 (10)	0.0369 (11)	−0.0075 (8)	0.0186 (8)	0.0011 (8)
N4	0.0312 (9)	0.0402 (10)	0.0309 (10)	−0.0045 (8)	0.0102 (8)	−0.0015 (8)

Geometric parameters (Å, º)

O1W—H2W	0.950 (18)	C4—C5	1.386 (3)
O1W—H1W	0.917 (18)	C4—H4A	0.9300
O3—S1	1.4472 (18)	C5—C6	1.389 (3)
O4—S1	1.4411 (18)	C5—H5A	0.9300
O5—S1	1.4508 (18)	C6—S1	1.779 (2)
N1—O1	1.222 (3)	C7—N4	1.304 (3)
N1—O2	1.224 (3)	C7—N2	1.326 (3)
N1—C3	1.470 (3)	C7—H7A	0.9300
C1—C6	1.382 (3)	C8—N3	1.291 (3)
C1—C2	1.384 (3)	C8—N2	1.355 (3)
C1—H1A	0.9300	C8—H8A	0.9300
C2—C3	1.379 (3)	N2—H1NB	0.86 (3)
C2—H2A	0.9300	N3—N4	1.362 (3)
C3—C4	1.376 (3)	N4—H1NA	0.87 (3)
H2W—O1W—H1W	110 (3)	C5—C6—S1	118.29 (16)
O1—N1—O2	123.5 (2)	O4—S1—O3	113.45 (12)

O1—N1—C3	118.0 (2)	O4—S1—O5	112.82 (11)
O2—N1—C3	118.42 (19)	O3—S1—O5	112.42 (12)
C6—C1—C2	119.7 (2)	O4—S1—C6	106.58 (10)
C6—C1—H1A	120.1	O3—S1—C6	105.01 (10)
C2—C1—H1A	120.1	O5—S1—C6	105.75 (10)
C3—C2—C1	118.4 (2)	N4—C7—N2	107.0 (2)
C3—C2—H2A	120.8	N4—C7—H7A	126.5
C1—C2—H2A	120.8	N2—C7—H7A	126.5
C4—C3—C2	123.16 (19)	N3—C8—N2	111.8 (2)
C4—C3—N1	118.48 (19)	N3—C8—H8A	124.1
C2—C3—N1	118.35 (19)	N2—C8—H8A	124.1
C3—C4—C5	117.9 (2)	C7—N2—C8	106.2 (2)
C3—C4—H4A	121.0	C7—N2—H1NB	125 (2)
C5—C4—H4A	121.0	C8—N2—H1NB	129 (2)
C4—C5—C6	120.0 (2)	C8—N3—N4	103.57 (19)
C4—C5—H5A	120.0	C7—N4—N3	111.5 (2)
C6—C5—H5A	120.0	C7—N4—H1NA	128 (2)
C1—C6—C5	120.84 (19)	N3—N4—H1NA	121 (2)
C1—C6—S1	120.85 (16)		

C6—C1—C2—C3	1.4 (3)	C4—C5—C6—S1	−179.78 (17)
C1—C2—C3—C4	−2.0 (3)	C1—C6—S1—O4	16.1 (2)
C1—C2—C3—N1	177.05 (19)	C5—C6—S1—O4	−165.17 (17)
O1—N1—C3—C4	−0.7 (3)	C1—C6—S1—O3	−104.6 (2)
O2—N1—C3—C4	178.5 (2)	C5—C6—S1—O3	74.2 (2)
O1—N1—C3—C2	−179.8 (2)	C1—C6—S1—O5	136.35 (19)
O2—N1—C3—C2	−0.6 (3)	C5—C6—S1—O5	−44.87 (19)
C2—C3—C4—C5	1.1 (3)	N4—C7—N2—C8	0.1 (3)
N1—C3—C4—C5	−178.02 (19)	N3—C8—N2—C7	−0.3 (3)
C3—C4—C5—C6	0.5 (3)	N2—C8—N3—N4	0.3 (3)
C2—C1—C6—C5	0.0 (3)	N2—C7—N4—N3	0.0 (3)
C2—C1—C6—S1	178.76 (17)	C8—N3—N4—C7	−0.2 (2)
C4—C5—C6—C1	−1.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1*NA*···O5^{i}	0.88 (4)	1.88 (4)	2.744 (3)	169 (2)
O1*W*—H1*W*···N3	0.91 (4)	2.17 (4)	3.041 (3)	160 (4)
N2—H1*NB*···O1*W*^{ii}	0.86 (4)	1.84 (4)	2.692 (3)	171 (3)
O1*W*—H2*W*···O3^{iii}	0.95 (4)	1.86 (4)	2.774 (3)	161 (5)
C7—H7*A*···O4^{iv}	0.93	2.36	3.063 (3)	132
C8—H8*A*···O1	0.93	2.54	3.186 (4)	126

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