Acta Cryst E
CRYSTALLOGRAPHIC
COMMUNICATIONS

ISSN 2056-9890

Crystal structure of 2-amino-5-nitropyridinium sulfamate

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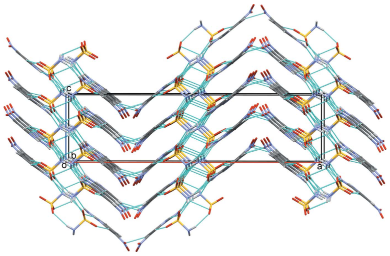
Received 15 December 2014
Accepted 8 January 2015

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

Keywords: crystal structure; sulfamic acid; 2-amino-5-nitropyridine; sulfamate; 2-amino-5-pyridinium; molecular salt; hydrogen bonding.

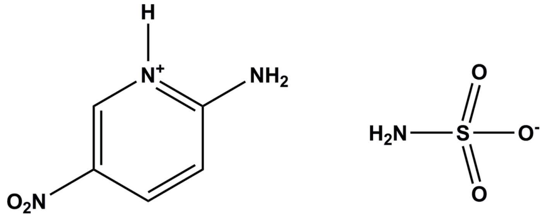
CCDC reference: 1042506
Supporting information: this article has supporting information at journals.iucr.org/e

The title molecular salt, $C_5H_6N_3O_2^+ \cdot H_2NO_3S^-$, was obtained from the reaction of sulfamic acid with 2-amino-5-nitropyridine. A proton transfer from sulfamic acid to the pyridine N atom occurred, resulting in the formation of a salt. As expected, this protonation leads to the widening of the C—N—C angle of the pyridine ring, to 122.9 (3)°, with the pyridinium ring being essentially planar (r.m.s. deviation = 0.025 Å). In the crystal, the ion pairs are joined by three N—H⋯O and one N—H⋯N hydrogen bonds in which the pyridinium N atom and the amino N atom act as donors, and are hydrogen bonded to the carboxylate O atoms and the N atom of the sulfamate anion, thus generating an $R_3^3(22)$ ring motif. These motifs are linked by further N—H⋯O hydrogen bonds enclosing $R_3^3(8)$ loops, forming sheets parallel to (100). The sheets are linked *via* weak C—H⋯O hydrogen bonds, forming a three-dimensional structure. The O atoms of the nitro group are disordered over two sets of sites with a refined occupancy ratio of 0.737 (19):0.263 (19).



1. Chemical context

Pyridine heterocycles and their derivatives are present in many large molecules having photo-chemical, electrochemical and catalytic applications. Some pyridine derivatives possess non-linear optical (NLO) properties (Babu *et al.*, 2014*a*,*b*). Simple organic–inorganic salts containing strong intermolecular hydrogen bonds have attracted attention as materials which display ferroelectric–paraelectric phase transitions (Sethuram, *et al.*, 2013*a*,*b*; Huq *et al.*, 2013; Shihabuddeen Syed *et al.*, 2013; Showrilu *et al.*, 2013). We have recently reported the crystal structures of 2-amino-6-methylpyridinium 2,2,2-trichloroacetate (Babu *et al.*, 2014*a*), 2-amino-6-methylpyridinium 4-methylbenzenesulfonate (Babu *et al.*, 2014*b*) and 2-amino-5-nitropyridinium hydrogen oxalate (Rajkumar *et al.*, 2014). In a continuation of our studies of pyridinium salts, we report herein on the crystal structure of the title molecular salt, obtained by the reaction of 2-amino-5-nitropyridine with sulfamic acid.



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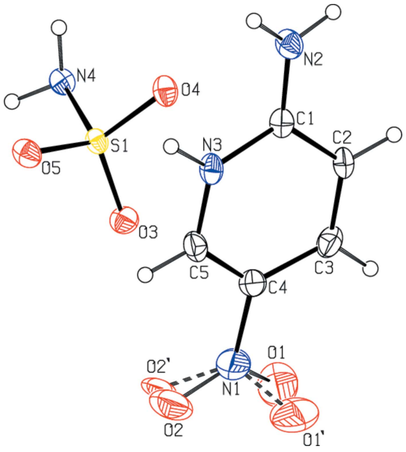

Figure 1
View of the molecular structure of the title molecular salt, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

2. Structural commentary

The asymmetric unit of the title compound, Fig. 1, consists of a 2-amino-5-nitropyridin-1-ium cation and a sulfamate anion. The bond lengths and angles are within normal ranges and comparable with those in closely related structures (Babu *et al.*, 2014*a*,*b*; Rajkumar *et al.*, 2014). A proton transfer from the sulfamic acid to the pyridine atom N3 resulted in the formation of a salt. This protonation leads to the widening of the C5—N3—C1 angle of the pyridine ring to 122.9 (3)°, compared with 115.25 (13)° in unprotonated aminopyridine (Anderson *et al.*, 2005). This type of protonation is observed in various aminopyridine acid complexes (Babu *et al.*, 2014*a*,*b*; Rajkumar *et al.*, 2014). In the sulfamate anion the S—O distances vary from 1.440 (3) to 1.460 (2) Å, and O—S—O angles vary from 111.59 (15) to 114.22 (15) °.

In the cation, the N2—C1 [1.317 (5) Å] bond is shorter than the N3—C1 [1.357 (4) Å] and N3—C5 [1.340 (5) Å] bonds, and the C1—C2 [1.411 (5) Å] and C3—C4 [1.402 (6) Å] bonds lengths are significantly longer than bonds C2—C3 [1.348 (5) Å] and C4—C5 [1.338 (6) Å], similar to those observed previously for the aminopyridinium cation (Babu *et al.*, 2014*a*,*b*; Rajkumar *et al.*, 2014). In contrast, in the solid-state structure of aminopyridinium, the C—N(H_2) bond is clearly longer than that in the ring (Nahringbauer & Kvick, 1977). The geometrical features of the aminopyridinium cation (N1/N3/C1–C5) resemble those observed in other 2-aminopyridinium structures (Babu *et al.*, 2014*a*,*b*; Rajkumar *et al.*, 2014) that are believed to be involved in amine–imine tautomerism (Ishikawa *et al.*, 2002). However, previous studies have shown that a pyridinium cation always possesses an expanded C—N—C angle in comparison with pyridine itself (Jin *et al.*, 2005).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O4^{i}	0.88 (2)	1.98 (2)	2.861 (4)	176 (4)
N2—H2*A*···N4^{ii}	0.88 (2)	2.18 (2)	3.044 (4)	169 (4)
N3—H3*A*···O5^{iii}	0.89 (2)	1.91 (2)	2.766 (4)	163 (4)
N4—H4*B*···O4^{iv}	0.89 (2)	2.20 (2)	3.073 (4)	166 (3)
N4—H4*A*···O5^{v}	0.89 (2)	2.20 (2)	2.960 (4)	143 (3)
C2—H2···O3^{i}	0.93	2.57	3.469 (4)	163
C3—H3···O2^{vi}	0.93	2.46	3.328 (13)	155
C5—H5···O3^{iii}	0.93	2.41	3.187 (4)	141

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

In this atomic arrangement, one can distinguish the inter-cation-to-anion contact C5—H5···O3 (H5···O5 = 2.41 Å), which induces the aggregation of the independent organic cation 2-amino-5-nitropyridinium. This kind of arrangement is also observed in the related structure of 2-amino-5-nitropyridinium hydrogen selenate (Akriche & Rzaigui, 2009). These pairs are located between the anionic layers to link them by various interactions. The geometric features of the organic cation are usual and comparable with values observed for other 2-amino nitropyridinium compounds (Akriche & Rzaigui, 2009). It is worth noticing that the C—NH_2 [1.317 (5) Å] and C—NO_2 [1.449 (6) Å] distances in the cations are, respectively, shortened and lengthened with respect to the same bond lengths [1.337 (4) and 1.429 (4) Å] observed for 2-amino-nitropyridine (Aakeroy *et al.*, 1998). All the 2-amino-nitropyridinium cations encapsulated in various anionic sub-networks show the same changes in the C—NH_2 and C—NO_2 distances, revealing a weak increase of π bond character in the bond C—NH_2 and a decrease in the bond C—NO_2.

3. Supramolecular features

In the crystal, the ion pairs are linked by the N—H···O and N—H···N hydrogen bonds (Table 1 and Fig. 2). The protonated atom (N3) and the 2-amino group (N2) of the cation are hydrogen bonded to the carboxylate oxygen atoms (O5 and O4) and the nitrogen atom (N4) of the sulfamate anion *via* a pair of N—H···O and N—H···N (N3—H3*A*···O5, N2—H2*B*···O4 and N2—H2*A*···N4) hydrogen bonds (Table 1),

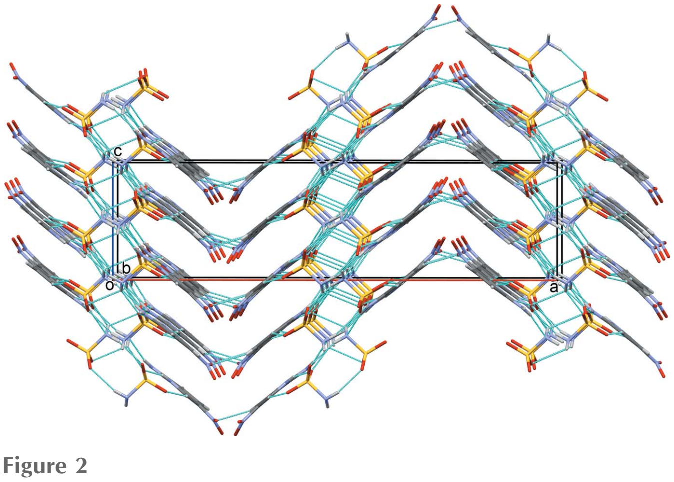

Figure 2
The crystal packing of the title salt, viewed along the *b* axis. The hydrogen bonds are shown as dashed lines (see Table 1 for details; only the major components of the disordered nitro O atoms are shown).

forming an $R_3^3(22)$ring motif. These motifs are further linked by N—H⋯O hydrogen bonds, enclosing $R_3^3(8)$ loops, and forming sheets lying parallel to (100). Weak C—H⋯O hydrogen bonds link the sheets, forming a three-dimensional structure (Fig. 2 and Table 1). The identification of such supramolecular patterns will help us design and construct preferred hydrogen-bonding patterns of drug-like molecules.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.35, May 2014; Groom & Allen, 2014) for the cation 2-amino-5-nitropyridinium gave 42 hits for which there were 36 hits with atomic coordinates present. For these structures, the average C—N—C bond angle is *ca* 123°, while the average C—N(H$_2$) and C—N(O$_2$) bond lengths are *ca* 1.32 and 1.45 Å, respectively. A search for the anion aminosulfamate gave 23 hits but only 17 contained atomic coordinates. Here the S—O bond lengths vary from *ca* 1.399 to 1.469 Å, while the N—S bond length varies from *ca* 1.63 to 1.80 Å. The bond lengths and angles in the title salt are very similar to those reported for the various structures in the CSD.

5. Synthesis and crystallization

The starting material 2-amino-5-nitropyridine was obtained by treating 3-nitropyridine with ammonia in the presence of KMnO$_4$. Colourless block-like crystals of the title salt were obtained by slow evaporation of a 1:1 equimolar mixture of 2-amino-5-nitropyridine and sulfamic acid in methanol at room temperature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The N-bound H atoms were located in a difference Fourier map and refined with distance restraints: N—H = 0.89 (2) Å. The C-bound H atoms were positioned geometrically and refined using a riding model: C—H = 0.93 Å with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The O atoms of the nitro group are disordered over two sets of sites (O1/O1′ and O2/O2′) with a refined occupancy ratio of 0.737 (19):0.263 (19).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_5H_6N_3O_2^+ \cdot H_2NO_3S^-$
M_r	236.21
Crystal system, space group	Orthorhombic, *Pbcn*
Temperature (K)	293
a, *b*, *c* (Å)	28.0866 (10), 9.0052 (3), 7.4023 (2)
V (Å^3)	1872.23 (10)
Z	8
Radiation type	Mo *Kα*
μ (mm^{-1})	0.36
Crystal size (mm)	0.35 × 0.30 × 0.25
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
$T_{\rm min}$, $T_{\rm max}$	0.887, 0.917
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15358, 1653, 1557
$R_{\rm int}$	0.024
$(\sin \theta/\lambda)_{\rm max}$ (Å^{-1})	0.594
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.055, 0.111, 1.28
No. of reflections	1653
No. of parameters	175
No. of restraints	50
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max}$, $\Delta\rho_{\rm min}$ (e Å^{-3})	0.27, −0.45

Computer programs: *APEX2*, *SAINT* and *XPREP* (Bruker, 2004), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009).

Acknowledgements

MAR, PAD and SSJX would like to thank the Board of Research in the Nuclear Sciences Department of Atomic Energy (BRNS–DAE) (File No. 2012/34/63/BRNS/2865; date: 01 March 2013) for funding this major research project.

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supporting information

Acta Cryst. (2015). E**71**, 231-233   [doi:10.1107/S2056989015000365]

Crystal structure of 2-amino-5-nitropyridinium sulfamate

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Computing details

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: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

2-Amino-5-nitropyridinium sulfamate

Crystal data

$C_5H_6N_3O_2^+\cdot H_2NO_3S^-$
$M_r = 236.21$
Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
$a = 28.0866$ (10) Å
$b = 9.0052$ (3) Å
$c = 7.4023$ (2) Å
$V = 1872.23$ (10) Å^3
$Z = 8$

$F(000) = 976$
$D_x = 1.676$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1653 reflections
$\theta = 2.4–31.1°$
$\mu = 0.36$ mm^{-1}
$T = 293$ K
Block, colourless
0.35 × 0.30 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{\min} = 0.887$, $T_{\max} = 0.917$

15358 measured reflections
1653 independent reflections
1557 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 25.0°$, $\theta_{\min} = 2.4°$
$h = -33 \rightarrow 33$
$k = -10 \rightarrow 10$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.111$
$S = 1.28$
1653 reflections
175 parameters
50 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

$w = 1/[\sigma^2(F_o^2) + (0.0116P)^2 + 5.4481P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ 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}	Occ. (<1)
C1	0.38918 (12)	0.1439 (4)	0.6248 (5)	0.0269 (8)	
C2	0.35730 (13)	0.0484 (4)	0.5342 (5)	0.0292 (8)	
H2	0.3602	−0.0538	0.5477	0.035*	
C3	0.32268 (13)	0.1047 (4)	0.4283 (5)	0.0373 (9)	
H3	0.3014	0.0421	0.3693	0.045*	
C4	0.31924 (13)	0.2591 (4)	0.4085 (6)	0.0371 (9)	
C5	0.34829 (13)	0.3498 (4)	0.4998 (5)	0.0345 (9)	
H5	0.3453	0.4522	0.4880	0.041*	
N1	0.28526 (15)	0.3204 (5)	0.2816 (7)	0.0721 (15)	
N2	0.42542 (12)	0.0961 (4)	0.7215 (4)	0.0343 (8)	
N3	0.38170 (11)	0.2922 (3)	0.6083 (4)	0.0298 (7)	
N4	0.47684 (11)	0.3034 (3)	−0.0187 (4)	0.0267 (7)	
O1	0.2679 (4)	0.2322 (7)	0.1676 (14)	0.105 (4)	0.737 (19)
O2	0.2783 (4)	0.4550 (6)	0.280 (2)	0.078 (4)	0.737 (19)
O1′	0.2456 (5)	0.258 (2)	0.279 (4)	0.089 (7)	0.263 (19)
O2′	0.2901 (9)	0.4556 (11)	0.254 (6)	0.050 (6)	0.263 (19)
O3	0.39040 (9)	0.3227 (3)	0.0338 (4)	0.0330 (6)	
O4	0.44135 (9)	0.2156 (3)	0.2645 (3)	0.0325 (6)	
O5	0.44161 (9)	0.4796 (3)	0.2125 (3)	0.0336 (6)	
S1	0.43441 (3)	0.33299 (9)	0.13275 (11)	0.0231 (2)	
H4B	0.5038 (10)	0.282 (4)	0.038 (5)	0.040 (12)*	
H4A	0.4797 (12)	0.384 (3)	−0.087 (4)	0.040 (12)*	
H3A	0.4008 (12)	0.356 (4)	0.663 (5)	0.043 (12)*	
H2B	0.4319 (13)	0.001 (2)	0.732 (6)	0.046 (12)*	
H2A	0.4435 (13)	0.154 (3)	0.787 (5)	0.051 (14)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0336 (18)	0.0247 (18)	0.0225 (17)	−0.0018 (15)	0.0077 (16)	−0.0052 (15)
C2	0.038 (2)	0.0201 (17)	0.0297 (19)	−0.0061 (15)	0.0054 (17)	−0.0005 (16)
C3	0.034 (2)	0.039 (2)	0.039 (2)	−0.0128 (17)	−0.0037 (18)	0.0011 (19)

C4	0.0303 (19)	0.039 (2)	0.042 (2)	−0.0001 (17)	0.0015 (17)	0.0127 (19)
C5	0.038 (2)	0.0248 (18)	0.040 (2)	0.0018 (16)	0.0134 (18)	0.0053 (17)
N1	0.046 (2)	0.070 (3)	0.100 (4)	−0.007 (2)	−0.023 (3)	0.039 (3)
N2	0.0415 (19)	0.0261 (17)	0.0353 (19)	−0.0004 (15)	−0.0064 (15)	−0.0048 (15)
N3	0.0382 (18)	0.0210 (15)	0.0303 (17)	−0.0046 (13)	0.0038 (14)	−0.0074 (14)
N4	0.0323 (17)	0.0268 (16)	0.0211 (15)	0.0009 (13)	−0.0010 (13)	0.0003 (13)
O1	0.097 (7)	0.089 (5)	0.129 (7)	−0.034 (4)	−0.080 (6)	0.036 (4)
O2	0.055 (6)	0.073 (5)	0.105 (8)	0.032 (3)	0.014 (5)	0.029 (4)
O1′	0.062 (10)	0.099 (11)	0.106 (14)	0.002 (9)	−0.043 (9)	0.003 (11)
O2′	0.032 (10)	0.050 (10)	0.068 (11)	0.023 (6)	0.008 (10)	0.026 (7)
O3	0.0326 (13)	0.0323 (14)	0.0340 (14)	−0.0008 (11)	−0.0074 (12)	−0.0047 (12)
O4	0.0407 (15)	0.0289 (13)	0.0277 (13)	−0.0039 (12)	0.0010 (12)	0.0047 (11)
O5	0.0415 (14)	0.0245 (13)	0.0349 (14)	0.0030 (12)	−0.0083 (12)	−0.0085 (11)
S1	0.0293 (4)	0.0191 (4)	0.0210 (4)	−0.0007 (3)	−0.0026 (4)	−0.0015 (3)

Geometric parameters (Å, º)

C1—N2	1.317 (5)	N1—O2′	1.243 (8)
C1—N3	1.357 (4)	N1—O1′	1.246 (8)
C1—C2	1.411 (5)	N1—O1	1.257 (6)
C2—C3	1.348 (5)	N2—H2B	0.884 (18)
C2—H2	0.9300	N2—H2A	0.877 (18)
C3—C4	1.402 (6)	N3—H3A	0.886 (19)
C3—H3	0.9300	N4—S1	1.657 (3)
C4—C5	1.338 (6)	N4—H4B	0.889 (18)
C4—N1	1.449 (6)	N4—H4A	0.890 (18)
C5—N3	1.340 (5)	O3—S1	1.440 (3)
C5—H5	0.9300	O4—S1	1.451 (3)
N1—O2	1.228 (6)	O5—S1	1.460 (2)

N2—C1—N3	119.3 (3)	O1′—N1—O1	50.3 (11)
N2—C1—C2	123.4 (3)	O2—N1—C4	119.1 (8)
N3—C1—C2	117.3 (3)	O2′—N1—C4	114.0 (14)
C3—C2—C1	120.3 (3)	O1′—N1—C4	115.3 (10)
C3—C2—H2	119.8	O1—N1—C4	116.7 (5)
C1—C2—H2	119.8	C1—N2—H2B	122 (2)
C2—C3—C4	118.9 (4)	C1—N2—H2A	124 (3)
C2—C3—H3	120.5	H2B—N2—H2A	114 (3)
C4—C3—H3	120.5	C5—N3—C1	122.9 (3)
C5—C4—C3	120.7 (4)	C5—N3—H3A	116 (3)
C5—C4—N1	119.8 (4)	C1—N3—H3A	120 (3)
C3—C4—N1	119.4 (4)	S1—N4—H4B	109 (3)
C4—C5—N3	119.6 (3)	S1—N4—H4A	108 (2)
C4—C5—H5	120.2	H4B—N4—H4A	112 (3)
N3—C5—H5	120.2	O3—S1—O4	114.22 (15)
O2—N1—O2′	17.8 (19)	O3—S1—O5	112.50 (15)
O2—N1—O1′	107.6 (12)	O4—S1—O5	111.59 (15)
O2′—N1—O1′	122.5 (12)	O3—S1—N4	105.26 (16)

O2—N1—O1	123.8 (8)	O4—S1—N4	103.93 (15)
O2′—N1—O1	123.5 (19)	O5—S1—N4	108.63 (15)
N2—C1—C2—C3	−175.9 (4)	C5—C4—N1—O2′	8 (2)
N3—C1—C2—C3	3.6 (5)	C3—C4—N1—O2′	−169 (2)
C1—C2—C3—C4	0.6 (6)	C5—C4—N1—O1′	−141.5 (16)
C2—C3—C4—C5	−3.2 (6)	C3—C4—N1—O1′	41.3 (16)
C2—C3—C4—N1	174.0 (4)	C5—C4—N1—O1	162.1 (8)
C3—C4—C5—N3	1.4 (6)	C3—C4—N1—O1	−15.1 (9)
N1—C4—C5—N3	−175.7 (4)	C4—C5—N3—C1	3.1 (6)
C5—C4—N1—O2	−11.3 (11)	N2—C1—N3—C5	174.0 (3)
C3—C4—N1—O2	171.4 (9)	C2—C1—N3—C5	−5.5 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O4^{i}	0.88 (2)	1.98 (2)	2.861 (4)	176 (4)
N2—H2*A*···N4^{ii}	0.88 (2)	2.18 (2)	3.044 (4)	169 (4)
N3—H3*A*···O5^{iii}	0.89 (2)	1.91 (2)	2.766 (4)	163 (4)
N4—H4*B*···O4^{iv}	0.89 (2)	2.20 (2)	3.073 (4)	166 (3)
N4—H4*A*···O5^{v}	0.89 (2)	2.20 (2)	2.960 (4)	143 (3)
C2—H2···O3^{i}	0.93	2.57	3.469 (4)	163
C3—H3···O2^{vi}	0.93	2.46	3.328 (13)	155
C5—H5···O3^{iii}	0.93	2.41	3.187 (4)	141

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