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Crystal structure of 4-{(*E*)-[2-(pyridin-4-ylcarbonyl)hydrazin-1-ylidene]methyl}-phenyl acetate monohydrate

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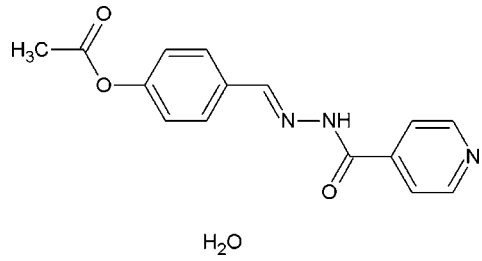
The asymmetric unit of the title compound, $C_{15}H_{13}N_3O_3\cdot H_2O$, comprises a 4-{(*E*)-[2-(pyridin-4-ylcarbonyl)hydrazinylidene]methyl}phenyl acetate molecule and a solvent water molecule linked by O—H···O and O—H···N hydrogen bonds from the water molecule and a C—H···O contact from the organic molecule. The compound adopts an *E* conformation with respect to the azomethine bond and the dihedral angle between the pyridine and benzene rings is 21.90 (7)°. The azomethine bond [1.275 (2) Å] distance is very close to the formal C=N bond length, which confirms the azomethine bond formation. An extensive set of O—H···O, O—H···N, N—H···O and C—H···O hydrogen bonds builds a two-dimensional network progressing along the *c* axis.

Keywords: crystal structure; hydrazone; aroyl hydrazone; hydrogen bonding.

CCDC reference: 1040455

1. Related literature

For biological applications of hydrazone derivatives, see: Sreeja *et al.* (2004); Prasanna & Kumar (2013). For the synthesis of related compounds, see: Joseph *et al.* (2013); Thilagavathi *et al.* (2010). For the anticancer activity of hydrazones against cervical cancer, see: Nair *et al.* (2014).



2. Experimental

2.1. Crystal data

$C_{15}H_{13}N_3O_3\cdot H_2O$
M_r = 301.30
Monoclinic, $P2_1/c$
a = 17.3297 (15) Å
b = 7.3058 (7) Å
c = 12.4632 (10) Å
β = 111.034 (3)°
V = 1472.8 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.50 × 0.45 × 0.40 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.951, T_{max} = 0.961
8648 measured reflections
2614 independent reflections
2153 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.113
S = 0.94
2614 reflections
213 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O1S^{i}	0.93	2.56	3.375 (2)	147
C7—H7···O1S^{i}	0.93	2.56	3.3655 (19)	145
C12—H12···O3^{i}	0.93	2.54	3.329 (2)	143
N2—H2′···O1S^{i}	0.88 (1)	2.08 (1)	2.9529 (18)	170 (2)
O1S—H1S···N3	0.86 (2)	2.65 (2)	3.2897 (18)	133 (2)
O1S—H1S···O1	0.86 (2)	2.02 (2)	2.8382 (17)	159 (2)
O1S—H2S···O3^{ii}	0.86 (2)	2.38 (2)	3.1754 (19)	154 (2)

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

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: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2010); software used to prepare material for publication: *SHELXL97* and *publCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SJ5434).

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supporting information

Acta Cryst. (2015). E**71**, o79–o80 [doi:10.1107/S2056989014027819]

Crystal structure of 4-{(*E*)-[2-(pyridin-4-ylcarbonyl)hydrazin-1-ylidene]methyl}-phenyl acetate monohydrate

Riya Datta, V. Ramya, M. Sithambaresan and M. R. Prathapachandra Kurup

S1. Structural commentary

Hydrazone derivatives are found to have structural diversity due to their coordinative ability (Sreeja *et al.*, 2004; Prasanna & Kumar, 2013) arising from thioamido-thioiminol tautomerism. Ruthenium(II) complexes with such compounds as ligands have been shown to function as catalysts (Thilagavathi *et al.*, 2010). The title compound $[C_{15}H_{13}N_3O_3].(H_2O)$ adopts an *E* configuration with respect to C7═N3 bond and O1 and N3 are cis with respect to the C6—N2 bond (Fig. 1). The dihedral angle between the pyridine and benzene rings is 21.90 (7) Å. The C7═N3 [1.275 (2) Å] bond distances are very close to the formal C═N bond length, which confirms the azomethine bond formation.

There are four classical intermolecular O—H···O, O—H···N and N—H···O hydrogen bonds and three non-classical C—H···O interactions, (Table 1, Figure 2). These intermolecular hydrogen bonds build a two-dimensional network progressing along the *c* axis (Fig. 3). Fig. 4 shows the packing diagram of the title compound along the *a* axis.

S2. Synthesis and crystallization

The title compound was synthesised by adapting a reported procedure (Joseph *et al.*, 2013). A solution of isonicotinic acid hydrazide (0.137 g, 1 mmol) in methanol/DMF (1:1 v/v, 10 ml) was mixed with a methanol /DMF solution (10 ml) of 4-formylphenyl acetate (0.164 g, 1 mmol). The mixture was refluxed for 6 h and then cooled to room temperature. The resulting solid was recrystallized from chloroform/methanol (1:1 v/v). Colorless, block shaped crystals suitable for XRD studies were obtained after slow evaporation of the solution in air over several days.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms bound to C were placed in calculated positions, guided by difference maps, with C—H bond distances of 0.93-0.96 Å. H atoms were assigned U_{iso}(H) values of 1.2Ueq(carrier). H2 on N2 and H1S & H2S of the water molecule were located in a difference Fourier map and refined with the bond distances restrained to 0.88±0.01 and 0.86±0.02 Å respectively. The low angle reflection (1 0 0) was omitted from the refinement.

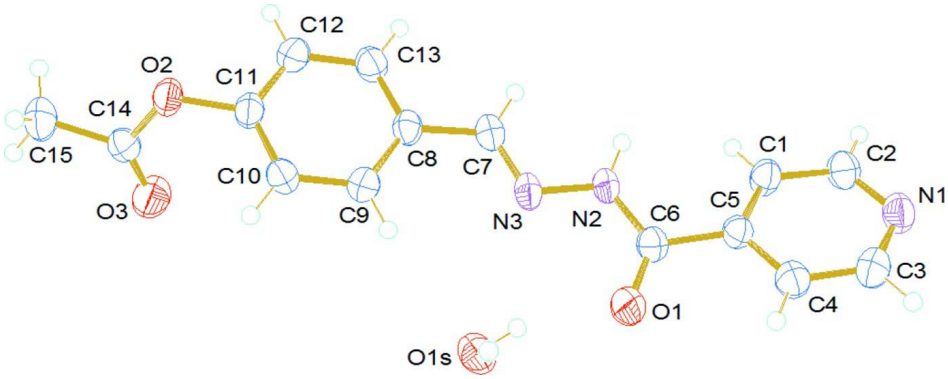

Figure 1

An *ORTEP* view of the compound, with 50% probability displacement ellipsoids for the non-H atoms.

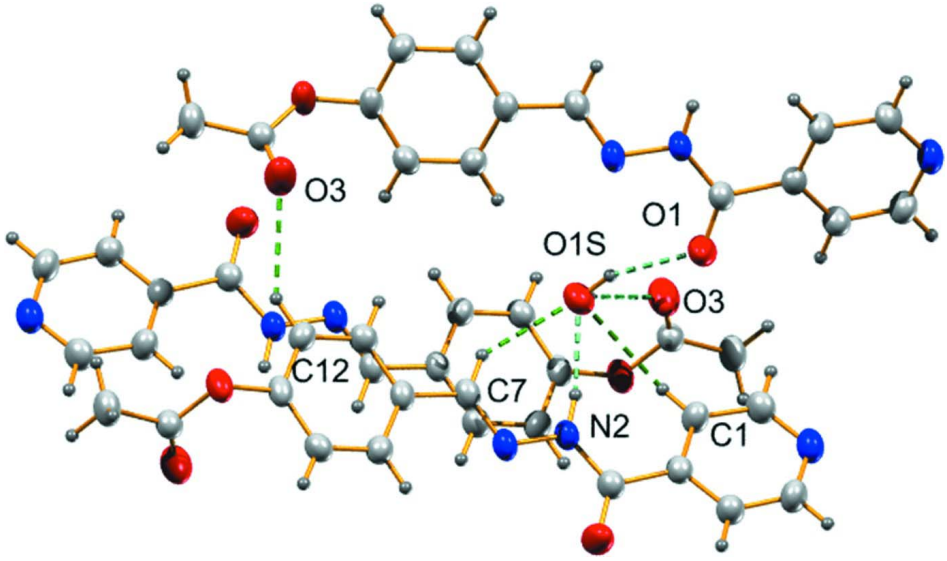

Figure 2

Graphical representation showing hydrogen bonding interactions in the crystal structure of $[C_{15}H_{13}N_3O_3]\cdot(H_2O)$.

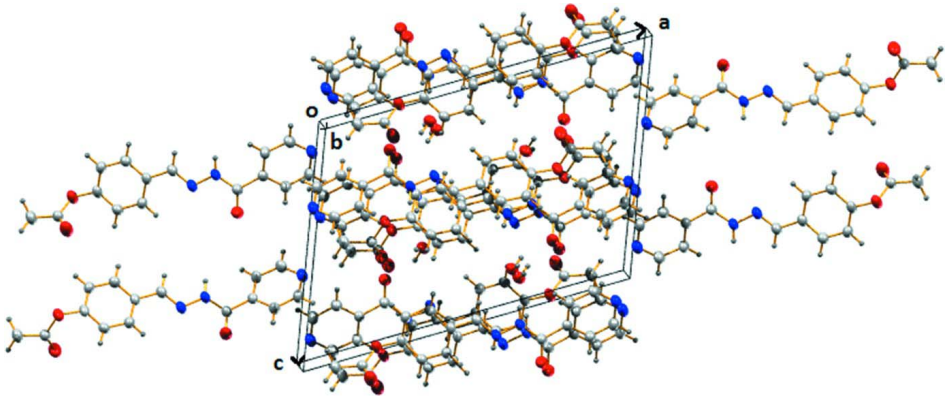

Figure 3

The hydrogen bonding interactions build a double layer progressing along the *c* axis in the title compound.

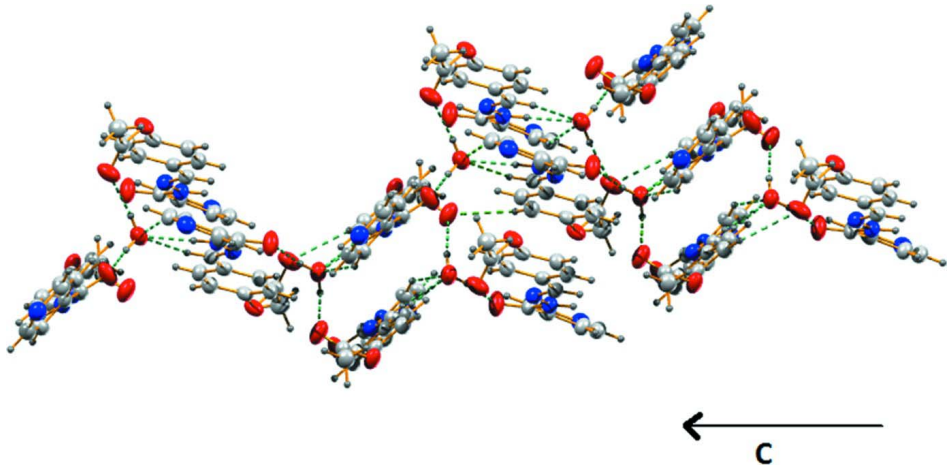

Figure 4

A view of the overall crystal packing along the *a* axis.

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Crystal data

$C_{15}H_{13}N_3O_3·H_2O$
M_r = 301.30
Monoclinic, $P2_1/c$
a = 17.3297 (15) Å
b = 7.3058 (7) Å
c = 12.4632 (10) Å
β = 111.034 (3)°
V = 1472.8 (2) Å^3
Z = 4
$F(000)$ = 632
D_x = 1.359 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4335 reflections
θ = 3.1–28.1°
μ = 0.10 mm^{-1}
T = 296 K
Block, colorless
0.50 × 0.45 × 0.40 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.951, T_{max} = 0.961
8648 measured reflections
2614 independent reflections
2153 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.1°, θ_{min} = 3.1°
h = −20→15
k = −8→8
l = −11→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.113
S = 0.94
2614 reflections
213 parameters
4 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2 + 0.4633P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.024 (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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.16188 (10)	0.0685 (2)	0.93233 (14)	0.0400 (4)
H1	0.2096	0.0125	0.9817	0.048*
C2	0.08732 (10)	0.0473 (2)	0.94865 (14)	0.0427 (4)
H2	0.0868	−0.0231	1.0105	0.051*
C3	0.01996 (10)	0.2228 (3)	0.79347 (15)	0.0476 (4)
H3	−0.0287	0.2763	0.7449	0.057*
C4	0.09113 (10)	0.2527 (2)	0.77045 (14)	0.0423 (4)
H4	0.0899	0.3245	0.7082	0.051*
C5	0.16422 (9)	0.1743 (2)	0.84140 (12)	0.0340 (4)
C6	0.24113 (9)	0.2125 (2)	0.81590 (13)	0.0369 (4)
C7	0.45140 (9)	0.2232 (2)	0.97566 (13)	0.0367 (4)
H7	0.4483	0.1754	1.0431	0.044*
C8	0.53158 (9)	0.2825 (2)	0.97427 (13)	0.0340 (4)
C9	0.54021 (9)	0.3707 (2)	0.87968 (13)	0.0380 (4)
H9	0.4941	0.3887	0.8134	0.046*
C10	0.61707 (10)	0.4314 (2)	0.88411 (13)	0.0388 (4)
H10	0.6229	0.4915	0.8216	0.047*
C11	0.68496 (9)	0.4011 (2)	0.98292 (14)	0.0364 (4)
C12	0.67832 (9)	0.3173 (2)	1.07787 (13)	0.0397 (4)
H12	0.7247	0.3000	1.1440	0.048*
C13	0.60111 (10)	0.2590 (2)	1.07295 (14)	0.0395 (4)
H13	0.5957	0.2030	1.1369	0.047*
C14	0.80143 (9)	0.3873 (2)	0.92676 (13)	0.0367 (4)
C15	0.88679 (10)	0.4588 (3)	0.95589 (16)	0.0483 (4)
H15A	0.9127	0.3993	0.9089	0.072*
H15B	0.9181	0.4351	1.0354	0.072*
H15C	0.8847	0.5884	0.9421	0.072*
N1	0.01652 (8)	0.1220 (2)	0.88093 (12)	0.0455 (4)
N2	0.31362 (7)	0.18494 (18)	0.90293 (11)	0.0358 (3)
N3	0.38562 (8)	0.23535 (18)	0.88687 (11)	0.0369 (3)
O1	0.23649 (7)	0.2689 (2)	0.72122 (10)	0.0589 (4)
O1S	0.35113 (8)	0.4455 (2)	0.64055 (10)	0.0480 (3)
O2	0.76382 (7)	0.46558 (16)	0.99337 (10)	0.0446 (3)
O3	0.76890 (8)	0.27267 (19)	0.85709 (12)	0.0598 (4)
H1S	0.3278 (13)	0.387 (3)	0.6800 (18)	0.080 (7)*
H2S	0.3334 (15)	0.554 (2)	0.644 (2)	0.094 (9)*
H2′	0.3186 (11)	0.148 (2)	0.9725 (10)	0.049 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0310 (8)	0.0445 (10)	0.0443 (9)	−0.0027 (7)	0.0131 (7)	0.0066 (7)
C2	0.0399 (9)	0.0451 (10)	0.0471 (9)	−0.0080 (8)	0.0205 (7)	0.0024 (7)
C3	0.0328 (9)	0.0593 (12)	0.0497 (10)	0.0064 (8)	0.0138 (7)	0.0032 (8)
C4	0.0376 (9)	0.0498 (10)	0.0412 (8)	0.0021 (8)	0.0162 (7)	0.0055 (7)
C5	0.0308 (8)	0.0363 (9)	0.0357 (8)	−0.0045 (7)	0.0132 (6)	−0.0039 (6)
C6	0.0343 (8)	0.0403 (9)	0.0385 (8)	−0.0059 (7)	0.0158 (7)	0.0003 (7)
C7	0.0347 (9)	0.0365 (9)	0.0435 (9)	−0.0015 (7)	0.0199 (7)	0.0004 (7)
C8	0.0303 (8)	0.0330 (8)	0.0419 (8)	0.0002 (6)	0.0169 (6)	−0.0039 (6)
C9	0.0317 (8)	0.0445 (9)	0.0373 (8)	−0.0010 (7)	0.0118 (6)	−0.0013 (7)
C10	0.0391 (9)	0.0441 (9)	0.0379 (8)	−0.0048 (7)	0.0196 (7)	−0.0012 (7)
C11	0.0304 (8)	0.0386 (9)	0.0443 (8)	−0.0071 (7)	0.0184 (7)	−0.0121 (7)
C12	0.0315 (8)	0.0465 (10)	0.0395 (8)	0.0005 (7)	0.0106 (7)	−0.0013 (7)
C13	0.0388 (9)	0.0426 (9)	0.0400 (8)	−0.0003 (7)	0.0176 (7)	0.0040 (7)
C14	0.0351 (8)	0.0394 (9)	0.0378 (8)	0.0001 (7)	0.0157 (7)	−0.0018 (7)
C15	0.0375 (9)	0.0532 (11)	0.0600 (10)	−0.0045 (8)	0.0246 (8)	−0.0013 (8)
N1	0.0352 (8)	0.0526 (9)	0.0534 (8)	−0.0053 (7)	0.0216 (7)	−0.0049 (7)
N2	0.0286 (7)	0.0437 (8)	0.0394 (7)	−0.0049 (6)	0.0174 (6)	0.0019 (6)
N3	0.0305 (7)	0.0402 (8)	0.0455 (7)	−0.0042 (6)	0.0203 (6)	−0.0011 (6)
O1	0.0413 (7)	0.0937 (11)	0.0431 (7)	−0.0115 (7)	0.0168 (5)	0.0149 (7)
O1S	0.0471 (7)	0.0572 (9)	0.0459 (7)	0.0022 (6)	0.0241 (6)	0.0071 (6)
O2	0.0342 (6)	0.0543 (7)	0.0512 (7)	−0.0151 (5)	0.0223 (5)	−0.0191 (5)
O3	0.0482 (8)	0.0701 (9)	0.0660 (8)	−0.0102 (7)	0.0266 (6)	−0.0306 (7)

Geometric parameters (Å, º)

C1—C2	1.387 (2)	C9—H9	0.9300
C1—C5	1.384 (2)	C10—C11	1.382 (2)
C1—H1	0.9300	C10—H10	0.9300
C2—N1	1.331 (2)	C11—C12	1.374 (2)
C2—H2	0.9300	C11—O2	1.4066 (18)
C3—N1	1.334 (2)	C12—C13	1.384 (2)
C3—C4	1.380 (2)	C12—H12	0.9300
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.382 (2)	C14—O3	1.1921 (19)
C4—H4	0.9300	C14—O2	1.3527 (18)
C5—C6	1.503 (2)	C14—C15	1.486 (2)
C6—O1	1.2252 (19)	C15—H15A	0.9600
C6—N2	1.348 (2)	C15—H15B	0.9600
C7—N3	1.275 (2)	C15—H15C	0.9600
C7—C8	1.462 (2)	N2—N3	1.3827 (17)
C7—H7	0.9300	N2—H2′	0.883 (9)
C8—C13	1.389 (2)	O1S—H1S	0.857 (16)
C8—C9	1.398 (2)	O1S—H2S	0.860 (16)
C9—C10	1.386 (2)		

C2—C1—C5	119.01 (15)	C11—C10—H10	120.6
C2—C1—H1	120.5	C9—C10—H10	120.6
C5—C1—H1	120.5	C12—C11—C10	122.10 (14)
N1—C2—C1	123.81 (15)	C12—C11—O2	116.65 (14)
N1—C2—H2	118.1	C10—C11—O2	121.14 (14)
C1—C2—H2	118.1	C11—C12—C13	118.52 (14)
N1—C3—C4	124.23 (16)	C11—C12—H12	120.7
N1—C3—H3	117.9	C13—C12—H12	120.7
C4—C3—H3	117.9	C12—C13—C8	121.23 (15)
C3—C4—C5	118.91 (15)	C12—C13—H13	119.4
C3—C4—H4	120.5	C8—C13—H13	119.4
C5—C4—H4	120.5	O3—C14—O2	122.55 (14)
C4—C5—C1	117.75 (14)	O3—C14—C15	126.45 (15)
C4—C5—C6	117.81 (14)	O2—C14—C15	110.97 (14)
C1—C5—C6	124.43 (14)	C14—C15—H15A	109.5
O1—C6—N2	123.04 (14)	C14—C15—H15B	109.5
O1—C6—C5	120.63 (14)	H15A—C15—H15B	109.5
N2—C6—C5	116.31 (13)	C14—C15—H15C	109.5
N3—C7—C8	121.78 (14)	H15A—C15—H15C	109.5
N3—C7—H7	119.1	H15B—C15—H15C	109.5
C8—C7—H7	119.1	C2—N1—C3	116.29 (14)
C13—C8—C9	118.84 (14)	C6—N2—N3	118.20 (12)
C13—C8—C7	118.62 (14)	C6—N2—H2′	124.8 (11)
C9—C8—C7	122.45 (14)	N3—N2—H2′	116.7 (11)
C10—C9—C8	120.40 (14)	C7—N3—N2	115.33 (13)
C10—C9—H9	119.8	H1S—O1S—H2S	100.4 (19)
C8—C9—H9	119.8	C14—O2—C11	117.88 (12)
C11—C10—C9	118.88 (14)		

C5—C1—C2—N1	0.6 (3)	C9—C10—C11—O2	−177.75 (14)
N1—C3—C4—C5	0.1 (3)	C10—C11—C12—C13	1.1 (2)
C3—C4—C5—C1	0.2 (2)	O2—C11—C12—C13	177.22 (14)
C3—C4—C5—C6	−178.71 (15)	C11—C12—C13—C8	0.6 (2)
C2—C1—C5—C4	−0.5 (2)	C9—C8—C13—C12	−1.5 (2)
C2—C1—C5—C6	178.31 (14)	C7—C8—C13—C12	−178.10 (15)
C4—C5—C6—O1	−18.5 (2)	C1—C2—N1—C3	−0.3 (2)
C1—C5—C6—O1	162.62 (17)	C4—C3—N1—C2	−0.1 (3)
C4—C5—C6—N2	160.04 (15)	O1—C6—N2—N3	4.8 (2)
C1—C5—C6—N2	−18.8 (2)	C5—C6—N2—N3	−173.74 (12)
N3—C7—C8—C13	−177.64 (15)	C8—C7—N3—N2	−176.06 (13)
N3—C7—C8—C9	5.9 (2)	C6—N2—N3—C7	173.26 (14)
C13—C8—C9—C10	0.8 (2)	O3—C14—O2—C11	3.2 (2)
C7—C8—C9—C10	177.24 (15)	C15—C14—O2—C11	−174.82 (14)
C8—C9—C10—C11	0.8 (2)	C12—C11—O2—C14	115.34 (16)
C9—C10—C11—C12	−1.8 (2)	C10—C11—O2—C14	−68.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1*S*^{i}	0.93	2.56	3.375 (2)	147
C7—H7···O1*S*^{i}	0.93	2.56	3.3655 (19)	145
C12—H12···O3^{i}	0.93	2.54	3.329 (2)	143
N2—H2′···O1*S*^{i}	0.88 (1)	2.08 (1)	2.9529 (18)	170 (2)
O1*S*—H1*S*···N3	0.86 (2)	2.65 (2)	3.2897 (18)	133 (2)
O1*S*—H1*S*···O1	0.86 (2)	2.02 (2)	2.8382 (17)	159 (2)
O1*S*—H2*S*···O3^{ii}	0.86 (2)	2.38 (2)	3.1754 (19)	154 (2)

Symmetry codes: (i) x, $-y+1/2$, $z+1/2$; (ii) $-x+1$, $y+1/2$, $-z+3/2$.