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Crystal structure of aqua(nitrato-κO)dioxido{2-[3-(pyridin-2-yl-κN)-1*H*-1,2,4-triazol-5-yl-κN^4]-phenolato-κO}uranium(VI) acetonitrile monosolvate monohydrate

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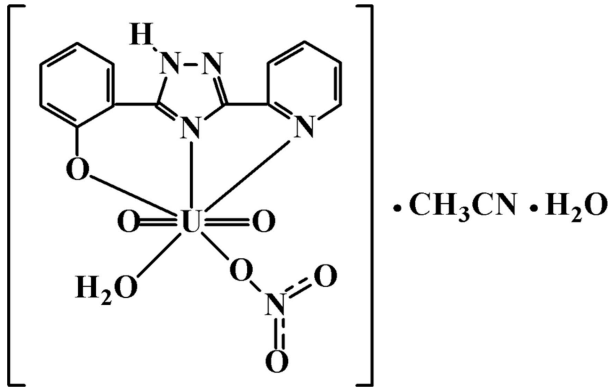
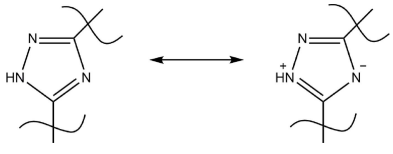
In the title compound, $[U(C_{13}H_9N_4O)(NO_3)O_2(H_2O)]\cdot CH_3CN\cdot H_2O$, the U^{VI} atom is seven-coordinated in a distorted pentagonal–bipyramidal N_2O_5 manner by one tridentate triazole ligand, one monodentate nitrate anion and one water molecule in the equatorial plane and by two uranyl(VI) O atoms in the axial positions. In the crystal, the U^{VI} complex molecule is linked to the water and acetonitrile solvent molecules through N—H···N, O—H···O and O—H···N hydrogen bonds, forming a sheet structure parallel to the *bc* plane. The sheets are further linked by an additional O—H···O hydrogen bond, forming a three-dimensional network.

1. Chemical context

The synthesis of coordination compounds with *N*-donor heterocyclic ligands is one of the fastest growing areas of coordination chemistry. 1,2,4-Triazoles and their derivatives can be assigned for such types of ligands. The presence of the 1,2,4-triazole ring in the organic ligand provides an additional site for coordination (Aromí *et al.*, 2011). The presence of additional donor groups in the 3- and 5-positions of the triazole moiety provides a greater number of possibilities for chelation of metal ions, involving tridentate bis-chelate functions.





It should be noted that UO_2^{2+} complexes with such types of ligands have rarely been investigated. Thus, only three uranyl complexes with 1,2,4-triazole derivatives have been characterized (Daro *et al.*, 2001; Weng *et al.*, 2012; Raspertova *et al.*, 2012). As part of our continuing study of uranium coordination compounds with nitrogen-donor ligands (Raspertova *et al.*, 2012), we report here the structure of the title compound.

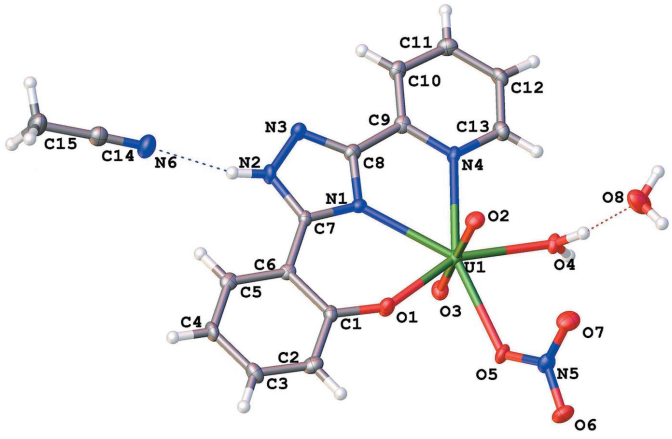


Figure 1
The molecular structure of the title compound, shown with 50% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

2. Structural commentary

The coordination polyhedron of the U^{VI} atom in the title complex is a distorted pentagonal bipyramid. It is coordinated in a tridentate manner by the 1,2,4,-triazole ligand together with the water molecule and the monodentate nitrate anion in the equatorial plane. Two oxido ligands are placed in the axial positions (Fig. 1). The U1—O1 bond length [2.206 (3) Å] is comparable with those reported for related six-membered chelate fragments involving phenolate and *N*-atom donors (Sopo *et al.*, 2008; Ahmadi *et al.*, 2012). The U—N bond lengths [2.489 (4) and 2.658 (4) Å] are consistent with the situation in other pyridine-bonded uranium complexes (Amoroso *et al.*, 1996; Gatto *et al.*, 2004). The uranyl group is not exactly linear [O2═U1═O3 = 175.36 (14)°]. Non-linear O═U═ groups are generally found in uranyl complexes with five non-symmetrically bonding equatorial ligands. All non-hydrogen atoms of the organic ligand are coplanar within 0.01 Å. The N1—C7 and C7—N2 bond lengths of the triazole ring are equalized [1.336 (5) Å for both]. This value is longer than a Csp^2 ═N double bond (1.276 Å) and shorter than a Csp^2—N single bond (1.347 Å) (Orpen *et al.*, 1994). It can be assumed that the structure of the triazole ring is the superposition of two possible resonance structures as shown in Fig. 2.

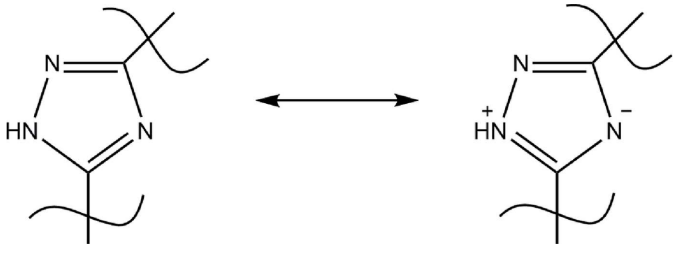


Figure 2
Scheme showing two possible resonance structures in the triazole ligand.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*A*···O5^{i}	0.86 (1)	1.92 (2)	2.752 (4)	162 (5)
O4—H4*B*···O8	0.86 (1)	1.73 (1)	2.581 (5)	168 (5)
N2—H2···N6	0.86	2.07	2.909 (5)	165
O8—H8*A*···N3^{ii}	0.86 (1)	2.05 (2)	2.890 (5)	164 (6)
O8—H8*B*···O6^{iii}	0.86 (1)	2.22 (3)	3.001 (5)	150 (6)

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

3. Supramolecular features

In the crystal, the complex molecule is linked to the water and acetonitrile solvent molecules through N2—H2···N6, O4—H4*B*···O8, O8—H8*A*···N3^{ii} and O8—H8*B*···O6^{iii} hydrogen bonds (symmetry codes in Table 1), forming a sheet structure parallel to the *bc* plane. The sheets are further linked by an O4—H4*A*···O5^{i} hydrogen bond (Table 1), forming a three-dimensional network (Fig. 3).

4. Database survey

In the Cambridge Structural Database (Version 5.36, November 2014; Groom & Allen, 2014), only three uranyl complexes with derivatives of 1,2,4-triazole are reported (Daro *et al.*, 2001; Weng *et al.*, 2012; Raspertova *et al.*, 2012). 72 structures containing a 5-pyridin-1*H*-1,2,4-triazole fragment are found. A search for the 3-hydroxyphenyl-1,2,4-triazole fragment yielded 14 hits, including: 2,2′-[1-(2,4,6-trichlorophenyl)-1*H*-1,2,4-triazole-3,5-diyl]diphenol (Li *et al.*, 2008); 2-[5-(2-pyridyl)-1,2,4-triazole-3-yl]phenol 2-[3-(2-pyridyl)-1,2,4-triazole-5-yl]phenol bis(7,7,8,8-tetracyanoquinodimethane) (Bentiss *et al.*, 2002); bis[μ_2-1-phenyl-3,5-bis(2-oxyphenyl)-1,2,4-triazole]bis(pyridine)dicopper (Steinhauser *et al.*, 2004). Only one compound containing both hydroxyphenyl and pyridyl, as substituents in the 3- and 5-positions of 1,2,4-triazole, was found (Bentiss *et al.*, 2002).

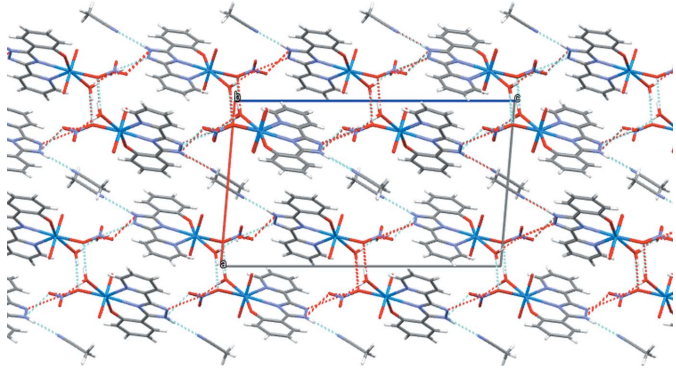


Figure 3
Packing diagram of the title compound, viewed along the *b* axis. Intermolecular hydrogen bonds are shown as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[U(C_{13}H_9N_4O)(NO_3)O_2(H_2O)]\cdot CH_3CN\cdot H_2O$
M_r	646.37
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	294
a, b, c (Å)	12.0962 (3), 7.87839 (17), 20.4041 (4)
β (°)	94.829 (2)
V (Å^3)	1937.57 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	8.44
Crystal size (mm)	0.5 × 0.3 × 0.2
Data collection	
Diffractometer	Agilent Xcalibur, Sapphire3
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.055, 0.185
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9385, 4446, 3936
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.064, 1.05
No. of reflections	4446
No. of parameters	284
No. of restraints	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.81, −0.88

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

5. Synthesis and crystallization

A mixture of 3-(2-hydroxyphenyl)-5-(pyridin-2-yl)-1*H*-1,2,4-triazole (0.5 mmol) and $[UO_2(NO_3)_2]\cdot 2H_2O$ (0.5 mmol) in acetonitrile (20 ml) was stirred for 20 min. The solution was left to evaporate slowly at room temperature. Red single crystals suitable for X-ray analysis were obtained after 2 d.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were located in a difference Fourier map. The positional parameters of water H atoms were refined, with the restraint O—H = 0.860 (2) Å and the constraint $U_{iso}(H) = 1.5U_{eq}(O)$. All other H atoms were constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C, N)$.

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Crystal structure of aqua(nitrato-κO)dioxido{2-[3-(pyridin-2-yl-κN)-1*H*-1,2,4-triazol-5-yl-κN^4]phenolato-κO}uranium(VI) acetonitrile monosolvate monohydrate

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015*b*); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Aqua(nitrato-κO)dioxido{2-[3-(pyridin-2-yl-κN)-1*H*-1,2,4-triazol-5-yl-κN^4]phenolato-κO}uranium(VI) acetonitrile monosolvate monohydrate

Crystal data

$[U(C_{13}H_9N_4O)(NO_3)O_2(H_2O)]\cdot CH_3CN\cdot H_2O$
M_r = 646.37
Monoclinic, $P2_1/c$
a = 12.0962 (3) Å
b = 7.87839 (17) Å
c = 20.4041 (4) Å
β = 94.829 (2)°
V = 1937.57 (7) Å^3
Z = 4
$F(000)$ = 1216
D_x = 2.216 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4414 reflections
θ = 3.3–31.9°
μ = 8.44 mm^{-1}
T = 294 K
Block, red
0.5 × 0.3 × 0.2 mm

Data collection

Agilent Xcalibur, Sapphire3 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1827 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.055, T_{max} = 0.185
9385 measured reflections
4446 independent reflections
3936 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −15→15
k = −5→10
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.064
S = 1.05
4446 reflections
284 parameters
4 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0276P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.81$ e Å^{-3}
$\Delta\rho_{min} = -0.88$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
U1	0.81873 (2)	0.51832 (2)	0.40174 (2)	0.01286 (6)
O1	0.7272 (3)	0.2815 (4)	0.37710 (15)	0.0193 (7)
O2	0.6934 (2)	0.6205 (4)	0.41869 (15)	0.0189 (7)
O3	0.9494 (2)	0.4323 (4)	0.38551 (15)	0.0179 (6)
O4	0.9187 (2)	0.7071 (4)	0.47688 (15)	0.0164 (6)
H4A	0.9886 (8)	0.709 (7)	0.489 (2)	0.025*
H4B	0.890 (3)	0.768 (5)	0.5061 (16)	0.025*
O5	0.8563 (2)	0.3461 (4)	0.50215 (14)	0.0186 (7)
O6	0.8368 (3)	0.2773 (4)	0.60322 (15)	0.0234 (7)
O7	0.7619 (3)	0.5091 (4)	0.56334 (18)	0.0258 (8)
N1	0.7589 (3)	0.5025 (4)	0.28218 (18)	0.0128 (7)
N2	0.6933 (3)	0.4457 (5)	0.18299 (18)	0.0162 (7)
H2	0.6608	0.3920	0.1501	0.019*
N3	0.7399 (3)	0.6016 (4)	0.17900 (18)	0.0159 (8)
N4	0.8707 (3)	0.7819 (4)	0.32904 (18)	0.0134 (7)
N5	0.8162 (3)	0.3796 (5)	0.55859 (18)	0.0174 (8)
N6	0.5829 (3)	0.3255 (5)	0.0595 (2)	0.0266 (9)
C1	0.6794 (3)	0.1782 (5)	0.3324 (2)	0.0138 (8)
C2	0.6414 (3)	0.0177 (5)	0.3517 (2)	0.0184 (9)
H2A	0.6503	−0.0141	0.3957	0.022*
C3	0.5915 (3)	−0.0919 (5)	0.3057 (2)	0.0183 (9)
H3	0.5661	−0.1965	0.3194	0.022*
C4	0.5781 (3)	−0.0498 (5)	0.2396 (2)	0.0166 (9)
H4	0.5443	−0.1250	0.2090	0.020*
C5	0.6158 (3)	0.1056 (5)	0.2200 (2)	0.0155 (9)
H5	0.6081	0.1343	0.1756	0.019*
C6	0.6654 (3)	0.2206 (5)	0.2653 (2)	0.0126 (8)
C7	0.7042 (3)	0.3861 (5)	0.2445 (2)	0.0114 (8)
C8	0.7785 (3)	0.6308 (5)	0.2401 (2)	0.0142 (9)
C9	0.8385 (3)	0.7813 (5)	0.2635 (2)	0.0142 (9)
C10	0.8617 (3)	0.9142 (5)	0.2225 (2)	0.0163 (9)
H10	0.8386	0.9105	0.1778	0.020*
C11	0.9198 (3)	1.0527 (6)	0.2489 (2)	0.0184 (9)
H11	0.9361	1.1435	0.2222	0.022*

C12	0.9532 (3)	1.0555 (6)	0.3147 (2)	0.0169 (9)
H12	0.9923	1.1478	0.3331	0.020*
C13	0.9277 (3)	0.9180 (5)	0.3534 (2)	0.0176 (9)
H13	0.9510	0.9201	0.3980	0.021*
C14	0.5369 (4)	0.2758 (6)	0.0120 (2)	0.0199 (10)
C15	0.4767 (4)	0.2097 (6)	−0.0472 (2)	0.0271 (11)
H15A	0.4567	0.3017	−0.0767	0.041*
H15B	0.4107	0.1531	−0.0358	0.041*
H15C	0.5227	0.1308	−0.0682	0.041*
O8	0.8271 (3)	0.9203 (5)	0.55170 (19)	0.0344 (9)
H8A	0.811 (5)	0.899 (8)	0.5911 (11)	0.052*
H8B	0.844 (5)	1.026 (2)	0.554 (3)	0.052*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
U1	0.01190 (9)	0.01556 (9)	0.01102 (9)	0.00016 (6)	0.00039 (6)	−0.00106 (6)
O1	0.0240 (17)	0.0209 (15)	0.0128 (16)	−0.0051 (14)	0.0006 (13)	0.0014 (13)
O2	0.0149 (15)	0.0223 (16)	0.0193 (16)	0.0039 (13)	0.0005 (13)	0.0015 (14)
O3	0.0129 (14)	0.0216 (15)	0.0190 (17)	0.0029 (13)	0.0000 (13)	−0.0040 (14)
O4	0.0138 (15)	0.0233 (15)	0.0120 (16)	0.0025 (14)	−0.0002 (13)	−0.0054 (13)
O5	0.0216 (16)	0.0249 (16)	0.0089 (15)	0.0012 (13)	−0.0011 (13)	−0.0012 (13)
O6	0.0261 (18)	0.0266 (17)	0.0173 (17)	0.0002 (15)	0.0008 (14)	0.0083 (15)
O7	0.0230 (17)	0.0289 (18)	0.0260 (19)	0.0110 (14)	0.0042 (15)	0.0026 (15)
N1	0.0115 (16)	0.0133 (17)	0.0135 (18)	0.0012 (14)	0.0007 (14)	0.0015 (14)
N2	0.0154 (17)	0.0184 (17)	0.0144 (19)	−0.0051 (15)	−0.0009 (15)	−0.0043 (16)
N3	0.0167 (18)	0.0177 (18)	0.0134 (19)	−0.0033 (15)	0.0017 (15)	0.0001 (15)
N4	0.0104 (16)	0.0174 (17)	0.0120 (18)	0.0003 (14)	−0.0015 (14)	−0.0026 (15)
N5	0.0140 (18)	0.0236 (19)	0.0150 (19)	−0.0034 (16)	0.0033 (15)	0.0007 (16)
N6	0.027 (2)	0.033 (2)	0.019 (2)	−0.0034 (19)	−0.0013 (18)	−0.0018 (19)
C1	0.0110 (19)	0.0156 (19)	0.015 (2)	0.0022 (16)	0.0003 (16)	−0.0033 (18)
C2	0.012 (2)	0.023 (2)	0.020 (2)	0.0018 (18)	0.0005 (18)	0.0047 (19)
C3	0.013 (2)	0.016 (2)	0.027 (3)	−0.0022 (18)	0.0048 (19)	−0.0059 (19)
C4	0.013 (2)	0.016 (2)	0.020 (2)	−0.0016 (18)	−0.0021 (17)	−0.0070 (19)
C5	0.0103 (19)	0.020 (2)	0.017 (2)	0.0040 (17)	0.0032 (17)	−0.0005 (18)
C6	0.0065 (18)	0.017 (2)	0.015 (2)	0.0020 (16)	0.0026 (16)	0.0008 (17)
C7	0.0084 (18)	0.0141 (19)	0.012 (2)	0.0010 (16)	0.0011 (16)	−0.0027 (17)
C8	0.0112 (19)	0.018 (2)	0.014 (2)	0.0017 (17)	0.0039 (17)	0.0002 (18)
C9	0.0075 (19)	0.020 (2)	0.016 (2)	0.0015 (17)	0.0026 (17)	−0.0018 (18)
C10	0.013 (2)	0.023 (2)	0.014 (2)	−0.0004 (18)	0.0044 (17)	−0.0014 (19)
C11	0.014 (2)	0.020 (2)	0.022 (2)	−0.0016 (18)	0.0037 (18)	0.004 (2)
C12	0.013 (2)	0.0138 (19)	0.024 (2)	−0.0029 (17)	0.0018 (18)	−0.0033 (19)
C13	0.013 (2)	0.017 (2)	0.022 (2)	−0.0021 (18)	0.0002 (18)	−0.0036 (19)
C14	0.019 (2)	0.022 (2)	0.020 (2)	−0.0022 (19)	0.0042 (19)	0.001 (2)
C15	0.030 (3)	0.029 (3)	0.022 (3)	−0.007 (2)	−0.002 (2)	0.000 (2)
O8	0.049 (2)	0.0243 (18)	0.033 (2)	0.0018 (18)	0.0193 (19)	−0.0056 (18)

Geometric parameters (Å, º)

U1—O1	2.206 (3)	C2—H2A	0.9300
U1—O2	1.776 (3)	C2—C3	1.376 (6)
U1—O3	1.777 (3)	C3—H3	0.9300
U1—O4	2.390 (3)	C3—C4	1.386 (6)
U1—O5	2.467 (3)	C4—H4	0.9300
U1—N1	2.489 (4)	C4—C5	1.378 (6)
U1—N4	2.658 (4)	C5—H5	0.9300
O1—C1	1.319 (5)	C5—C6	1.394 (6)
O4—H4A	0.860 (2)	C6—C7	1.461 (5)
O4—H4B	0.860 (2)	C8—C9	1.451 (6)
O5—N5	1.313 (4)	C9—C10	1.385 (6)
O6—N5	1.226 (5)	C10—H10	0.9300
O7—N5	1.221 (5)	C10—C11	1.383 (6)
N1—C7	1.336 (5)	C11—H11	0.9300
N1—C8	1.360 (5)	C11—C12	1.368 (6)
N2—H2	0.8600	C12—H12	0.9300
N2—N3	1.357 (5)	C12—C13	1.391 (6)
N2—C7	1.336 (5)	C13—H13	0.9300
N3—C8	1.314 (5)	C14—C15	1.453 (6)
N4—C9	1.360 (5)	C15—H15A	0.9600
N4—C13	1.347 (5)	C15—H15B	0.9600
N6—C14	1.146 (6)	C15—H15C	0.9600
C1—C2	1.413 (6)	O8—H8A	0.860 (2)
C1—C6	1.405 (6)	O8—H8B	0.860 (2)
O1—U1—O4	152.83 (11)	C3—C2—C1	120.4 (4)
O1—U1—O5	77.17 (10)	C3—C2—H2A	119.8
O1—U1—N1	68.61 (11)	C2—C3—H3	119.3
O1—U1—N4	132.17 (11)	C2—C3—C4	121.5 (4)
O2—U1—O1	90.45 (12)	C4—C3—H3	119.3
O2—U1—O3	175.36 (14)	C3—C4—H4	120.6
O2—U1—O4	89.24 (12)	C5—C4—C3	118.8 (4)
O2—U1—O5	100.84 (12)	C5—C4—H4	120.6
O2—U1—N1	91.78 (13)	C4—C5—H5	119.3
O2—U1—N4	90.02 (12)	C4—C5—C6	121.3 (4)
O3—U1—O1	94.15 (13)	C6—C5—H5	119.3
O3—U1—O4	86.96 (12)	C1—C6—C7	118.7 (4)
O3—U1—O5	80.83 (12)	C5—C6—C1	120.1 (4)
O3—U1—N1	89.32 (13)	C5—C6—C7	121.2 (4)
O3—U1—N4	86.43 (13)	N1—C7—C6	126.9 (4)
O4—U1—O5	76.21 (10)	N2—C7—N1	107.7 (4)
O4—U1—N1	138.56 (11)	N2—C7—C6	125.3 (4)
O4—U1—N4	75.00 (11)	N1—C8—C9	120.5 (4)
O5—U1—N1	143.57 (10)	N3—C8—N1	113.7 (4)
O5—U1—N4	149.01 (10)	N3—C8—C9	125.7 (4)
N1—U1—N4	63.57 (10)	N4—C9—C8	114.9 (4)

C1—O1—U1	149.5 (3)	N4—C9—C10	122.4 (4)
U1—O4—H4A	129 (4)	C10—C9—C8	122.7 (4)
U1—O4—H4B	126 (3)	C9—C10—H10	120.6
H4A—O4—H4B	104 (4)	C11—C10—C9	118.8 (4)
N5—O5—U1	124.2 (2)	C11—C10—H10	120.6
C7—N1—U1	133.5 (3)	C10—C11—H11	120.2
C7—N1—C8	104.5 (4)	C12—C11—C10	119.6 (4)
C8—N1—U1	122.0 (3)	C12—C11—H11	120.2
N3—N2—H2	124.3	C11—C12—H12	120.5
C7—N2—H2	124.3	C11—C12—C13	119.0 (4)
C7—N2—N3	111.5 (3)	C13—C12—H12	120.5
C8—N3—N2	102.6 (3)	N4—C13—C12	122.7 (4)
C9—N4—U1	118.9 (3)	N4—C13—H13	118.6
C13—N4—U1	123.6 (3)	C12—C13—H13	118.6
C13—N4—C9	117.5 (4)	N6—C14—C15	178.4 (5)
O6—N5—O5	116.9 (4)	C14—C15—H15A	109.5
O7—N5—O5	118.6 (4)	C14—C15—H15B	109.5
O7—N5—O6	124.6 (4)	C14—C15—H15C	109.5
O1—C1—C2	119.5 (4)	H15A—C15—H15B	109.5
O1—C1—C6	122.5 (4)	H15A—C15—H15C	109.5
C6—C1—C2	118.0 (4)	H15B—C15—H15C	109.5
C1—C2—H2A	119.8	H8A—O8—H8B	102 (6)

U1—O1—C1—C2	172.3 (4)	C1—C6—C7—N1	4.2 (6)
U1—O1—C1—C6	−7.1 (8)	C1—C6—C7—N2	−177.7 (4)
U1—O5—N5—O6	175.9 (2)	C2—C1—C6—C5	−0.3 (6)
U1—O5—N5—O7	−4.3 (5)	C2—C1—C6—C7	180.0 (4)
U1—N1—C7—N2	179.1 (3)	C2—C3—C4—C5	−0.1 (6)
U1—N1—C7—C6	−2.6 (6)	C3—C4—C5—C6	−0.8 (6)
U1—N1—C8—N3	−179.2 (3)	C4—C5—C6—C1	1.0 (6)
U1—N1—C8—C9	1.8 (5)	C4—C5—C6—C7	−179.3 (4)
U1—N4—C9—C8	−0.6 (4)	C5—C6—C7—N1	−175.5 (4)
U1—N4—C9—C10	179.5 (3)	C5—C6—C7—N2	2.6 (6)
U1—N4—C13—C12	−179.3 (3)	C6—C1—C2—C3	−0.6 (6)
O1—C1—C2—C3	−180.0 (4)	C7—N1—C8—N3	−0.1 (5)
O1—C1—C6—C5	179.0 (4)	C7—N1—C8—C9	−179.2 (4)
O1—C1—C6—C7	−0.7 (6)	C7—N2—N3—C8	0.1 (4)
N1—C8—C9—N4	−0.7 (5)	C8—N1—C7—N2	0.2 (4)
N1—C8—C9—C10	179.2 (4)	C8—N1—C7—C6	178.5 (4)
N2—N3—C8—N1	0.0 (4)	C8—C9—C10—C11	−179.8 (4)
N2—N3—C8—C9	179.0 (4)	C9—N4—C13—C12	0.6 (6)
N3—N2—C7—N1	−0.2 (5)	C9—C10—C11—C12	0.1 (6)
N3—N2—C7—C6	−178.6 (4)	C10—C11—C12—C13	0.0 (6)
N3—C8—C9—N4	−179.6 (4)	C11—C12—C13—N4	−0.4 (6)
N3—C8—C9—C10	0.2 (6)	C13—N4—C9—C8	179.4 (4)
N4—C9—C10—C11	0.1 (6)	C13—N4—C9—C10	−0.4 (6)
C1—C2—C3—C4	0.8 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*A*···O5^{i}	0.86 (1)	1.92 (2)	2.752 (4)	162 (5)
O4—H4*B*···O8	0.86 (1)	1.73 (1)	2.581 (5)	168 (5)
N2—H2···N6	0.86	2.07	2.909 (5)	165
O8—H8*A*···N3^{ii}	0.86 (1)	2.05 (2)	2.890 (5)	164 (6)
O8—H8*B*···O6^{iii}	0.86 (1)	2.22 (3)	3.001 (5)	150 (6)

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