



Binary solvent participation in crystals of a multi-aromatic 1,2,3-triazole

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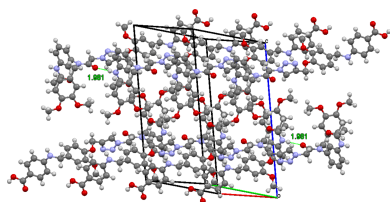
Keywords: crystal structure; click chemistry; three-center hydrogen bond.**CCDC reference:** 2408502**Supporting information:** this article has supporting information at journals.iucr.org/e

The X-ray crystal structure of a multi-aromatic substituted 1,2,3-triazole is presented, which shows an extensive three-dimensional hydrogen-bonding network involving two water molecules and two acetonitrile molecules. The structure of 4-[[[4-[[1-[[[(3,4-dimethoxyphenyl)methyl](3-acetamidophenyl)carbamoyl)methyl]-1*H*-1,2,3-triazol-4-yl]methoxy}-3-methoxyphenyl)methyl]-amino]benzoic acid–acetonitrile–water (1/2/2), $C_{37}H_{38}N_6O_8 \cdot 2C_2H_3N \cdot 2H_2O$, features amine-linked aromatic groups that have a variety of functionality including a carboxylic acid, an acetamido group, and methoxy ethers. All $X-H$ groups, and seven out of ten heteroatoms with available lone-pair electrons, participate in hydrogen bonding, with the aid of dimer-bridging water molecules and acetonitrile molecules whose methyl groups form close contacts with oxygen atoms. The triazole itself is a dimer made using click chemistry from readily available and inexpensive starting materials and is a precursor to larger oligomers, as well as to compounds with a wide array of readily manipulated functionality.

1. Chemical context

Foldamers, or folding oligomers, are synthetic organic molecules that have a propensity to form weak intramolecular interactions and have secondary structure analogous to biomacromolecules (Hill *et al.*, 2001; Gellman, 1998). With the goal of preparing functional foldamers, we prepared an unusual dimer molecule that crystallized easily from a mixture of acetonitrile and water (compound **1**). An X-ray structure determination was undertaken to understand whether or not its aromatic components stack intramolecularly (*i.e.*, fold). While the molecule adopts an extended conformation in the crystal, and therefore may be unlikely to form a folding nucleus for larger oligomers based upon it, certain features of the molecule and its interactions with solvent molecules are reported here.

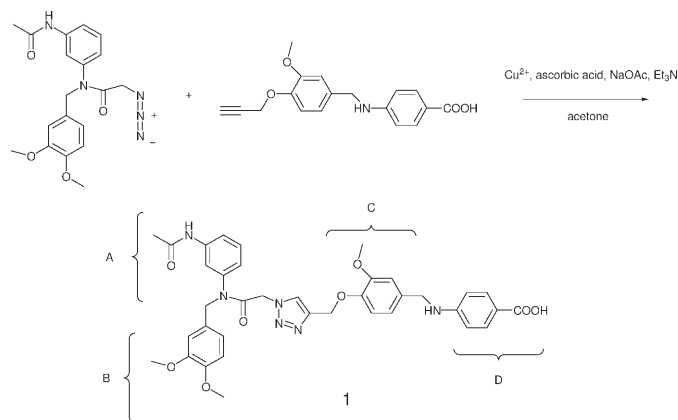
The linking chemistry used to make the title compound is the copper-catalyzed alkyne–azide cycloaddition reaction, also known as the CuAAC click reaction (Kolb *et al.*, 2001). It is composed of an aromatic 1,2,3-triazole substituted at the 1 and 4 positions with groups that each feature two aromatic rings bearing a variety of functionality. The main criteria for linking these groups are the ready availability and low cost of the starting materials, which are transformed into the moieties indicated with brackets, and are: *A*: 3-acetamidoaniline, *B*: 3,4-dimethoxybenzaldehyde, *C*: vanillin, and *D*: ethyl 4-amino-benzoate. The starting materials were also chosen for their functionality, and the ease with which they can be modified. For instance, the acetamido group in *A* could be changed to a longer chain amide (or an amide containing additional functionality), the 4-methoxy group in *B* can be changed to other



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ethers (including ones featuring alkynes) using vanillin as the starting material, the methoxy group in *C* can easily be obtained as an ethoxy group, which could be a useful spectroscopic handle, and leaving the carboxylic acid in *D* as an ester decreases its polarity. While the main objective for making the title compound was to use it as a starting point for making larger oligomers, it is also possible to envision it for preparing a large variety of molecules with a wide range of functionality.



2. Structural commentary

The extended conformation is shown in Fig. 1, which also shows two water molecules and two acetonitrile molecules as solvents of crystallization. The molecular structure confirms the cycloaddition reaction proceeds with the expected regiochemistry to give a 1,4-substituted 1,2,3-triazole. The amine nitrogen atom is essentially planar [C6—C5—N1—C8 torsion angle = 0.0 (2)°], indicating the lone pair electrons are conjugated with the aromatic ring, facilitated by the electron withdrawing carboxylic acid, while the aromatic ring attached at the amide nitrogen atom is essentially non-planar [C20—N5—C30—C31 torsion angle = 107.1 (2)°] indicating the amide lone pair is not conjugated to the aromatic ring.

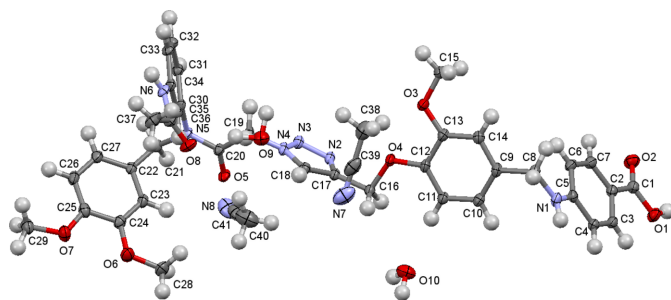


Figure 1

A view of the title compound with two water and two acetonitrile molecules as solvents of crystallization. Atoms are displayed as ellipsoids at the 50% probability displacement level.

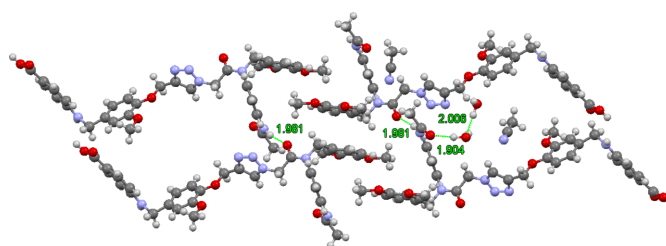


Figure 2

Four molecules highlighting the acetamido–amide hydrogen bond, the water dimer bound to the acetamide carbonyl group, π -stacking, and the solvents of crystallization.

3. Supramolecular features

The crystal packing in Fig. 2 shows four molecules with solvent molecules included on the right side of the image. It can be seen that an acetonitrile molecule helps fill the void space between two alkoxy benzenes, the water molecules are hydrogen bonded together [2.01 (2) Å, see Table 1], and one molecule of this pair is hydrogen bonded to an acetamide carbonyl [1.90 (3) Å]. The amide hydrogen atom of this acetamide is hydrogen bonded [1.98 (2) Å] to the linking amide adjacent to the triazole of the next molecule in the crystal. The dimethoxy benzenes are π -stacked in a face-to-face manner on a pairwise basis, with the local dipoles oriented antiparallel, and with the methylene carbon atom (C21) positioned almost directly over an ether oxygen atom (O7). The second pair of stacked dimethoxy benzenes is offset several bond lengths such that a methyl group (C29) is located almost directly over a benzene ring carbon (C27).

Fig. 3 shows four molecules focusing on the hydrogen bonding to the triazole, as well as the bridge hydrogen bonding facilitated by the two water molecules. The carboxylic acid hydrogen bonds to the 3-position of the triazole [1.87 (3) Å] and the amine NH forms a hydrogen bond to the 2-position of the triazole [2.29 (2) Å]. The carboxylic acid serves as an hydrogen-bond acceptor from a water molecule [1.80 (3) Å]. This water molecule forms the hydrogen bond noted above to the other water molecule, which forms a three-center hydrogen bond to the dimethoxy benzene of the fourth molecule displayed [2.12 (3), 2.17 (3) Å]. In total, seven of the ten heteroatoms of the title compound with available lone-

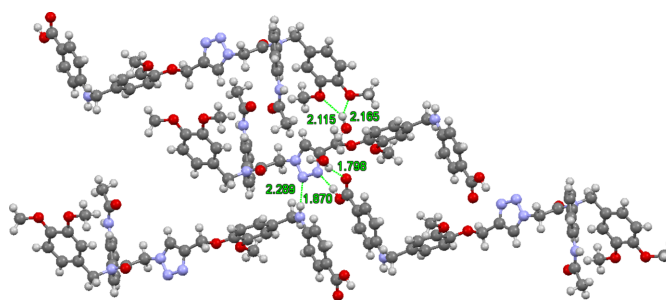


Figure 3

Four molecules showing hydrogen bonding to the 1,2,3-triazole, water hydrogen bonding to the carboxylic acid carbonyl group, and water participating in the three-center hydrogen bond.

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O9—H9A...O2 ⁱ	0.96 (3)	1.80 (3)	2.7173 (17)	160 (2)
O9—H9B...O8	0.99 (3)	1.90 (3)	2.8610 (18)	164 (2)
O10—H10A...O6 ⁱⁱ	0.96 (3)	2.12 (3)	2.9441 (17)	144 (2)
O10—H10A...O7 ⁱⁱ	0.96 (3)	2.17 (3)	2.9928 (17)	143 (2)
O10—H10B...O9 ⁱⁱⁱ	0.95 (3)	2.00 (3)	2.9346 (19)	167 (2)
N1—H1A...N3 ^{iv}	0.90 (2)	2.29 (2)	3.1834 (17)	174.6 (18)
N6—H6...O5 ^v	0.85 (2)	1.98 (2)	2.8200 (15)	168.6 (19)
O1—H1...N2 ^{vi}	0.91 (3)	1.87 (3)	2.7614 (16)	166 (3)

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

pair electrons behave as hydrogen-bond acceptors, creating an extensive three-dimensional hydrogen-bonded network. The dimethoxy benzene π -stacking in Fig. 2 suggests molecules with multi-aromatic side chains bearing dimethoxy benzene units could encourage folding.

The close contacts between the C38 hydrogen atoms of an acetonitrile molecule and oxygen atoms are highlighted in Fig. 4. The oxygen atoms are O6 of the dialkoxy benzene unit [2.50 (3) Å], and O2 of the carboxylic acid [2.59 (3) Å]. Two molecules are intertwined about the dialkoxy benzene units with symmetrically disposed acetonitrile molecules. Fig. 5 shows the unit cell superimposed on amide-to-amide hydrogen bonded tetramers with the solvent molecules omitted, with the top unit oriented with N1, N2, and N3 of the triazoles facing the viewer, and the same atoms of the bottom unit facing away from the viewer. Images of molecular structures were manipulated using *Mercury* (Macrae *et al.*, 2020).

4. Database survey

Since crystalline carboxylic acids have a tendency to form dimers analogous to the well-known acetic acid dimer, as in the case of 5-bromo-2-(phenylamino)benzoic acid (Kang & Long, 2024), it is notable that the carboxylic acid of the title compound forms a hydrogen bond to the triazole. Carboxylic acids hydrogen bonded to heterocyclic nitrogen atoms (Ladraa *et al.*, 2010) and to the 3-position of 1,2,3-triazoles (Lin, 2010) have been examined. A 1,2,3-triazole click reaction product has also been structurally characterized (Zukerman-Schpector *et al.*, 2017). A similar arrangement to the π -stacking with dipoles aligned anti-parallel seen for the dimethoxy benzene units has been observed in 2-methoxy-5-nitroaniline (Filley, 2024).

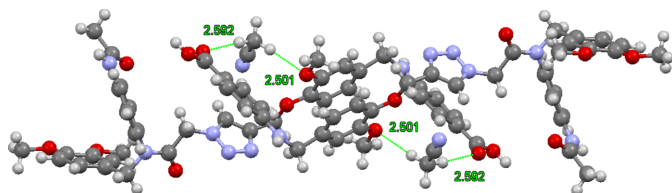


Figure 4

An intertwined dimer showing close contacts between an acetonitrile molecule and oxygen atoms.

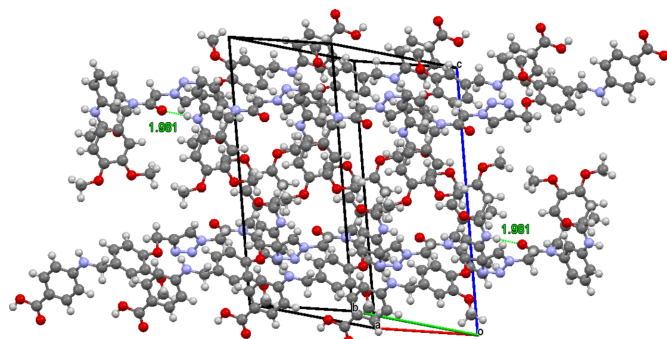


Figure 5

Unit cell superimposed on two sets of hydrogen bonded tetramers, showing the amide hydrogen bonding.

5. Synthesis and crystallization

The azide and alkyne in the scheme were prepared in seven steps as detailed in the supporting information. Reductive aminations were performed according to Touchette (2006). The title compound was made as follows: A 50 ml round-bottomed flask was charged with 0.767 g (2.00 mmole) of *N*-(3-acetamidophenyl)-*N*-(3,4-dimethoxybenzyl)azidoacetamide, 0.623 g (2.00 mmole) of 4-(4-propargyloxy-3-methoxybenzyl-amino)benzoic acid, 0.28 ml (2.0 mmole) of triethylamine and 20 ml of acetone. The solids were dissolved with stirring and treated with 2.0 ml of an aqueous solution containing a mixture of 0.1 *M* ascorbic acid and 0.1 *M* sodium acetate, the buffered mixture was then treated with 0.4 ml of aqueous 0.1 *M* CuSO₄. The flask was flushed with N₂, capped with a septum, and stirred overnight. The resulting inhomogeneous mixture was poured into 100 ml of water containing 15 mg of Na₂EDTA and 300 mg of NaOH, giving a clear solution. When this solution was acidified with 5 ml of 6*M* HCl, a gooey precipitate resulted, which was isolated by decanting off the aqueous solution, boiling in 10 ml of ethanol, and pouring the hot ethanol solution into 100 ml of water followed by about 15 ml of saturated NaCl. The resultant precipitate was filtered off and allowed to air dry, 1.23 g (88%) snow white powder. ¹H NMR (400 MHz, CD₃CN/1% D₂O): 2.04 (*s*, 3H), 3.70 (*s*, 3H), 3.73 (*s*, 3H), 3.74 (*s*, 3H), 4.27 (*s*, 2H), 4.80 (*s*, 2H), 4.96 (*s*, 2H), 5.02 (*s*, 2H), 6.60–7.75 (*m*, 14H), 7.84 (*s*, 1H). ¹³C NMR (CD₃CN/1% D₂O): 23.3, 46.2, 51.5, 52.6, 55.1, 55.2, 55.3, 61.9, 111.2, 111.4, 111.6, 112.0, 113.6, 117.3, 117.5, 119.1, 119.2, 119.3, 121.0, 123.4, 125.9, 129.2, 130.1, 131.5, 132.5, 140.2, 140.6, 143.1, 146.7, 148.5, 149.0, 149.5, 152.5, 165.4, 167.7, 169.3. X-ray quality crystals can be obtained by dissolving 20 mg of the product in 0.8 ml of acetonitrile + 1 drop water, followed by the addition of 0.8 ml additional water, and setting aside for several days at room temperature. The solvents of crystallization should be preserved by not allowing the crystals to dry out.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H-atom parameters were refined.

Acknowledgements

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Table 2

Experimental details.

Crystal data	
Chemical formula	C ₃₇ H ₃₈ N ₆ O ₈ ·2C ₂ H ₃ N·2H ₂ O
<i>M</i> _r	812.87
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	9.1493 (2), 12.3058 (3), 20.1432 (5)
α , β , γ (°)	76.076 (2), 84.791 (2), 71.829 (2)
<i>V</i> (Å ³)	2091.19 (10)
<i>Z</i>	2
Radiation type	Cu <i>K</i> α
μ (mm ^{−1})	0.78
Crystal size (mm)	0.9 × 0.3 × 0.05
Data collection	
Diffractometer	Xcalibur, Onyx, Ultra
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> , Agilent, 2014)
<i>T</i> _{min} , <i>T</i> _{max}	0.728, 1.000
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	32961, 8593, 7480
<i>R</i> _{int}	0.048
(sin θ/λ) _{max} (Å ^{−1})	0.630
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.045, 0.130, 1.04
No. of reflections	8593
No. of parameters	724
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ^{−3})	0.32, −0.25

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXT* (Sheldrick, 2015a), *SHELXL2013/4* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

supporting information

Acta Cryst. (2025). E81, 38–41 [https://doi.org/10.1107/S2056989024011915]

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Computing details

4-[[[4-[[1-([[(3,4-Dimethoxyphenyl)methyl](3-acetamidophenyl)carbamoyl)methyl]-1*H*-1,2,3-triazol-4-yl)methoxy]-3-methoxyphenyl)methyl]amino]benzoic acid–acetonitrile–water (1/2/2)

Crystal data

$C_{37}H_{38}N_6O_8 \cdot 2C_2H_3N \cdot 2H_2O$

$M_r = 812.87$

Triclinic, $P\bar{1}$

$a = 9.1493$ (2) Å

$b = 12.3058$ (3) Å

$c = 20.1432$ (5) Å

$\alpha = 76.076$ (2)°

$\beta = 84.791$ (2)°

$\gamma = 71.829$ (2)°

$V = 2091.19$ (10) Å³

$Z = 2$

$F(000) = 860$

$D_x = 1.291$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 15358 reflections

$\theta = 3.8\text{--}75.8^\circ$

$\mu = 0.78$ mm⁻¹

$T = 100$ K

Plate, clear colourless

$0.9 \times 0.3 \times 0.05$ mm

Data collection

Xcalibur, Onyx, Ultra
diffractometer

Detector resolution: 8.2603 pixels mm⁻¹

ω scans

Absorption correction: multi-scan
(CrysAlisPro, Agilent, 2014)

$T_{\min} = 0.728$, $T_{\max} = 1.000$

32961 measured reflections

8593 independent reflections

7480 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\max} = 76.4^\circ$, $\theta_{\min} = 3.9^\circ$

$h = -11 \rightarrow 11$

$k = -15 \rightarrow 15$

$l = -25 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.130$

$S = 1.04$

8593 reflections

724 parameters

0 restraints

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.6126P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.32$ e Å⁻³

$\Delta\rho_{\min} = -0.25$ e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	1.17136 (13)	0.88775 (10)	−0.09107 (6)	0.0306 (2)
O2	1.09733 (13)	0.79667 (11)	−0.15897 (6)	0.0352 (3)
O3	0.27550 (12)	0.47527 (9)	0.04633 (5)	0.0271 (2)
O4	0.43548 (12)	0.35313 (8)	0.15043 (5)	0.0260 (2)
O5	0.52857 (11)	−0.19991 (9)	0.30496 (5)	0.0246 (2)
O6	0.33685 (14)	−0.32839 (10)	0.56423 (6)	0.0334 (2)
O7	0.19717 (14)	−0.48177 (10)	0.60142 (6)	0.0337 (2)
O8	−0.13498 (12)	−0.02355 (11)	0.38717 (6)	0.0329 (3)
O9	−0.00690 (15)	0.13658 (12)	0.29127 (7)	0.0398 (3)
H9A	−0.045 (3)	0.144 (2)	0.2472 (14)	0.059 (7)*
H9B	−0.049 (3)	0.074 (3)	0.3171 (15)	0.069 (8)*
O10	0.76143 (15)	0.36388 (12)	0.29092 (6)	0.0416 (3)
H10A	0.745 (3)	0.382 (2)	0.3354 (14)	0.060 (7)*
H10B	0.840 (3)	0.291 (2)	0.2981 (13)	0.055 (7)*
N1	0.54927 (14)	0.86351 (10)	0.06410 (7)	0.0253 (3)
H1A	0.548 (2)	0.8990 (19)	0.0983 (11)	0.035 (5)*
N2	0.57880 (13)	0.09002 (10)	0.17503 (6)	0.0220 (2)
N3	0.52279 (13)	0.00184 (10)	0.18158 (6)	0.0224 (2)
N4	0.40357 (12)	0.01865 (10)	0.22590 (6)	0.0211 (2)
N5	0.33120 (12)	−0.26564 (10)	0.29033 (6)	0.0197 (2)
N6	−0.22795 (13)	−0.11497 (10)	0.32250 (6)	0.0220 (2)
H6	−0.309 (2)	−0.1305 (17)	0.3157 (10)	0.030 (5)*
N7	0.1766 (2)	0.37004 (18)	0.29711 (11)	0.0564 (5)
N8	0.23890 (19)	−0.01332 (18)	0.40928 (10)	0.0548 (5)
C1	1.07409 (16)	0.83931 (13)	−0.10892 (7)	0.0249 (3)
C2	0.94033 (16)	0.84063 (12)	−0.06241 (7)	0.0237 (3)
C3	0.92719 (17)	0.88189 (12)	−0.00259 (7)	0.0242 (3)
H3	1.007 (2)	0.9052 (16)	0.0086 (9)	0.027 (4)*
C4	0.79857 (16)	0.88776 (12)	0.03891 (7)	0.0234 (3)
H4	0.790 (2)	0.9183 (16)	0.0795 (10)	0.027 (4)*
C5	0.67743 (16)	0.85103 (11)	0.02278 (7)	0.0218 (3)
C6	0.69374 (16)	0.80324 (12)	−0.03534 (7)	0.0243 (3)
H6A	0.618 (2)	0.7733 (16)	−0.0472 (9)	0.024 (4)*
C7	0.82255 (17)	0.80019 (13)	−0.07722 (7)	0.0248 (3)
H7	0.833 (2)	0.7680 (16)	−0.1162 (9)	0.025 (4)*
C8	0.41784 (16)	0.82927 (12)	0.05275 (8)	0.0258 (3)
H8A	0.396 (2)	0.8517 (17)	0.0036 (11)	0.033 (5)*
H8B	0.325 (2)	0.8750 (17)	0.0789 (10)	0.033 (5)*
C9	0.43676 (15)	0.69880 (12)	0.07817 (7)	0.0221 (3)
C10	0.52712 (15)	0.63117 (12)	0.13346 (7)	0.0225 (3)
H10	0.590 (2)	0.6636 (16)	0.1549 (9)	0.027 (4)*
C11	0.53054 (15)	0.51384 (12)	0.16017 (7)	0.0219 (3)
H11	0.593 (2)	0.4681 (17)	0.1983 (10)	0.028 (4)*
C12	0.44282 (15)	0.46574 (12)	0.13011 (7)	0.0214 (3)
C13	0.35354 (15)	0.53324 (12)	0.07247 (7)	0.0219 (3)

C14	0.35076 (16)	0.64869 (12)	0.04727 (7)	0.0228 (3)
H14	0.290 (2)	0.6954 (17)	0.0081 (10)	0.028 (4)*
C15	0.19367 (19)	0.53567 (14)	−0.01514 (8)	0.0301 (3)
H15A	0.153 (2)	0.4791 (19)	−0.0284 (11)	0.040 (5)*
H15B	0.262 (2)	0.5605 (18)	−0.0502 (11)	0.036 (5)*
H15C	0.106 (2)	0.6029 (18)	−0.0067 (10)	0.032 (5)*
C16	0.51982 (16)	0.27934 (12)	0.20980 (7)	0.0227 (3)
H16A	0.629 (2)	0.2738 (16)	0.2041 (9)	0.025 (4)*
H16B	0.480 (2)	0.3104 (16)	0.2517 (10)	0.027 (4)*
C17	0.49516 (15)	0.16367 (12)	0.21461 (7)	0.0212 (3)
C18	0.38211 (15)	0.11808 (12)	0.24765 (7)	0.0222 (3)
H18	0.303 (2)	0.1451 (17)	0.2770 (10)	0.028 (4)*
C19	0.31026 (15)	−0.06093 (12)	0.24188 (8)	0.0236 (3)
H19A	0.218 (2)	−0.0224 (18)	0.2680 (10)	0.036 (5)*
H19B	0.276 (2)	−0.0694 (16)	0.1975 (10)	0.028 (4)*
C20	0.40083 (14)	−0.18197 (12)	0.28243 (7)	0.0201 (3)
C21	0.40887 (15)	−0.38716 (12)	0.32711 (7)	0.0223 (3)
H21A	0.3859 (19)	−0.4391 (15)	0.3010 (9)	0.020 (4)*
H21B	0.519 (2)	−0.3942 (15)	0.3252 (9)	0.020 (4)*
C22	0.34860 (15)	−0.41398 (12)	0.39953 (7)	0.0218 (3)
C23	0.37111 (15)	−0.35337 (12)	0.44665 (7)	0.0235 (3)
H23	0.425 (2)	−0.2960 (17)	0.4345 (10)	0.031 (5)*
C24	0.31919 (16)	−0.37859 (12)	0.51336 (8)	0.0252 (3)
C25	0.24218 (16)	−0.46476 (13)	0.53414 (8)	0.0269 (3)
C26	0.21904 (17)	−0.52333 (13)	0.48759 (8)	0.0283 (3)
H26	0.164 (2)	−0.5831 (19)	0.5014 (11)	0.039 (5)*
C27	0.27249 (16)	−0.49770 (13)	0.42003 (8)	0.0265 (3)
H27	0.255 (2)	−0.5410 (17)	0.3872 (10)	0.029 (4)*
C28	0.4173 (2)	−0.24290 (15)	0.54789 (9)	0.0375 (4)
H28A	0.362 (2)	−0.1763 (19)	0.5127 (11)	0.038 (5)*
H28B	0.420 (3)	−0.219 (2)	0.5891 (13)	0.052 (6)*
H28C	0.526 (3)	−0.277 (2)	0.5303 (12)	0.050 (6)*
C29	0.14031 (19)	−0.57922 (15)	0.62854 (9)	0.0339 (3)
H29A	0.043 (3)	−0.5705 (19)	0.6095 (11)	0.041 (5)*
H29B	0.129 (2)	−0.5808 (18)	0.6776 (11)	0.036 (5)*
H29C	0.221 (2)	−0.6525 (19)	0.6222 (10)	0.035 (5)*
C30	0.17665 (14)	−0.24101 (11)	0.26830 (7)	0.0190 (3)
C31	0.15460 (15)	−0.27707 (12)	0.21066 (7)	0.0217 (3)
H31	0.238 (2)	−0.3187 (16)	0.1868 (9)	0.026 (4)*
C32	0.00458 (16)	−0.25343 (13)	0.18981 (7)	0.0237 (3)
H32	−0.013 (2)	−0.2783 (15)	0.1487 (9)	0.023 (4)*
C33	−0.11947 (15)	−0.19647 (12)	0.22621 (7)	0.0226 (3)
H33	−0.223 (2)	−0.1797 (15)	0.2128 (9)	0.022 (4)*
C34	−0.09591 (15)	−0.16253 (11)	0.28466 (7)	0.0198 (3)
C35	0.05423 (15)	−0.18473 (11)	0.30612 (7)	0.0198 (3)
H35	0.0685 (18)	−0.1614 (14)	0.3472 (9)	0.017 (4)*
C36	−0.23841 (16)	−0.05732 (13)	0.37295 (7)	0.0251 (3)
C37	−0.39062 (19)	−0.03660 (17)	0.41090 (10)	0.0367 (4)

H37A	−0.449 (3)	−0.081 (2)	0.4053 (12)	0.050 (6)*
H37B	−0.459 (3)	0.044 (3)	0.3891 (15)	0.074 (8)*
H37C	−0.378 (3)	−0.026 (3)	0.4552 (15)	0.069 (8)*
C38	0.0199 (2)	0.43962 (19)	0.18561 (11)	0.0466 (4)
H38A	−0.021 (3)	0.377 (2)	0.1815 (14)	0.062 (7)*
H38B	0.089 (3)	0.456 (3)	0.1440 (16)	0.075 (9)*
H38C	−0.070 (4)	0.508 (3)	0.1896 (15)	0.077 (9)*
C39	0.1066 (2)	0.40144 (17)	0.24797 (11)	0.0424 (4)
C40	0.0287 (3)	0.1594 (3)	0.44546 (12)	0.0588 (6)
H40A	0.065 (3)	0.223 (3)	0.4272 (15)	0.064 (8)*
H40B	0.035 (4)	0.156 (3)	0.4940 (19)	0.091 (10)*
H40C	−0.071 (4)	0.163 (3)	0.4316 (18)	0.091 (10)*
C41	0.1481 (2)	0.06327 (19)	0.42515 (9)	0.0446 (4)
H1	1.250 (3)	0.887 (2)	−0.1221 (14)	0.064 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0311 (5)	0.0353 (6)	0.0329 (6)	−0.0196 (5)	0.0045 (4)	−0.0105 (5)
O2	0.0362 (6)	0.0463 (7)	0.0314 (6)	−0.0201 (5)	0.0051 (4)	−0.0159 (5)
O3	0.0324 (5)	0.0224 (5)	0.0296 (5)	−0.0128 (4)	−0.0083 (4)	−0.0028 (4)
O4	0.0312 (5)	0.0170 (5)	0.0316 (5)	−0.0117 (4)	−0.0086 (4)	0.0003 (4)
O5	0.0168 (4)	0.0234 (5)	0.0345 (5)	−0.0088 (4)	−0.0057 (4)	−0.0025 (4)
O6	0.0450 (6)	0.0288 (6)	0.0304 (5)	−0.0154 (5)	0.0025 (5)	−0.0094 (4)
O7	0.0404 (6)	0.0292 (6)	0.0307 (5)	−0.0144 (5)	0.0049 (5)	−0.0021 (4)
O8	0.0261 (5)	0.0434 (6)	0.0404 (6)	−0.0178 (5)	0.0028 (4)	−0.0218 (5)
O9	0.0431 (7)	0.0417 (7)	0.0372 (6)	−0.0162 (5)	−0.0080 (5)	−0.0063 (5)
O10	0.0418 (7)	0.0474 (7)	0.0309 (6)	−0.0043 (6)	−0.0078 (5)	−0.0091 (5)
N1	0.0298 (6)	0.0197 (6)	0.0297 (6)	−0.0121 (5)	0.0026 (5)	−0.0066 (5)
N2	0.0206 (5)	0.0183 (5)	0.0281 (6)	−0.0088 (4)	−0.0020 (4)	−0.0026 (4)
N3	0.0196 (5)	0.0197 (5)	0.0288 (6)	−0.0082 (4)	−0.0004 (4)	−0.0041 (4)
N4	0.0180 (5)	0.0180 (5)	0.0278 (6)	−0.0080 (4)	−0.0008 (4)	−0.0024 (4)
N5	0.0158 (5)	0.0173 (5)	0.0264 (6)	−0.0065 (4)	−0.0033 (4)	−0.0028 (4)
N6	0.0153 (5)	0.0238 (6)	0.0306 (6)	−0.0093 (4)	−0.0010 (4)	−0.0084 (5)
N7	0.0436 (9)	0.0611 (11)	0.0759 (13)	−0.0141 (8)	0.0043 (9)	−0.0409 (10)
N8	0.0297 (8)	0.0633 (11)	0.0567 (10)	−0.0044 (8)	−0.0040 (7)	0.0026 (9)
C1	0.0266 (7)	0.0235 (7)	0.0256 (7)	−0.0101 (5)	−0.0030 (5)	−0.0029 (5)
C2	0.0254 (6)	0.0209 (6)	0.0252 (7)	−0.0089 (5)	−0.0030 (5)	−0.0025 (5)
C3	0.0272 (7)	0.0204 (6)	0.0279 (7)	−0.0110 (5)	−0.0057 (5)	−0.0034 (5)
C4	0.0298 (7)	0.0195 (6)	0.0231 (6)	−0.0107 (5)	−0.0036 (5)	−0.0034 (5)
C5	0.0258 (6)	0.0142 (6)	0.0242 (6)	−0.0071 (5)	−0.0031 (5)	0.0002 (5)
C6	0.0246 (6)	0.0223 (7)	0.0296 (7)	−0.0103 (5)	−0.0040 (5)	−0.0067 (5)
C7	0.0278 (7)	0.0239 (7)	0.0252 (7)	−0.0097 (5)	−0.0037 (5)	−0.0062 (5)
C8	0.0246 (7)	0.0192 (7)	0.0333 (8)	−0.0086 (5)	0.0012 (6)	−0.0035 (6)
C9	0.0202 (6)	0.0184 (6)	0.0279 (7)	−0.0074 (5)	0.0039 (5)	−0.0048 (5)
C10	0.0199 (6)	0.0204 (6)	0.0294 (7)	−0.0083 (5)	0.0009 (5)	−0.0071 (5)
C11	0.0208 (6)	0.0189 (6)	0.0264 (7)	−0.0071 (5)	−0.0008 (5)	−0.0041 (5)
C12	0.0227 (6)	0.0157 (6)	0.0266 (6)	−0.0084 (5)	0.0009 (5)	−0.0033 (5)

C13	0.0213 (6)	0.0210 (6)	0.0257 (6)	−0.0087 (5)	−0.0002 (5)	−0.0062 (5)
C14	0.0226 (6)	0.0211 (6)	0.0241 (6)	−0.0069 (5)	0.0004 (5)	−0.0037 (5)
C15	0.0338 (8)	0.0310 (8)	0.0286 (7)	−0.0139 (7)	−0.0072 (6)	−0.0043 (6)
C16	0.0228 (6)	0.0178 (6)	0.0279 (7)	−0.0085 (5)	−0.0044 (5)	−0.0015 (5)
C17	0.0201 (6)	0.0179 (6)	0.0259 (6)	−0.0070 (5)	−0.0032 (5)	−0.0025 (5)
C18	0.0209 (6)	0.0174 (6)	0.0274 (7)	−0.0059 (5)	−0.0017 (5)	−0.0029 (5)
C19	0.0175 (6)	0.0200 (6)	0.0335 (7)	−0.0090 (5)	−0.0033 (5)	−0.0010 (5)
C20	0.0166 (6)	0.0202 (6)	0.0244 (6)	−0.0071 (5)	−0.0011 (5)	−0.0045 (5)
C21	0.0184 (6)	0.0175 (6)	0.0300 (7)	−0.0047 (5)	−0.0036 (5)	−0.0034 (5)
C22	0.0176 (6)	0.0175 (6)	0.0282 (7)	−0.0036 (5)	−0.0049 (5)	−0.0017 (5)
C23	0.0224 (6)	0.0173 (6)	0.0307 (7)	−0.0076 (5)	−0.0019 (5)	−0.0024 (5)
C24	0.0249 (6)	0.0200 (6)	0.0302 (7)	−0.0053 (5)	−0.0022 (5)	−0.0058 (5)
C25	0.0246 (7)	0.0226 (7)	0.0290 (7)	−0.0049 (5)	−0.0005 (5)	−0.0004 (5)
C26	0.0264 (7)	0.0243 (7)	0.0350 (8)	−0.0130 (6)	−0.0037 (6)	0.0002 (6)
C27	0.0260 (7)	0.0224 (7)	0.0319 (7)	−0.0092 (5)	−0.0073 (6)	−0.0026 (6)
C28	0.0511 (10)	0.0288 (8)	0.0385 (9)	−0.0171 (7)	−0.0040 (8)	−0.0108 (7)
C29	0.0319 (8)	0.0321 (8)	0.0341 (8)	−0.0131 (7)	0.0004 (6)	0.0034 (6)
C30	0.0166 (6)	0.0162 (6)	0.0242 (6)	−0.0079 (5)	−0.0029 (5)	−0.0001 (5)
C31	0.0196 (6)	0.0212 (6)	0.0241 (6)	−0.0069 (5)	−0.0002 (5)	−0.0042 (5)
C32	0.0232 (6)	0.0259 (7)	0.0236 (6)	−0.0085 (5)	−0.0037 (5)	−0.0059 (5)
C33	0.0179 (6)	0.0241 (7)	0.0273 (7)	−0.0089 (5)	−0.0039 (5)	−0.0043 (5)
C34	0.0172 (6)	0.0178 (6)	0.0249 (6)	−0.0072 (5)	−0.0007 (5)	−0.0028 (5)
C35	0.0187 (6)	0.0188 (6)	0.0238 (6)	−0.0090 (5)	−0.0022 (5)	−0.0034 (5)
C36	0.0216 (6)	0.0272 (7)	0.0293 (7)	−0.0100 (5)	0.0010 (5)	−0.0084 (6)
C37	0.0266 (7)	0.0482 (10)	0.0472 (10)	−0.0189 (7)	0.0108 (7)	−0.0268 (8)
C38	0.0472 (10)	0.0452 (11)	0.0556 (11)	−0.0218 (9)	0.0120 (9)	−0.0208 (9)
C39	0.0365 (9)	0.0405 (10)	0.0609 (12)	−0.0156 (7)	0.0110 (8)	−0.0307 (9)
C40	0.0542 (13)	0.0686 (16)	0.0432 (11)	−0.0025 (11)	−0.0065 (9)	−0.0120 (11)
C41	0.0292 (8)	0.0584 (12)	0.0374 (9)	−0.0112 (8)	−0.0062 (7)	0.0054 (8)

Geometric parameters (Å, °)

O1—C1	1.3300 (17)	C13—C14	1.382 (2)
O1—H1	0.91 (3)	C14—H14	0.961 (19)
O2—C1	1.2178 (18)	C15—H15A	0.98 (2)
O3—C13	1.3648 (16)	C15—H15B	0.96 (2)
O3—C15	1.4228 (18)	C15—H15C	0.99 (2)
O4—C12	1.3683 (16)	C16—H16A	0.975 (19)
O4—C16	1.4347 (17)	C16—H16B	1.002 (19)
O5—C20	1.2280 (16)	C16—C17	1.4887 (19)
O6—C24	1.3619 (18)	C17—C18	1.373 (2)
O6—C28	1.425 (2)	C18—H18	0.922 (19)
O7—C25	1.3661 (19)	C19—H19A	1.00 (2)
O7—C29	1.424 (2)	C19—H19B	1.012 (19)
O8—C36	1.2255 (18)	C19—C20	1.5297 (19)
O9—H9A	0.96 (3)	C21—H21A	0.997 (17)
O9—H9B	0.99 (3)	C21—H21B	0.979 (17)
O10—H10A	0.96 (3)	C21—C22	1.509 (2)

O10—H10B	0.95 (3)	C22—C23	1.4041 (19)
N1—H1A	0.90 (2)	C22—C27	1.381 (2)
N1—C5	1.3650 (19)	C23—H23	0.96 (2)
N1—C8	1.4450 (19)	C23—C24	1.379 (2)
N2—N3	1.3127 (16)	C24—C25	1.412 (2)
N2—C17	1.3590 (18)	C25—C26	1.379 (2)
N3—N4	1.3419 (16)	C26—H26	0.99 (2)
N4—C18	1.3492 (18)	C26—C27	1.400 (2)
N4—C19	1.4530 (17)	C27—H27	0.995 (19)
N5—C20	1.3428 (17)	C28—H28A	0.98 (2)
N5—C21	1.4798 (17)	C28—H28B	0.95 (3)
N5—C30	1.4387 (16)	C28—H28C	1.02 (2)
N6—H6	0.85 (2)	C29—H29A	0.96 (2)
N6—C34	1.4104 (17)	C29—H29B	0.98 (2)
N6—C36	1.3537 (18)	C29—H29C	1.00 (2)
N7—C39	1.147 (3)	C30—C31	1.3900 (19)
N8—C41	1.135 (3)	C30—C35	1.3891 (19)
C1—C2	1.470 (2)	C31—H31	0.940 (19)
C2—C3	1.398 (2)	C31—C32	1.3935 (19)
C2—C7	1.4007 (19)	C32—H32	0.991 (18)
C3—H3	0.926 (19)	C32—C33	1.385 (2)
C3—C4	1.373 (2)	C33—H33	0.955 (18)
C4—H4	0.967 (19)	C33—C34	1.3949 (19)
C4—C5	1.4096 (19)	C34—C35	1.4019 (18)
C5—C6	1.4083 (19)	C35—H35	0.971 (17)
C6—H6A	0.953 (18)	C36—C37	1.506 (2)
C6—C7	1.382 (2)	C37—H37A	0.91 (3)
C7—H7	0.946 (18)	C37—H37B	1.01 (3)
C8—H8A	0.98 (2)	C37—H37C	0.95 (3)
C8—H8B	1.04 (2)	C38—H38A	0.98 (3)
C8—C9	1.5222 (19)	C38—H38B	1.02 (3)
C9—C10	1.382 (2)	C38—H38C	0.99 (3)
C9—C14	1.4010 (19)	C38—C39	1.446 (3)
C10—H10	0.971 (19)	C40—H40A	0.93 (3)
C10—C11	1.4057 (19)	C40—H40B	0.98 (4)
C11—H11	0.95 (2)	C40—H40C	0.96 (4)
C11—C12	1.3851 (19)	C40—C41	1.451 (3)
C12—C13	1.4106 (19)		
C1—O1—H1	110.4 (17)	N4—C19—H19B	108.4 (11)
C13—O3—C15	117.34 (11)	N4—C19—C20	111.40 (11)
C12—O4—C16	117.77 (10)	H19A—C19—H19B	109.4 (15)
C24—O6—C28	117.66 (12)	C20—C19—H19A	112.2 (12)
C25—O7—C29	117.23 (13)	C20—C19—H19B	109.1 (11)
H9A—O9—H9B	98 (2)	O5—C20—N5	123.36 (12)
H10A—O10—H10B	103 (2)	O5—C20—C19	121.54 (12)
C5—N1—H1A	115.9 (13)	N5—C20—C19	115.09 (11)
C5—N1—C8	123.62 (12)	N5—C21—H21A	105.7 (10)

C8—N1—H1A	120.4 (13)	N5—C21—H21B	105.6 (10)
N3—N2—C17	109.57 (11)	N5—C21—C22	112.09 (11)
N2—N3—N4	106.74 (11)	H21A—C21—H21B	111.7 (14)
N3—N4—C18	111.20 (11)	C22—C21—H21A	109.2 (10)
N3—N4—C19	120.82 (11)	C22—C21—H21B	112.4 (10)
C18—N4—C19	127.87 (12)	C23—C22—C21	119.45 (12)
C20—N5—C21	120.23 (11)	C27—C22—C21	120.83 (13)
C20—N5—C30	122.53 (11)	C27—C22—C23	119.72 (13)
C30—N5—C21	117.11 (10)	C22—C23—H23	122.0 (11)
C34—N6—H6	115.9 (13)	C24—C23—C22	120.06 (13)
C36—N6—H6	115.8 (13)	C24—C23—H23	117.9 (11)
C36—N6—C34	128.01 (12)	O6—C24—C23	125.84 (13)
O1—C1—C2	113.95 (12)	O6—C24—C25	114.18 (13)
O2—C1—O1	121.94 (13)	C23—C24—C25	119.97 (13)
O2—C1—C2	124.10 (13)	O7—C25—C24	114.25 (13)
C3—C2—C1	121.20 (13)	O7—C25—C26	125.94 (14)
C3—C2—C7	118.35 (13)	C26—C25—C24	119.81 (14)
C7—C2—C1	120.45 (13)	C25—C26—H26	120.5 (12)
C2—C3—H3	118.7 (11)	C25—C26—C27	119.96 (14)
C4—C3—C2	120.75 (13)	C27—C26—H26	119.5 (12)
C4—C3—H3	120.5 (11)	C22—C27—C26	120.47 (13)
C3—C4—H4	119.9 (11)	C22—C27—H27	120.8 (11)
C3—C4—C5	120.93 (13)	C26—C27—H27	118.7 (11)
C5—C4—H4	119.2 (11)	O6—C28—H28A	109.8 (12)
N1—C5—C4	118.57 (12)	O6—C28—H28B	106.2 (15)
N1—C5—C6	122.84 (13)	O6—C28—H28C	111.1 (14)
C6—C5—C4	118.58 (13)	H28A—C28—H28B	110.3 (19)
C5—C6—H6A	121.9 (10)	H28A—C28—H28C	108.8 (18)
C7—C6—C5	119.59 (13)	H28B—C28—H28C	110.6 (19)
C7—C6—H6A	118.5 (10)	O7—C29—H29A	113.0 (13)
C2—C7—H7	119.2 (11)	O7—C29—H29B	103.1 (12)
C6—C7—C2	121.65 (13)	O7—C29—H29C	108.7 (12)
C6—C7—H7	119.2 (11)	H29A—C29—H29B	111.0 (17)
N1—C8—H8A	109.6 (12)	H29A—C29—H29C	112.3 (17)
N1—C8—H8B	106.9 (11)	H29B—C29—H29C	108.2 (17)
N1—C8—C9	114.37 (12)	C31—C30—N5	118.76 (12)
H8A—C8—H8B	109.2 (16)	C35—C30—N5	119.15 (11)
C9—C8—H8A	108.6 (12)	C35—C30—C31	122.07 (12)
C9—C8—H8B	108.1 (11)	C30—C31—H31	121.3 (11)
C10—C9—C8	121.97 (13)	C30—C31—C32	118.46 (12)
C10—C9—C14	119.47 (12)	C32—C31—H31	120.2 (11)
C14—C9—C8	118.39 (12)	C31—C32—H32	119.4 (10)
C9—C10—H10	120.8 (11)	C33—C32—C31	120.63 (12)
C9—C10—C11	120.82 (13)	C33—C32—H32	119.9 (10)
C11—C10—H10	118.4 (11)	C32—C33—H33	121.9 (10)
C10—C11—H11	120.3 (11)	C32—C33—C34	120.35 (12)
C12—C11—C10	119.28 (13)	C34—C33—H33	117.8 (10)
C12—C11—H11	120.4 (11)	C33—C34—N6	116.94 (11)

O4—C12—C11	125.59 (12)	C33—C34—C35	119.84 (12)
O4—C12—C13	114.17 (12)	C35—C34—N6	123.03 (12)
C11—C12—C13	120.23 (12)	C30—C35—C34	118.63 (12)
O3—C13—C12	114.78 (12)	C30—C35—H35	122.6 (10)
O3—C13—C14	125.58 (13)	C34—C35—H35	118.8 (10)
C14—C13—C12	119.64 (12)	O8—C36—N6	124.00 (13)
C9—C14—H14	119.3 (11)	O8—C36—C37	121.88 (13)
C13—C14—C9	120.53 (13)	N6—C36—C37	114.12 (12)
C13—C14—H14	120.1 (11)	C36—C37—H37A	115.1 (15)
O3—C15—H15A	106.1 (13)	C36—C37—H37B	108.2 (17)
O3—C15—H15B	110.2 (12)	C36—C37—H37C	109.4 (17)
O3—C15—H15C	110.2 (11)	H37A—C37—H37B	100 (2)
H15A—C15—H15B	109.8 (17)	H37A—C37—H37C	121 (2)
H15A—C15—H15C	108.7 (17)	H37B—C37—H37C	101 (2)
H15B—C15—H15C	111.6 (17)	H38A—C38—H38B	109 (2)
O4—C16—H16A	111.0 (11)	H38A—C38—H38C	106 (2)
O4—C16—H16B	111.4 (11)	H38B—C38—H38C	114 (2)
O4—C16—C17	103.73 (10)	C39—C38—H38A	108.4 (16)
H16A—C16—H16B	107.9 (15)	C39—C38—H38B	110.6 (16)
C17—C16—H16A	111.3 (11)	C39—C38—H38C	108.8 (18)
C17—C16—H16B	111.5 (11)	N7—C39—C38	179.1 (2)
N2—C17—C16	120.91 (12)	H40A—C40—H40B	99 (3)
N2—C17—C18	107.84 (12)	H40A—C40—H40C	118 (3)
C18—C17—C16	130.64 (13)	H40B—C40—H40C	114 (3)
N4—C18—C17	104.64 (12)	C41—C40—H40A	102.5 (17)
N4—C18—H18	123.2 (12)	C41—C40—H40B	111 (2)
C17—C18—H18	132.1 (12)	C41—C40—H40C	111 (2)
N4—C19—H19A	106.2 (12)	N8—C41—C40	178.3 (2)
O1—C1—C2—C3	−4.54 (19)	C11—C12—C13—C14	−1.8 (2)
O1—C1—C2—C7	175.50 (13)	C12—O4—C16—C17	−176.54 (11)
O2—C1—C2—C3	174.42 (14)	C12—C13—C14—C9	0.5 (2)
O2—C1—C2—C7	−5.5 (2)	C14—C9—C10—C11	−1.7 (2)
O3—C13—C14—C9	−179.55 (12)	C15—O3—C13—C12	−174.93 (12)
O4—C12—C13—O3	−0.33 (17)	C15—O3—C13—C14	5.1 (2)
O4—C12—C13—C14	179.62 (12)	C16—O4—C12—C11	3.0 (2)
O4—C16—C17—N2	80.36 (15)	C16—O4—C12—C13	−178.42 (12)
O4—C16—C17—C18	−89.54 (17)	C16—C17—C18—N4	170.74 (13)
O6—C24—C25—O7	0.75 (18)	C17—N2—N3—N4	−0.44 (14)
O6—C24—C25—C26	−179.07 (13)	C18—N4—C19—C20	−115.62 (15)
O7—C25—C26—C27	−179.49 (14)	C19—N4—C18—C17	−176.24 (12)
N1—C5—C6—C7	175.90 (13)	C20—N5—C21—C22	101.22 (14)
N1—C8—C9—C10	29.93 (19)	C20—N5—C30—C31	107.10 (15)
N1—C8—C9—C14	−154.72 (13)	C20—N5—C30—C35	−74.49 (17)
N2—N3—N4—C18	0.34 (15)	C21—N5—C20—O5	−0.7 (2)
N2—N3—N4—C19	176.79 (11)	C21—N5—C20—C19	178.47 (12)
N2—C17—C18—N4	−0.16 (15)	C21—N5—C30—C31	−77.15 (16)
N3—N2—C17—C16	−171.58 (11)	C21—N5—C30—C35	101.26 (14)

N3—N2—C17—C18	0.38 (15)	C21—C22—C23—C24	−178.77 (12)
N3—N4—C18—C17	−0.11 (15)	C21—C22—C27—C26	179.01 (13)
N3—N4—C19—C20	68.58 (16)	C22—C23—C24—O6	178.35 (13)
N4—C19—C20—O5	8.79 (19)	C22—C23—C24—C25	−0.5 (2)
N4—C19—C20—N5	−170.36 (11)	C23—C22—C27—C26	−0.6 (2)
N5—C21—C22—C23	−64.78 (15)	C23—C24—C25—O7	179.76 (12)
N5—C21—C22—C27	115.57 (13)	C23—C24—C25—C26	−0.1 (2)
N5—C30—C31—C32	179.70 (12)	C24—C25—C26—C27	0.3 (2)
N5—C30—C35—C34	−179.20 (11)	C25—C26—C27—C22	0.0 (2)
N6—C34—C35—C30	174.52 (12)	C27—C22—C23—C24	0.9 (2)
C1—C2—C3—C4	177.01 (13)	C28—O6—C24—C23	−0.7 (2)
C1—C2—C7—C6	−178.31 (13)	C28—O6—C24—C25	178.27 (13)
C2—C3—C4—C5	0.8 (2)	C29—O7—C25—C24	−170.61 (13)
C3—C2—C7—C6	1.7 (2)	C29—O7—C25—C26	9.2 (2)
C3—C4—C5—N1	−177.16 (13)	C30—N5—C20—O5	174.96 (12)
C3—C4—C5—C6	2.7 (2)	C30—N5—C20—C19	−5.91 (18)
C4—C5—C6—C7	−4.0 (2)	C30—N5—C21—C22	−74.63 (14)
C5—N1—C8—C9	80.99 (17)	C30—C31—C32—C33	−0.7 (2)
C5—C6—C7—C2	1.8 (2)	C31—C30—C35—C34	−0.8 (2)
C7—C2—C3—C4	−3.0 (2)	C31—C32—C33—C34	−0.4 (2)
C8—N1—C5—C4	179.92 (13)	C32—C33—C34—N6	−174.22 (13)
C8—N1—C5—C6	0.0 (2)	C32—C33—C34—C35	0.9 (2)
C8—C9—C10—C11	173.58 (12)	C33—C34—C35—C30	−0.28 (19)
C8—C9—C14—C13	−174.25 (12)	C34—N6—C36—O8	10.5 (2)
C9—C10—C11—C12	0.5 (2)	C34—N6—C36—C37	−169.90 (14)
C10—C9—C14—C13	1.2 (2)	C35—C30—C31—C32	1.3 (2)
C10—C11—C12—O4	179.72 (13)	C36—N6—C34—C33	−167.21 (14)
C10—C11—C12—C13	1.3 (2)	C36—N6—C34—C35	17.8 (2)
C11—C12—C13—O3	178.29 (12)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O9—H9 <i>A</i> $\cdots$ O2 ⁱ	0.96 (3)	1.80 (3)	2.7173 (17)	160 (2)
O9—H9 <i>B</i> $\cdots$ O8	0.99 (3)	1.90 (3)	2.8610 (18)	164 (2)
O10—H10 <i>A</i> $\cdots$ O6 ⁱⁱ	0.96 (3)	2.12 (3)	2.9441 (17)	144 (2)
O10—H10 <i>A</i> $\cdots$ O7 ⁱⁱ	0.96 (3)	2.17 (3)	2.9928 (17)	143 (2)
O10—H10 <i>B</i> $\cdots$ O9 ⁱⁱⁱ	0.95 (3)	2.00 (3)	2.9346 (19)	167 (2)
N1—H1 <i>A</i> $\cdots$ N3 ^{iv}	0.90 (2)	2.29 (2)	3.1834 (17)	174.6 (18)
N6—H6 $\cdots$ O5 ^v	0.85 (2)	1.98 (2)	2.8200 (15)	168.6 (19)
O1—H1 $\cdots$ N2 ^{vi}	0.91 (3)	1.87 (3)	2.7614 (16)	166 (3)

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