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Crystal structure of 6-hydroxy-5-(2-methoxyphenoxy)-2,2′-bipyrimidin-4(3*H*)-one

Belakavadi K. Sagar,[a] Hemmige S. Yathirajan,[a]* Jerry P. Jasinski[b] and Christopher Glidewell[c]

[a]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysuru 570 006, India, [b]Department of Chemistry, Keene State College, 229 Main Street, Keene, NH 03435-2001, USA, and [c]School of Chemistry, University of St Andrews, St Andrews, Fife KY16 9ST, UK. *Correspondence e-mail: yathirajan@hotmail.com

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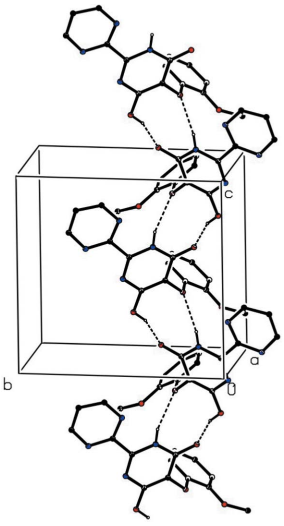
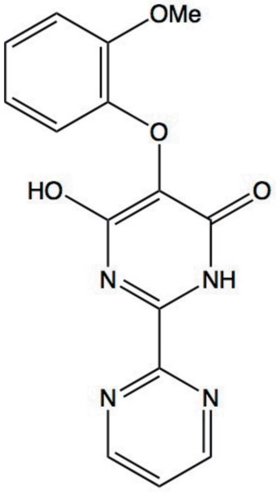
In the title compound, $C_{15}H_{12}N_4O_4$, the dihedral angle between the heterocyclic rings is 12.60 (8)°, and that between the benzene ring and the adjacent heterocyclic ring is 85.14 (6)°. In the crystal, a combination of N—H···O and O—H···O hydrogen bonds link molecules related by a glide plane into a $C(5)$ C(6)[$R_2^2(9)$] chain of rings, which is a distinctly different packing motif to those observed in hydrated modifications of this compound.



1. Chemical context

Pyrimidine derivatives exhibit a wide variety of biological actions (Önal & Yıldırım, 2007) and specific examples are of particular value in the treatment of cardiovascular diseases (Goldmann & Stoltefuss, 1991). One such derivative is bosentan, 4-*tert*-butyl-*N*-[6-(2-hydroxyethoxy)-5-(2-methoxyphenoxy)-2-(pyrimidin-2-yl)pyrimidin-4-yl]benzene-1-sulfonamide, which is used in the treatment of pulmonary artery hypertension (Pearl *et al.*, 1999; Hoeper *et al.*, 2003; Kenyon & Nappi, 2003).



4-Hydroxy-5-(2-methoxyphenoxy)-2,2′-bipyrimidin-6(1*H*)-one (I) (Fig. 1) is an intermediate in the synthesis of bosentan (Rebelli *et al.*, 2013; Kompella *et al.*, 2014) and accordingly it is of interest to determine its crystal and molecular structure, which we report here. Crystals of the anhydrous title compound (I) were obtained from a solution of a 1:1 mixture of dimethylsulfoxide and *N*,*N*-dimethylformamide in the presence of adipic acid: by contrast, a similar crystallization regime but omitting the adipic acid yielded the corresponding

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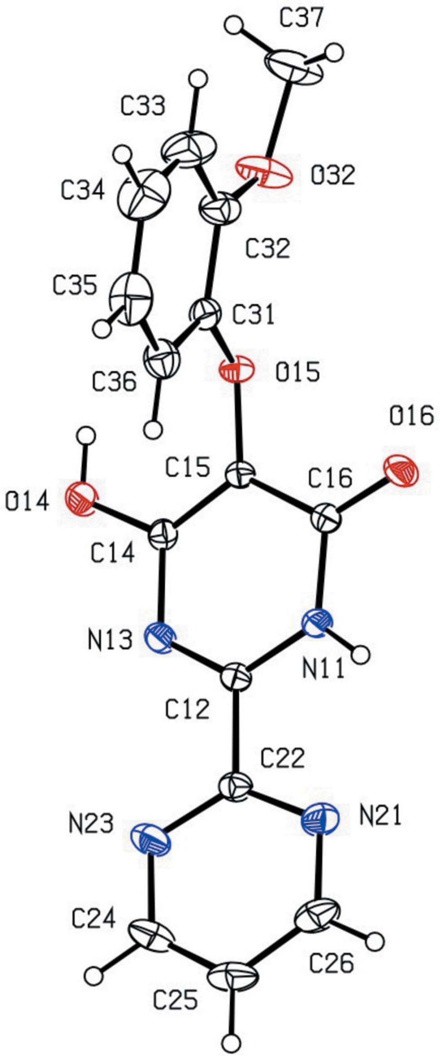

Figure 1
The molecular structure of compound (I) showing displacement ellipsoids drawn at the 30% probability level.

dihydrate (II) (Yamuna *et al.*, 2013), so permitting comparison of the anhydrous and hydrated forms.

2. Structural commentary

The bond distances in the ring containing atom N11 clearly show the presence of localized double bonds in the bonds C12═N13 and C14═C15 as well as the exocyclic C16═O16, fully consistent with the location of the H atoms on atoms N11 and O14, as deduced from difference maps and confirmed by the refinement. By contrast, the bond distances in the other heterocyclic ring indicate conventional aromatic-type delocalization.

At each of the sites C14, C31 and C32, the corresponding pairs of exocyclic O—C—N (at C14) or O—C—C angles (at C31 and C32) differ by almost 10°, as generally observed in the arenes of type Ar*OR* when the substituent *R* lies close to the plane of the aryl ring (Seip & Seip, 1973; Ferguson *et al.*, 1996). Here atoms C15 and C37 (Fig. 1) are displaced from the plane of the aryl ring (C31–C36) by 0.219 (3) and 0.204 (4) Å, respectively, with both substituents displaced to the same side of the aryl ring. The C—O—C angles at atoms O15 and O32, 115.41 (12) and 117.65 (18)° respectively, and the C—O—H angle at atom O14 is 114.2 (16)°; are all significantly larger the the idealized tetrahedral value of 109.5°.

The dihedral angle between the heterocyclic rings is 12.60 (8)° and that between the ring containing N11 and the aryl ring is 85.14 (6)°. Accordingly, the molecule of (I) exhibits

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O15^{i}	0.855 (19)	2.257 (19)	2.9733 (18)	141.4 (17)
O14—H14···O16^{ii}	0.85 (2)	1.80 (2)	2.6117 (18)	160 (2)

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

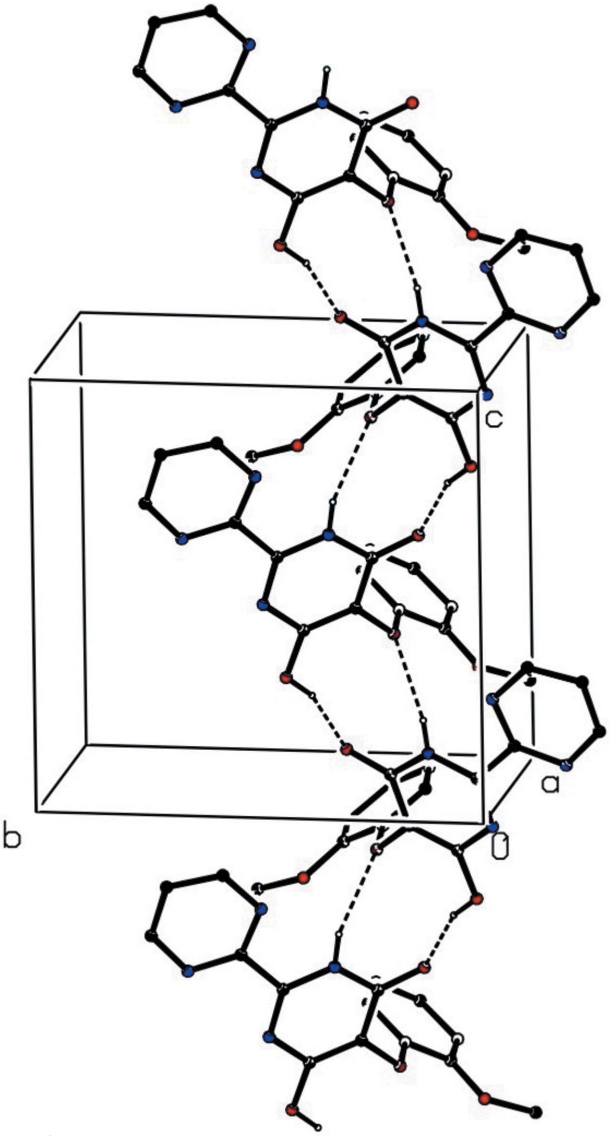

Figure 2
Part of the crystal structure of compound (I) showing the formation of a hydrogen-bonded $C(5)$ $C(6)[R_2^2)9)]$ chain of rings parallel to [001]. For the sake of clarity, the H atoms bonded to C atoms have all been omitted.

no internal symmetry and thus the compound is conformationally chiral: the centrosymmetric space group confirms that (I) crystallizes as a conformational racemate.

3. Supramolecular interactions

In the crystal, molecules of (I) are linked by a combination of O—H···N and N—H···N hydrogen bonds (Table 1) to form a $C(5)\ C(6)[R_2^2(9)]$ chain of rings running parallel to the [001] direction (Fig. 2): adjacent molecules are related by glide-plane symmetry. Two chains of this type, related to one another by inversion, pass through each unit cell, but there are no direction-specific interactions between adjacent chains.

4. Database survey

In the dihydrate (II), an extensive series of hydrogen bonds, encompassing N—H···O, O—H···N and O—H···O types links the molecular components into a complex sheet structure (Yamuna *et al.*, 2013), in contrast to the rather simple chains in (I) reported here. A sheet structure, built from a combination of the same three types of hydrogen bond is found also in the structure of bosentan monohydrate (Kaur *et al.*, 2013).

5. Synthesis and crystallization

A sample of compound (I) was a gift from Cadila Pharmaceuticals Ltd, Ahmedabad, Gujarat, India. Colourless plates of the anhydrous compound (I) were grown by slow evaporation, at room temperature of a solution of (I) in a mixture of dimethylsulfoxide and *N*,*N*-dimethylformamide (1:1, *v*/*v*) containing an excess of adipic acid (hexane-1,6-dioic acid).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were located in difference maps. The H atoms bonded to C atoms were then treated as riding atoms in geometrically idealized positions with C—H distances 0.93 Å (aromatic and heteroaromatic) or 0.96 Å (CH_3) and with $U_{iso}(H) = kU_{eq}(C)$ where $k = 1.5$ for the methyl group, which was permitted to rotate but not to tilt and 1.2 for all other H atoms bonded to C atoms. For the H atoms bonded to O or N atoms, the atomic coordinates were refined with $U_{iso}(H) = 1.5U_{eq}(O)$ or $1.2U_{eq}(N)$, giving the O—H and N—H distances shown in Table 1.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{12}N_4O_4$
M_r	312.29
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	298
a, b, c (Å)	12.1863 (9), 10.7079 (8), 11.1726 (8)
β (°)	105.412 (8)
V (Å^3)	1405.48 (19)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.49 × 0.46 × 0.28
Data collection	
Diffractometer	Agilent Xcalibur, Eos, Gemini CCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2003)
T_{min}, T_{max}	0.812, 0.969
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7121, 3113, 2311
R_{int}	0.037
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.134, 1.07
No. of reflections	3113
No. of parameters	215
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.25

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

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References

Agilent (2014). *CrysAlis PRO*. Agilent Technologies Ltd, Yarnton, England.

Ferguson, G., Glidewell, C. & Patterson, I. L. J. (1996). *Acta Cryst.* C**52**, 420–423.

Goldmann, S. & Stoltefuss, J. (1991). *Angew. Chem. Int. Ed. Engl.* **30**, 1559–1578.

Hoeper, M., Taha, N., Bekjarova, A., Gatzke, R. & Spiekerkoetter, E. (2003). *Eur. Respir. J.* **22**, 330–334.

Kaur, M., Jasinski, J. P., Keeley, A. C., Yathirajan, H. S., Betz, R., Gerber, T. & Butcher, R. J. (2013). *Acta Cryst.* E**69**, o12–o13.

Kenyon, K. W. & Nappi, J. M. (2003). *Ann. Pharmacother.* **37**, 1055–1062.

Kompella, A., Kasa, S., Balina, V. S., Kusumba, S., Adibhatla, B. R. K. & Muddasani, P. R. (2014). *Sci. J. Chem.* **2**, 9–15.

Önal, Z. & Yıldırım, İ. (2007). *Heterocycl. Commun.* **13**, 113–120.

Pearl, J. M., Wellmann, S. A., McNamara, J. L., Lombardi, J. P., Wagner, C. J., Raake, J. L. & Nelson, D. P. (1999). *Ann. Thorac. Surg.* **68**, 1714–21 discussion 1721–1714-21; discussion 1722.

Rebelli, P., Yerrabelly, J. R., Yalamanchili, B. K., Kommera, R., Ghojala, V. R. & Bairy, K. R. (2013). *Org. Process Res. Dev.* **17**, 1021–1026.

Seip, H. M. & Seip, R. (1973). *Acta Chem. Scand.* **27**, 4024–4027.

Sheldrick, G. M. (2003). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Yamuna, T. S., Jasinski, J. P., Anderson, B. J., Yathirajan, H. S. & Kaur, M. (2013). *Acta Cryst.* E**69**, o1707–o1708.

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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: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009).

6-Hydroxy-5-(2-methoxyphenoxy)-2,2′-bipyrimidin-4(3*H*)-one

Crystal data

$C_{15}H_{12}N_4O_4$
$M_r = 312.29$
Monoclinic, $P2_1/c$
$a = 12.1863$ (9) Å
$b = 10.7079$ (8) Å
$c = 11.1726$ (8) Å
$\beta = 105.412$ (8)°
$V = 1405.48$ (19) Å^3
$Z = 4$

$F(000) = 648$
$D_x = 1.476$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3266 reflections
$\theta = 3.5$–29.2°
$\mu = 0.11$ mm^{-1}
$T = 298$ K
Plate, colourles
0.49 × 0.46 × 0.28 mm

Data collection

Agilent Xcalibur, Eos, Gemini CCD diffractometer
Detector resolution: 16.0416 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{\min} = 0.812$, $T_{\max} = 0.969$
7121 measured reflections

3113 independent reflections
2311 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.037$
$\theta_{\max} = 27.5°$, $\theta_{\min} = 3.5°$
$h = -15 \rightarrow 15$
$k = -13 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.134$
$S = 1.07$
3113 reflections
215 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0582P)^2 + 0.1493P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.26$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
N11	0.18153 (12)	0.35790 (14)	0.63063 (13)	0.0295 (3)
H11	0.1817 (15)	0.3477 (17)	0.7066 (18)	0.035*
C12	0.14803 (12)	0.47018 (16)	0.58084 (14)	0.0257 (4)
N13	0.15092 (11)	0.50269 (13)	0.46986 (12)	0.0288 (3)
C14	0.19721 (13)	0.41910 (16)	0.40542 (14)	0.0259 (4)
C15	0.23862 (13)	0.30650 (15)	0.45349 (14)	0.0246 (4)
C16	0.22954 (14)	0.26765 (16)	0.57208 (15)	0.0286 (4)
O14	0.20095 (11)	0.45956 (12)	0.29445 (11)	0.0384 (3)
H14	0.2235 (18)	0.405 (2)	0.251 (2)	0.058*
O15	0.29101 (9)	0.22750 (11)	0.38791 (10)	0.0286 (3)
O16	0.26156 (12)	0.16663 (12)	0.62240 (11)	0.0435 (4)
N21	0.10457 (13)	0.51230 (15)	0.77235 (13)	0.0382 (4)
C22	0.11108 (13)	0.55897 (16)	0.66440 (15)	0.0287 (4)
N23	0.09267 (14)	0.67630 (15)	0.62621 (15)	0.0442 (4)
C24	0.06898 (19)	0.7543 (2)	0.7096 (2)	0.0534 (6)
H24	0.0564	0.8380	0.6881	0.064*
C25	0.06238 (17)	0.7179 (2)	0.8239 (2)	0.0503 (6)
H25	0.0467	0.7743	0.8805	0.060*
C26	0.07998 (16)	0.5938 (2)	0.85148 (18)	0.0472 (5)
H26	0.0746	0.5653	0.9283	0.057*
C31	0.41008 (14)	0.22844 (17)	0.42471 (15)	0.0305 (4)
C32	0.46353 (16)	0.1335 (2)	0.37687 (17)	0.0417 (5)
C33	0.58106 (19)	0.1307 (3)	0.4082 (2)	0.0639 (7)
H33	0.6184	0.0678	0.3771	0.077*
C34	0.64289 (19)	0.2205 (3)	0.4850 (3)	0.0713 (8)
H34	0.7220	0.2185	0.5043	0.086*
C35	0.59019 (18)	0.3127 (3)	0.5336 (2)	0.0595 (6)
H35	0.6330	0.3720	0.5868	0.071*
C36	0.47253 (15)	0.3169 (2)	0.50285 (17)	0.0414 (5)
H36	0.4358	0.3795	0.5351	0.050*
O32	0.39382 (13)	0.05064 (15)	0.30120 (14)	0.0585 (5)
C37	0.4443 (3)	−0.0572 (3)	0.2648 (2)	0.0755 (8)
H37A	0.4889	−0.0336	0.2095	0.113*
H37B	0.3857	−0.1143	0.2233	0.113*
H37C	0.4923	−0.0968	0.3369	0.113*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.0418 (8)	0.0269 (8)	0.0233 (7)	0.0053 (6)	0.0148 (6)	0.0020 (6)
C12	0.0255 (8)	0.0249 (9)	0.0261 (8)	0.0005 (7)	0.0059 (6)	−0.0018 (7)
N13	0.0345 (7)	0.0239 (8)	0.0278 (7)	0.0035 (6)	0.0078 (6)	0.0006 (6)
C14	0.0298 (8)	0.0261 (9)	0.0216 (7)	−0.0028 (7)	0.0063 (6)	−0.0010 (7)
C15	0.0283 (8)	0.0233 (9)	0.0235 (7)	0.0008 (7)	0.0090 (6)	−0.0023 (7)
C16	0.0342 (9)	0.0251 (10)	0.0283 (8)	0.0022 (7)	0.0112 (6)	0.0005 (7)
O14	0.0632 (8)	0.0300 (8)	0.0255 (6)	0.0066 (6)	0.0178 (6)	0.0038 (5)
O15	0.0310 (6)	0.0293 (7)	0.0267 (6)	0.0032 (5)	0.0098 (4)	−0.0058 (5)
O16	0.0722 (9)	0.0286 (8)	0.0360 (7)	0.0176 (7)	0.0256 (6)	0.0095 (6)
N21	0.0455 (9)	0.0392 (10)	0.0327 (8)	−0.0007 (7)	0.0155 (6)	−0.0068 (7)
C22	0.0273 (8)	0.0283 (10)	0.0304 (8)	−0.0007 (7)	0.0074 (6)	−0.0054 (7)
N23	0.0592 (10)	0.0287 (9)	0.0473 (9)	0.0076 (8)	0.0189 (8)	−0.0052 (7)
C24	0.0661 (14)	0.0319 (12)	0.0638 (14)	0.0109 (10)	0.0198 (11)	−0.0135 (10)
C25	0.0466 (11)	0.0519 (14)	0.0544 (13)	0.0037 (10)	0.0169 (9)	−0.0267 (11)
C26	0.0511 (11)	0.0596 (15)	0.0354 (10)	−0.0023 (11)	0.0194 (9)	−0.0162 (10)
C31	0.0315 (8)	0.0330 (10)	0.0278 (8)	0.0055 (7)	0.0091 (6)	0.0059 (7)
C32	0.0456 (11)	0.0453 (13)	0.0333 (9)	0.0162 (9)	0.0092 (8)	0.0034 (9)
C33	0.0484 (13)	0.085 (2)	0.0592 (14)	0.0317 (13)	0.0169 (11)	0.0048 (14)
C34	0.0340 (12)	0.101 (2)	0.0756 (17)	0.0110 (13)	0.0089 (11)	0.0157 (17)
C35	0.0424 (12)	0.0687 (17)	0.0600 (14)	−0.0114 (11)	0.0008 (10)	0.0076 (13)
C36	0.0394 (10)	0.0399 (12)	0.0438 (11)	−0.0031 (9)	0.0089 (8)	0.0016 (9)
O32	0.0649 (9)	0.0506 (10)	0.0543 (9)	0.0259 (8)	0.0057 (7)	−0.0188 (8)
C37	0.113 (2)	0.0547 (16)	0.0594 (15)	0.0433 (15)	0.0238 (14)	−0.0059 (13)

Geometric parameters (Å, º)

N11—C12	1.342 (2)	C26—N21	1.332 (2)
N11—H11	0.86 (2)	C16—O16	1.234 (2)
C12—N13	1.297 (2)	C26—H26	0.9300
C12—C22	1.484 (2)	C31—C36	1.373 (3)
N13—C14	1.361 (2)	C31—C32	1.389 (3)
C14—C15	1.361 (2)	C32—O32	1.357 (2)
C15—C16	1.421 (2)	C32—C33	1.381 (3)
C16—N11	1.381 (2)	C33—C34	1.373 (4)
C14—O14	1.3255 (19)	C33—H33	0.9300
C15—O15	1.3816 (18)	C34—C35	1.367 (4)
O14—H14	0.85 (2)	C34—H34	0.9300
O15—C31	1.3990 (19)	C35—C36	1.384 (3)
N21—C22	1.327 (2)	C35—H35	0.9300
C22—N23	1.327 (2)	C36—H36	0.9300
N23—C24	1.339 (2)	O32—C37	1.418 (3)
C24—C25	1.358 (3)	C37—H37A	0.9600
C24—H24	0.9300	C37—H37B	0.9600
C25—C26	1.368 (3)	C37—H37C	0.9600
C25—H25	0.9300		

C12—N11—C16	123.44 (14)	N21—C26—H26	118.8
C12—N11—H11	116.5 (13)	C25—C26—H26	118.8
C16—N11—H11	119.4 (13)	C36—C31—C32	120.81 (17)
N13—C12—N11	123.65 (15)	O15—C31—C32	115.95 (15)
N13—C12—C22	121.28 (15)	O15—C31—C36	123.24 (16)
N11—C12—C22	115.02 (14)	O32—C32—C31	116.00 (16)
C12—N13—C14	116.70 (14)	O32—C32—C33	125.34 (19)
O14—C14—N13	113.69 (14)	C15—O15—C31	115.41 (12)
O14—C14—C15	123.87 (15)	C32—O32—C37	117.65 (18)
C15—C14—N13	122.41 (14)	C33—C32—C31	118.7 (2)
C14—C15—O15	120.54 (13)	C34—C33—C32	120.2 (2)
C14—C15—C16	120.98 (15)	C34—C33—H33	119.9
O15—C15—C16	118.47 (14)	C32—C33—H33	119.9
O16—C16—N11	121.35 (15)	C35—C34—C33	121.1 (2)
O16—C16—C15	126.03 (16)	C35—C34—H34	119.4
N11—C16—C15	112.61 (14)	C33—C34—H34	119.4
C14—O14—H14	114.2 (16)	C34—C35—C36	119.4 (2)
C22—N21—C26	115.78 (17)	C34—C35—H35	120.3
N23—C22—N21	127.09 (16)	C36—C35—H35	120.3
N23—C22—C12	117.26 (15)	C31—C36—C35	119.9 (2)
N21—C22—C12	115.59 (15)	C31—C36—H36	120.1
C22—N23—C24	114.59 (17)	C35—C36—H36	120.1
N23—C24—C25	123.6 (2)	O32—C37—H37A	109.5
N23—C24—H24	118.2	O32—C37—H37B	109.5
C25—C24—H24	118.2	H37A—C37—H37B	109.5
C24—C25—C26	116.47 (19)	O32—C37—H37C	109.5
C24—C25—H25	121.8	H37A—C37—H37C	109.5
C26—C25—H25	121.8	H37B—C37—H37C	109.5
N21—C26—C25	122.44 (19)		

C16—N11—C12—N13	4.3 (2)	N11—C12—C22—N21	−6.7 (2)
C16—N11—C12—C22	−173.20 (14)	N21—C22—N23—C24	2.4 (3)
N11—C12—N13—C14	−4.0 (2)	C12—C22—N23—C24	−174.60 (16)
C22—C12—N13—C14	173.33 (13)	C22—N23—C24—C25	−0.9 (3)
C12—N13—C14—O14	−177.94 (14)	N23—C24—C25—C26	−0.8 (3)
C12—N13—C14—C15	0.1 (2)	C22—N21—C26—C25	0.0 (3)
O14—C14—C15—O15	1.7 (2)	C24—C25—C26—N21	1.3 (3)
N13—C14—C15—O15	−176.13 (13)	C15—O15—C31—C36	−12.1 (2)
O14—C14—C15—C16	−178.56 (15)	C15—O15—C31—C32	168.10 (14)
N13—C14—C15—C16	3.6 (2)	C36—C31—C32—O32	179.61 (17)
C12—N11—C16—O16	178.32 (16)	O15—C31—C32—O32	−0.6 (2)
C12—N11—C16—C15	−0.4 (2)	C36—C31—C32—C33	−0.8 (3)
C14—C15—C16—O16	178.06 (17)	O15—C31—C32—C33	178.93 (17)
O15—C15—C16—O16	−2.2 (3)	O32—C32—C33—C34	179.5 (2)
C14—C15—C16—N11	−3.2 (2)	C31—C32—C33—C34	−0.1 (3)
O15—C15—C16—N11	176.48 (13)	C32—C33—C34—C35	1.1 (4)
C14—C15—O15—C31	100.24 (17)	C33—C34—C35—C36	−1.2 (4)

C16—C15—O15—C31	−79.49 (18)	C32—C31—C36—C35	0.7 (3)
C26—N21—C22—N23	−2.0 (3)	O15—C31—C36—C35	−179.05 (17)
C26—N21—C22—C12	175.03 (15)	C34—C35—C36—C31	0.3 (3)
N13—C12—C22—N23	−6.9 (2)	C33—C32—O32—C37	9.3 (3)
N11—C12—C22—N23	170.67 (15)	C31—C32—O32—C37	−171.21 (19)
N13—C12—C22—N21	175.73 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O15^{i}	0.855 (19)	2.257 (19)	2.9733 (18)	141.4 (17)
O14—H14···O16^{ii}	0.85 (2)	1.80 (2)	2.6117 (18)	160 (2)

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