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3-(4-Hydroxyphenyl)-1,5-bis(pyridin-2-yl)pentane-1,5-dione

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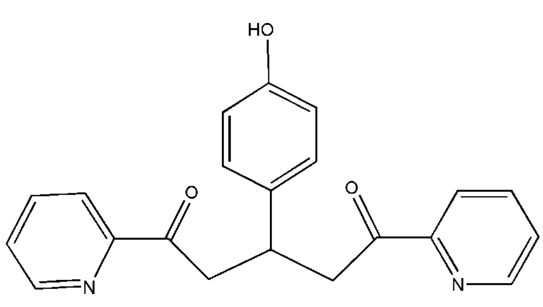
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.119; data-to-parameter ratio = 11.5.

In the title molecule, $C_{21}H_{18}N_2O_3$, the pyridine rings make a dihedral angle of 13.1 (1)°. The phenyl ring is approximately perpendicular to both of them, forming dihedral angles of 87.4 (1)and 81.9 (1)°. In the crystal, pairs of O—H$\cdots$N hydrogen bonds link the molecules into centrosymmetric dimers. Additional C—H$\cdots$O, π–π [centroid–centroid distance = 3.971 (2) Å] and C—H$\cdots\pi$ interactions consolidate the dimers into a three-dimensional network.

Related literature

For the synthesis of the title compound, see: Constable *et al.* (1990, 1998); He *et al.* (2006). For the syntheses of terpyridine compounds and their properties and applications, see: Ma *et al.* (2009, 2010, 2012, 2013). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{21}H_{18}N_2O_3$
M_r = 346.37
Triclinic, $P\overline{1}$
a = 8.4392 (6) Å
b = 10.6683 (7) Å
c = 11.0755 (8) Å
α = 100.623 (6)°
β = 103.867 (6)°
γ = 110.550 (6)°
V = 866.05 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 K
0.39 × 0.38 × 0.22 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*, Agilent, 2012)
T_{min} = 0.813, T_{max} = 1.000
6339 measured reflections
3544 independent reflections
2661 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.119
S = 1.03
3544 reflections
308 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

*Cg*3 is the centroid of the C16–C21 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H03$A\cdots$N2^{i}	0.93 (2)	2.00 (2)	2.8940 (19)	160 (2)
C12—H12$A\cdots$O2^{ii}	0.96 (2)	2.48 (2)	3.312 (3)	145 (2)
C4a—H4a$\cdots Cg3^{iii}$	0.99 (2)	0.98 (2)	3.825 (2)	144 (2)

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

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LD2113).

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supporting information

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3-(4-Hydroxyphenyl)-1,5-bis(pyridin-2-yl)pentane-1,5-dione

Lixia Pan, Huaduan Shi and Zhen Ma

S1. Comment

Metal terpyridene complexes is a topic of major current interest because they show very interesting properties, such as photoluminescence, catalytic and antibiological activities (Ma, Xing *et al.* 2009; Ma, Cao *et al.* 2010; Ma, Liang *et al.* 2012; Ma, Lu *et al.* 2013). Hence, the aim of our current work was to prepare a series of precursors to produce terpyridine ligands, investigate their coordination behavior toward metal ions and study their applications (Constable, Lewis *et al.* 1990; Constable, Neuburger *et al.* 1998). Here, we report the structure of a precursor compound for terpyridine synthesis, which was obtained by reaction of 4-hydroxybenzaldehyde with 2-acetylpyridine in a mixed water/ethanol solution of NaOH, and its structure was determined by X-ray crystal analysis.

The molecular structure is shown in Fig. 1. The average bond length C=O for two carbonyls is 1.1212 Å. Other averages are 1.336 Å for N-C bonds, 1.371 Å for C-C bonds of the pyridyl groups and 1.384 Å for C-C bonds of the aryl group. All bond lengths are within normal ranges (Allen *et al.*, 1987). The two pyridyl groups are not parallel, with a dihedral angle of 13.14 (10) °. The plane of aromatic ring with the hydroxyl group (with an r.m.s. deviation of 0.0041 Å) is approximately perpendicular to those of the two pyridyl groups, forming two dihedral angles of 87.36 (5) and 81.90 (6)°, respectively.

Each molecule forms hydrogen bonds (Table 1) involving its hydroxy group and a nitrogen pyridyl atom ($N2^{ii}$) of a neighboring molecule [symmetry code: (ii) 1-*x*, 1-*y*, -*z*]) and also an H-bond between C(12)-H group and a carbonyl oxygen ($O2^{iii}$) of a neighboring molecule [symmetry code: (iii) 2-*x*, 2-*y*, 1-*z*] (Table 1). These hydrogen bonds account for the formation of centrosymmetric dimers (see Fig 2). The structure also has one intermolecular $\pi\cdots\pi$ interaction with a distance of 3.971 Å between two neighboring pyridyl groups (see Fig 2)[Cg1 and Cg2(iv) or Cg2 and Cg1(v); Cg2 and Cg1 are the two centroids of the two six membered pyridyl rings of C1-C5-N1 or C11-C15-N2, symmetry code: (iv) -1+*x*, -1+*y*, *z*; (v) 1+*x*, 1+*y*, *z*]. Further structural stabilization is provided by an intermolecular C—H$\cdots\pi$ interaction between C(4)-H and its neighboring aryl group Cg3 (Fig 2) [H..Cg 2.98 Å; Cg(3) is the centroid of the six membered aromatic ring $C16^{i}$-$C21^{i}$, symmetry code: (i) *x*, -1+*y*-1, *z*]. These $\pi\cdots\pi$ and C—H$\cdots\pi$ interactions help to consolidate the H-bonded dimers into a three-dimensional network (Fig 3).

S2. Experimental

The title compound was obtained by reaction of 4-hydroxybenzaldehyde with 2-acetylpyridine in a 1.5 *M* NaOH mixed aqueous/ethanol solution according to a reported procedure (Constable, *et al.* 1990). In a 250 cm^3 flask fitted with a funnel, 4-hydroxybenzaldehyde (5.5 g, 45 m*M*) and 40 mL of the 1.5 M NaOH aqueous solution were mixed in 60 cm^3 of ethanol. To this solution was added dropwise a stoichiometric quantity of 2-acetylpyridine (10 mL, 89 m*M*) for a period of half an hour with stirring. The mixture was then stirred for 24 h at room temperature. A white solid formed was obtained by filtration and being washed with two times with distilled water (yield 70 %). The product (25 mg) and distilled water (20 mL) were sealed in a 25-mL stainless steel reactor with Teflon liner and heated at 393 K for 1 d.

Colourless crystals were obtained, which were suitable for X-ray characterization.

S3. Refinement

All H atoms were positioned from a Difference Fourier series and refined.

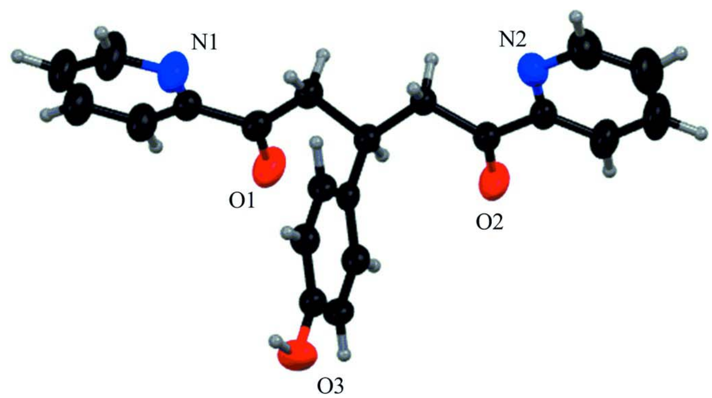

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

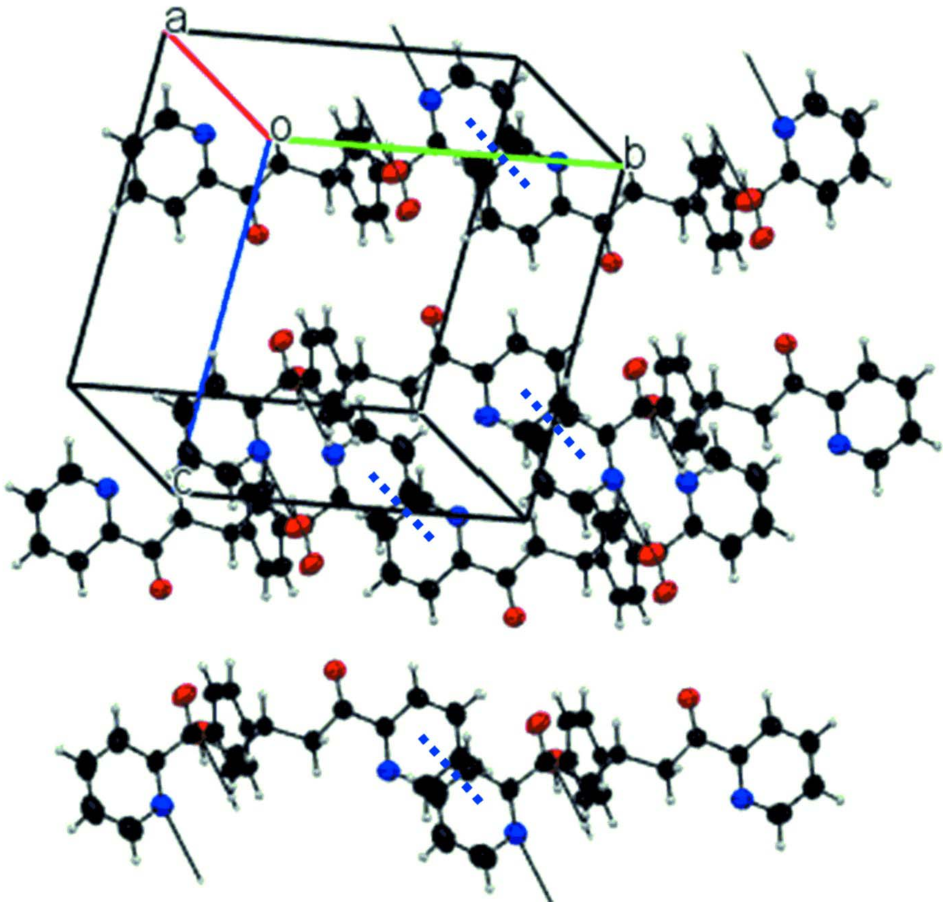

Figure 2

A view of the crystal packing to show the formation of centrosymmetric H-bonded dimers by the help of the hydrogen bonds and the $\pi\cdots\pi$ interactions between the pyridyl groups of the compound. The thin dashed lines are used to show the hydrogen bonds. The blue dotted lines are used to show $\pi\cdots\pi$ interactions between the pyridyl groups of the compound

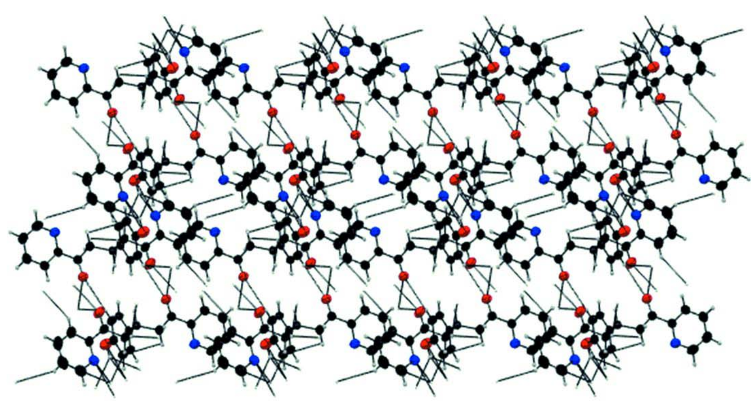

Figure 3

A view of the crystal packing along the *a* axis to show the three-dimensional network.

3-(4-Hydroxyphenyl)-1,5-bis(pyridin-2-yl)pentane-1,5-dione

Crystal data

$C_{21}H_{18}N_2O_3$
M_r = 346.37
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 8.4392 (6) Å
b = 10.6683 (7) Å
c = 11.0755 (8) Å
α = 100.623 (6)°
β = 103.867 (6)°
γ = 110.550 (6)°
V = 866.05 (10) Å^3
Z = 2
F(000) = 364
D_x = 1.328 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6339 reflections
θ = 3.2–26.4°
μ = 0.09 mm^{-1}
T = 298 K
Prism, colourless
0.39 × 0.38 × 0.22 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 0 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*, Agilent, 2012)
T_{min} = 0.813, T_{max} = 1.000
6339 measured reflections
3544 independent reflections
2661 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 26.4°, θ_{min} = 3.2°
h = −10→9
k = −13→13
l = −13→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.119
S = 1.03
3544 reflections
308 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 0.1436P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.018 (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.

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 > 2sigma(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	$U_{iso}*/U_{eq}$
O1	0.71359 (19)	0.30614 (12)	0.46604 (11)	0.0618 (4)

O2	0.91770 (19)	0.78344 (12)	0.42074 (12)	0.0667 (4)
O3	0.08708 (17)	0.40522 (14)	0.08964 (13)	0.0592 (3)
H03A	0.044 (3)	0.351 (3)	0.003 (2)	0.100 (8)*
N1	0.6613 (2)	0.06026 (14)	0.18572 (13)	0.0603 (4)
N2	1.06938 (19)	0.81798 (14)	0.15318 (13)	0.0493 (4)
C1	0.6711 (2)	0.10690 (15)	0.30835 (14)	0.0409 (4)
C2	0.6203 (3)	0.01932 (17)	0.38203 (17)	0.0545 (5)
H2A	0.632 (2)	0.0607 (19)	0.4701 (18)	0.060 (5)*
C3	0.5570 (3)	−0.12308 (18)	0.32835 (19)	0.0639 (5)
H3A	0.524 (3)	−0.184 (2)	0.3782 (19)	0.078 (6)*
C4	0.5449 (3)	−0.17259 (19)	0.20295 (19)	0.0680 (6)
H4A	0.501 (3)	−0.273 (2)	0.159 (2)	0.083 (6)*
C5	0.5963 (4)	−0.07886 (19)	0.1355 (2)	0.0791 (7)
H5A	0.586 (3)	−0.110 (2)	0.044 (2)	0.092 (7)*
C6	0.7379 (2)	0.26254 (15)	0.36597 (14)	0.0414 (4)
C7	0.8359 (2)	0.35918 (16)	0.29975 (17)	0.0432 (4)
H7A	0.966 (3)	0.3793 (18)	0.3406 (17)	0.060 (5)*
H7B	0.798 (2)	0.3108 (17)	0.2046 (16)	0.049 (4)*
C8	0.8128 (2)	0.49738 (14)	0.32322 (14)	0.0378 (3)
H8A	0.847 (2)	0.5387 (15)	0.4188 (15)	0.040 (4)*
C9	0.9371 (2)	0.60196 (15)	0.27268 (16)	0.0409 (4)
H9B	1.059 (2)	0.6076 (17)	0.2993 (15)	0.052 (5)*
H9A	0.897 (2)	0.5730 (17)	0.1737 (17)	0.052 (5)*
C10	0.9588 (2)	0.74944 (15)	0.32631 (15)	0.0426 (4)
C11	1.0436 (2)	0.85928 (15)	0.26587 (15)	0.0428 (4)
C12	1.0940 (3)	0.99903 (18)	0.3296 (2)	0.0623 (5)
H12A	1.072 (3)	1.023 (2)	0.410 (2)	0.075 (6)*
C13	1.1764 (3)	1.0996 (2)	0.2766 (2)	0.0798 (7)
H13A	1.215 (3)	1.200 (3)	0.322 (2)	0.108 (8)*
C14	1.2058 (3)	1.0592 (2)	0.1633 (2)	0.0808 (7)
H14A	1.263 (3)	1.123 (3)	0.122 (2)	0.100 (8)*
C15	1.1504 (3)	0.9187 (2)	0.1043 (2)	0.0665 (5)
H15A	1.167 (3)	0.886 (2)	0.0226 (19)	0.068 (6)*
C16	0.6193 (2)	0.47157 (13)	0.26132 (13)	0.0352 (3)
C17	0.5210 (2)	0.51095 (15)	0.33320 (15)	0.0404 (4)
H17A	0.582 (2)	0.5549 (16)	0.4262 (16)	0.047 (4)*
C18	0.3451 (2)	0.48794 (16)	0.27609 (15)	0.0439 (4)
H18A	0.281 (2)	0.5150 (18)	0.3262 (16)	0.055 (5)*
C19	0.2610 (2)	0.42459 (15)	0.14348 (15)	0.0412 (4)
C20	0.3557 (2)	0.38460 (16)	0.06964 (15)	0.0431 (4)
H20A	0.297 (2)	0.3412 (18)	−0.0231 (17)	0.056 (5)*
C21	0.5317 (2)	0.40803 (15)	0.12846 (14)	0.0412 (4)
H21A	0.598 (2)	0.3788 (17)	0.0732 (16)	0.052 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0992 (11)	0.0463 (6)	0.0503 (7)	0.0314 (7)	0.0388 (7)	0.0165 (5)

O2	0.0913 (11)	0.0402 (6)	0.0726 (8)	0.0205 (6)	0.0498 (8)	0.0096 (6)
O3	0.0476 (8)	0.0696 (8)	0.0573 (8)	0.0270 (6)	0.0158 (6)	0.0081 (6)
N1	0.0922 (13)	0.0422 (8)	0.0493 (8)	0.0227 (8)	0.0355 (8)	0.0142 (6)
N2	0.0491 (9)	0.0450 (7)	0.0508 (8)	0.0142 (6)	0.0180 (6)	0.0160 (6)
C1	0.0478 (10)	0.0381 (8)	0.0402 (8)	0.0191 (7)	0.0162 (7)	0.0144 (6)
C2	0.0769 (13)	0.0433 (9)	0.0463 (9)	0.0213 (9)	0.0281 (9)	0.0168 (8)
C3	0.0923 (16)	0.0427 (9)	0.0621 (11)	0.0231 (10)	0.0357 (11)	0.0241 (9)
C4	0.0978 (17)	0.0371 (9)	0.0669 (12)	0.0208 (10)	0.0369 (11)	0.0125 (9)
C5	0.132 (2)	0.0432 (10)	0.0587 (12)	0.0238 (11)	0.0485 (13)	0.0097 (9)
C6	0.0495 (10)	0.0409 (8)	0.0380 (8)	0.0218 (7)	0.0156 (7)	0.0135 (7)
C7	0.0499 (11)	0.0369 (8)	0.0492 (9)	0.0206 (7)	0.0211 (8)	0.0152 (7)
C8	0.0438 (9)	0.0325 (7)	0.0377 (8)	0.0152 (6)	0.0161 (7)	0.0093 (6)
C9	0.0419 (10)	0.0329 (7)	0.0491 (9)	0.0140 (7)	0.0199 (7)	0.0108 (7)
C10	0.0423 (9)	0.0346 (7)	0.0472 (8)	0.0126 (7)	0.0172 (7)	0.0075 (7)
C11	0.0404 (9)	0.0348 (7)	0.0496 (9)	0.0133 (7)	0.0132 (7)	0.0110 (7)
C12	0.0771 (14)	0.0387 (9)	0.0689 (12)	0.0200 (9)	0.0284 (11)	0.0131 (9)
C13	0.1020 (18)	0.0371 (10)	0.0973 (16)	0.0189 (11)	0.0391 (14)	0.0247 (11)
C14	0.0974 (18)	0.0550 (12)	0.0934 (16)	0.0188 (11)	0.0427 (14)	0.0405 (12)
C15	0.0765 (15)	0.0599 (11)	0.0652 (12)	0.0200 (10)	0.0317 (11)	0.0285 (10)
C16	0.0438 (9)	0.0261 (6)	0.0379 (7)	0.0128 (6)	0.0182 (6)	0.0112 (6)
C17	0.0494 (10)	0.0371 (7)	0.0351 (8)	0.0163 (7)	0.0183 (7)	0.0092 (6)
C18	0.0475 (10)	0.0432 (8)	0.0465 (9)	0.0205 (7)	0.0246 (8)	0.0102 (7)
C19	0.0417 (9)	0.0358 (7)	0.0475 (8)	0.0154 (7)	0.0174 (7)	0.0133 (7)
C20	0.0466 (10)	0.0410 (8)	0.0366 (8)	0.0135 (7)	0.0153 (7)	0.0071 (7)
C21	0.0464 (10)	0.0385 (8)	0.0405 (8)	0.0164 (7)	0.0222 (7)	0.0078 (6)

Geometric parameters (Å, º)

O1—C6	1.2135 (17)	C8—H8A	0.996 (15)
O2—C10	1.2114 (18)	C9—C10	1.504 (2)
O3—C19	1.3689 (19)	C9—H9B	0.975 (18)
O3—H03A	0.93 (2)	C9—H9A	1.017 (17)
N1—C1	1.3295 (19)	C10—C11	1.504 (2)
N1—C5	1.338 (2)	C11—C12	1.387 (2)
N2—C15	1.339 (2)	C12—C13	1.372 (3)
N2—C11	1.340 (2)	C12—H12A	0.96 (2)
C1—C2	1.375 (2)	C13—C14	1.360 (3)
C1—C6	1.504 (2)	C13—H13A	0.99 (3)
C2—C3	1.375 (2)	C14—C15	1.376 (3)
C2—H2A	0.960 (18)	C14—H14A	0.95 (3)
C3—C4	1.357 (3)	C15—H15A	0.966 (19)
C3—H3A	0.94 (2)	C16—C21	1.389 (2)
C4—C5	1.368 (3)	C16—C17	1.391 (2)
C4—H4A	0.99 (2)	C17—C18	1.378 (2)
C5—H5A	0.99 (2)	C17—H17A	0.971 (16)
C6—C7	1.502 (2)	C18—C19	1.384 (2)
C7—C8	1.538 (2)	C18—H18A	0.938 (18)
C7—H7A	1.006 (19)	C19—C20	1.384 (2)

C7—H7B	1.001 (16)	C20—C21	1.381 (2)
C8—C16	1.513 (2)	C20—H20A	0.967 (17)
C8—C9	1.532 (2)	C21—H21A	0.998 (17)

C19—O3—H03A	109.1 (15)	H9B—C9—H9A	104.6 (13)
C1—N1—C5	116.28 (15)	O2—C10—C9	121.67 (14)
C15—N2—C11	116.90 (15)	O2—C10—C11	119.15 (13)
N1—C1—C2	122.88 (14)	C9—C10—C11	119.08 (13)
N1—C1—C6	117.47 (13)	N2—C11—C12	122.63 (15)
C2—C1—C6	119.64 (14)	N2—C11—C10	118.46 (13)
C1—C2—C3	119.43 (16)	C12—C11—C10	118.91 (15)
C1—C2—H2A	118.1 (11)	C13—C12—C11	119.0 (2)
C3—C2—H2A	122.4 (11)	C13—C12—H12A	121.8 (12)
C4—C3—C2	118.47 (17)	C11—C12—H12A	119.2 (12)
C4—C3—H3A	121.3 (12)	C14—C13—C12	118.97 (19)
C2—C3—H3A	120.2 (12)	C14—C13—H13A	121.0 (15)
C3—C4—C5	118.65 (17)	C12—C13—H13A	120.0 (15)
C3—C4—H4A	122.3 (12)	C13—C14—C15	119.1 (2)
C5—C4—H4A	119.1 (12)	C13—C14—H14A	123.3 (14)
N1—C5—C4	124.27 (18)	C15—C14—H14A	117.6 (15)
N1—C5—H5A	114.4 (13)	N2—C15—C14	123.4 (2)
C4—C5—H5A	121.3 (13)	N2—C15—H15A	115.5 (12)
O1—C6—C7	122.05 (14)	C14—C15—H15A	121.1 (12)
O1—C6—C1	118.79 (14)	C21—C16—C17	116.72 (14)
C7—C6—C1	119.14 (13)	C21—C16—C8	121.07 (13)
C6—C7—C8	112.24 (13)	C17—C16—C8	122.21 (13)
C6—C7—H7A	104.7 (10)	C18—C17—C16	121.93 (14)
C8—C7—H7A	109.3 (10)	C18—C17—H17A	121.3 (9)
C6—C7—H7B	110.5 (9)	C16—C17—H17A	116.7 (10)
C8—C7—H7B	111.5 (9)	C17—C18—C19	120.22 (15)
H7A—C7—H7B	108.3 (14)	C17—C18—H18A	120.7 (10)
C16—C8—C9	110.96 (12)	C19—C18—H18A	119.0 (11)
C16—C8—C7	110.90 (12)	O3—C19—C20	122.26 (14)
C9—C8—C7	110.75 (12)	O3—C19—C18	118.70 (14)
C16—C8—H8A	107.9 (9)	C20—C19—C18	119.04 (15)
C9—C8—H8A	108.1 (9)	C21—C20—C19	119.99 (14)
C7—C8—H8A	108.1 (8)	C21—C20—H20A	121.1 (10)
C10—C9—C8	112.42 (13)	C19—C20—H20A	118.9 (10)
C10—C9—H9B	104.4 (10)	C20—C21—C16	122.10 (14)
C8—C9—H9B	112.2 (10)	C20—C21—H21A	118.6 (9)
C10—C9—H9A	110.8 (9)	C16—C21—H21A	119.3 (10)
C8—C9—H9A	111.9 (10)		

C5—N1—C1—C2	0.8 (3)	C9—C10—C11—N2	11.1 (2)
C5—N1—C1—C6	−178.05 (18)	O2—C10—C11—C12	8.4 (3)
N1—C1—C2—C3	0.3 (3)	C9—C10—C11—C12	−168.07 (16)
C6—C1—C2—C3	179.14 (17)	N2—C11—C12—C13	−1.1 (3)
C1—C2—C3—C4	−0.7 (3)	C10—C11—C12—C13	178.02 (18)

C2—C3—C4—C5	0.1 (3)	C11—C12—C13—C14	0.1 (4)
C1—N1—C5—C4	−1.6 (4)	C12—C13—C14—C15	0.7 (4)
C3—C4—C5—N1	1.1 (4)	C11—N2—C15—C14	−0.4 (3)
N1—C1—C6—O1	164.80 (16)	C13—C14—C15—N2	−0.6 (4)
C2—C1—C6—O1	−14.1 (2)	C9—C8—C16—C21	64.71 (16)
N1—C1—C6—C7	−16.5 (2)	C7—C8—C16—C21	−58.83 (17)
C2—C1—C6—C7	164.56 (16)	C9—C8—C16—C17	−114.63 (14)
O1—C6—C7—C8	−30.0 (2)	C7—C8—C16—C17	121.83 (14)
C1—C6—C7—C8	151.38 (14)	C21—C16—C17—C18	0.2 (2)
C6—C7—C8—C16	−65.15 (17)	C8—C16—C17—C18	179.58 (13)
C6—C7—C8—C9	171.20 (13)	C16—C17—C18—C19	−0.3 (2)
C16—C8—C9—C10	72.76 (16)	C17—C18—C19—O3	−179.03 (13)
C7—C8—C9—C10	−163.62 (14)	C17—C18—C19—C20	0.2 (2)
C8—C9—C10—O2	16.4 (2)	O3—C19—C20—C21	179.21 (13)
C8—C9—C10—C11	−167.22 (13)	C18—C19—C20—C21	0.0 (2)
C15—N2—C11—C12	1.2 (3)	C19—C20—C21—C16	−0.1 (2)
C15—N2—C11—C10	−177.89 (16)	C17—C16—C21—C20	0.0 (2)
O2—C10—C11—N2	−172.47 (15)	C8—C16—C21—C20	−179.38 (13)

Hydrogen-bond geometry (Å, º)

Cg3 is the centroid of the C16–C21 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H03*A*···N2^{i}	0.93 (2)	2.00 (2)	2.8940 (19)	160 (2)
C12—H12*A*···O2^{ii}	0.96 (2)	2.48 (2)	3.312 (3)	145 (2)
C4a—H4a···*Cg*3^{iii}	0.99 (2)	0.98 (2)	3.825 (2)	144 (2)

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