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3-Anilino-1,3-di-2-pyridylpropan-1-one

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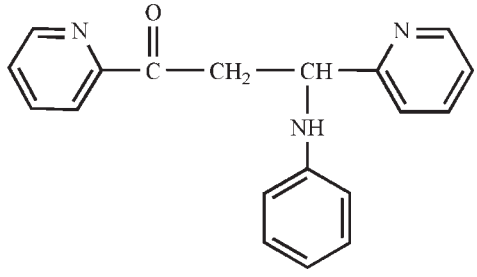
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.048; wR factor = 0.118; data-to-parameter ratio = 9.4.

The title compound, $C_{19}H_{17}N_3O$, was prepared by the 1,4-addition reaction of 1,3-di-2-pyridylprop-2-en-1-one with aniline, and includes one chiral C atom of the methine group with an *R* configuration. The crystal structure is stabilized by intermolecular N—H···N and C—H···O hydrogen bonds. The crystal structure also exhibits weak intermolecular C—H···π interactions between a pyridyl H atom and the phenyl ring of adjacent molecules.

Related literature

For properties of binucleating ligand coordination compounds, see: Casalino *et al.* (2009); Clare *et al.* (2004); Lam *et al.* (1996). For multiple pyridyl compounds, see: Huang *et al.* (2008). For related structures, see: Champouret *et al.* (2006); Murthy *et al.* (2001).



Experimental

Crystal data

$C_{19}H_{17}N_3O$
M_r = 303.36
Orthorhombic, $P2_12_12_1$
a = 9.316 (2) Å
b = 10.275 (2) Å
c = 16.652 (3) Å
V = 1594.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 K
0.35 × 0.30 × 0.24 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick (2000)
T_{min} = 0.950, T_{max} = 0.976
7562 measured reflections
1961 independent reflections
1040 reflections with $I > 2\sigma(I)$
R_{int} = 0.077

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.118
S = 1.00
1961 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3N···N2^{i}	0.86	2.35	3.191 (4)	164
C10—H10···O1^{i}	0.93	2.62	3.398 (5)	141
C3—H3···Cg^{ii}	0.93	2.77	3.548 (5)	142

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$. *Cg* is the centroid of the C14–C19 phenyl ring.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2115).

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supporting information

Acta Cryst. (2009). E**65**, o2739 [https://doi.org/10.1107/S160053680904121X]

3-Anilino-1,3-di-2-pyridylpropan-1-one

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S1. Comment

The binucleating ligand has continued to arouse interest among chemists, because the extensive investigation of binucleating ligands plays a key role in bimetallic chemistry. These coordination compounds were potentially applied in bioinorganic chemistry, homogeneous catalysis, magnetic exchange processes, and information of performance on important enzymes (Lam *et al.*, 1996, Clare *et al.*, 2004 & Casalino *et al.*, 2009). Furthermore, compounds comprising multiple pyridyl groups are widely used in the design and self-assembly of metal-organic architectures (Huang *et al.*, 2008). Here we report the crystal structure of title compound (I) (Fig. 1).

The bond distances and angles in (I) are consistent with the values in related structures (Champouret *et al.*, 2006 & Murthy *et al.*, 2001). The chiral C8 atom possesses the expected R configuaration. The molecualr packing (Fig. 2) is stabiized by intermolecular N—H···N and C—H···O hydrogen bonds; the first between the amino H atom and the pyridyl (C9–C13/N2) N atom, with a N3—H3···N2^{i}, the second between the pyridyl (C9–C13/N2) H atom and the oxygen of the C═O unit, with a C10—H10···O1^{i}, respectively (Table 1). The crystal packing (Fig. 3) is further stabilized by intermolecular C—H···π interactions between the pyridyl (C1–C5/N1) H atom and the phenyl ring, with a C3—H3···Cg^{ii} (Table 1; Cg is the centroid of the C14–C19 phenyl ring).

S2. Experimental

1,3-di-2-pyridyl-2-en-1-one (5 mmol/1.044 g) was mixed with aniline (6 mmol/0.558 g) in toluene (20 ml). And then the phosphotungsitic (0.01 g) in water (10 ml) was added dropwise and refluxed for 2 h. The insoluble materials were produced, and then removed by filtration. The organic layer was kept at room temperature for about two days. Yellow-colored and block shaped crystals were collected (yield 67.6%).

S3. Refinement

All the Friedel pairs were merged. H atoms were positioned geometrically and allowed to ride on their parent atoms, with N—H and C—H distances of 0.86 and 0.93–0.96 Å, respectively, and with U_{iso}(H) = 1.2U_{eq} of the parent atoms.

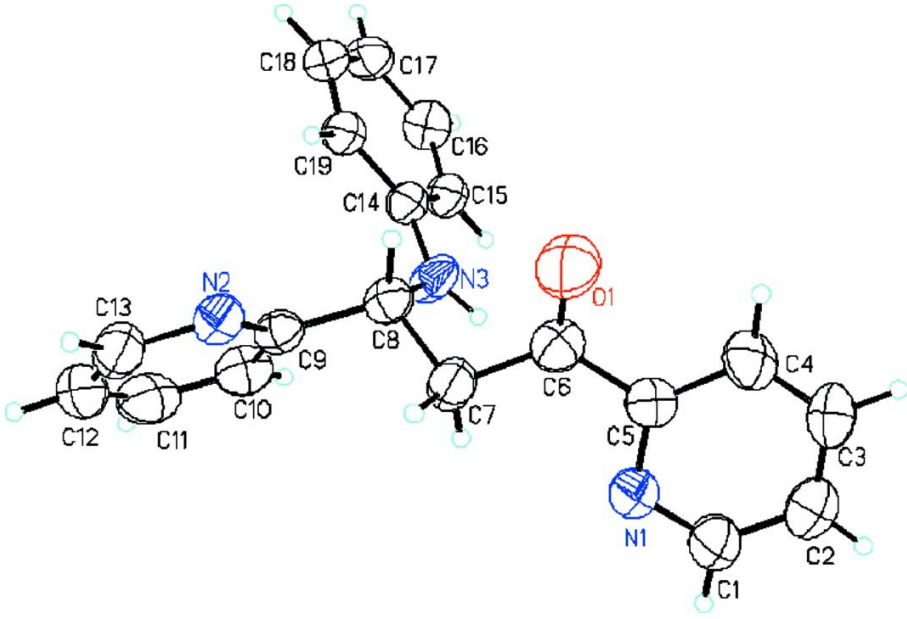

Figure 1

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 a small cycles of arbitrary radius.

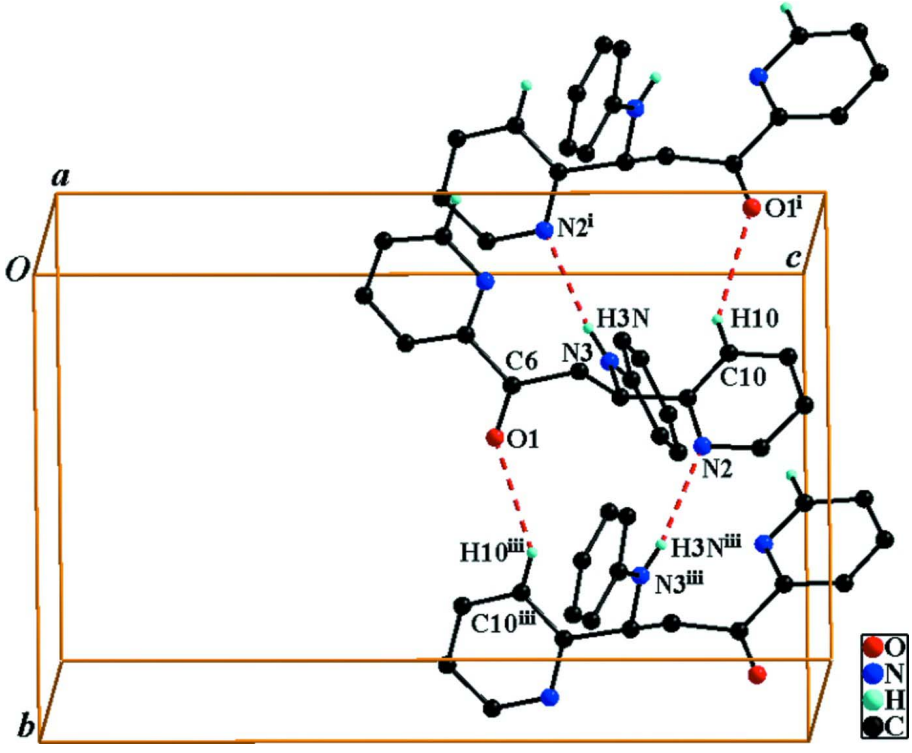

Figure 2

C—H···O and N—H···N hydrogen bonds (dotted lines) in the title compound. [Symmetry codes: (i) - *x* + 1 , *y* - 1/2, - *z* + 3/2; (iii) - *x* + 1, *y* + 1/2, - *z* + 3/2.]

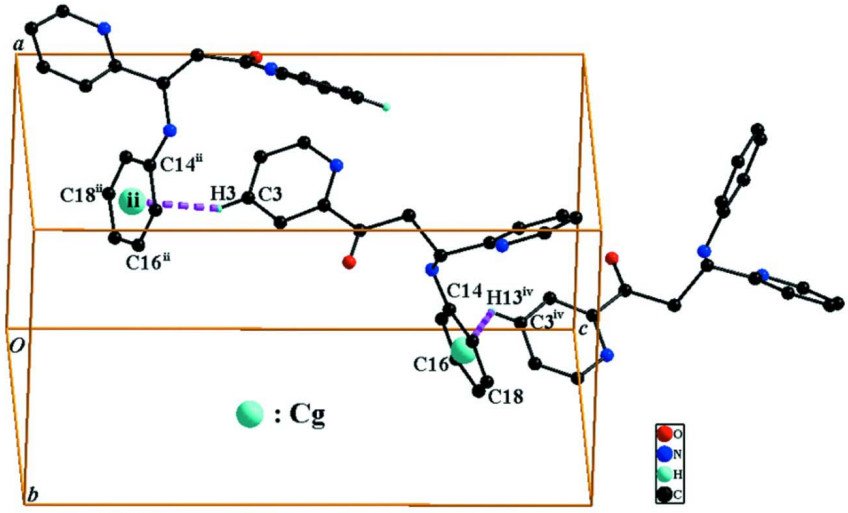

Figure 3

C—H···π interactions (dotted lines) in the title compound. Cg denotes the ring centroid. [Symmetry codes: (ii) x + 1/2, - y + 1/2, - z + 1; (iv) - x + 1/2, - y, z + 1/2.]

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Crystal data

$C_{19}H_{17}N_3O$
M_r = 303.36
Orthorhombic, $P2_12_12_1$
Hall symbol: p 2ac 2ab
a = 9.316 (2) Å
b = 10.275 (2) Å
c = 16.652 (3) Å
V = 1594.0 (5) Å^3
Z = 4
F(000) = 640
D_x = 1.264 Mg m^{-3}
Melting point: 400 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1025 reflections
θ = 2.3–27.0°
μ = 0.08 mm^{-1}
T = 293 K
Block, yellow
0.35 × 0.30 × 0.24 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.0 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick (2000)
T_{min} = 0.950, T_{max} = 0.976
7562 measured reflections
1961 independent reflections
1040 reflections with $I > 2\sigma(I)$
R_{int} = 0.077
θ_{max} = 27.0°, θ_{min} = 2.3°
h = −11→10
k = −13→13
l = −13→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.118$
$S = 1.00$
1961 reflections
208 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0005P)^2 + 0.0531P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ 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.

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 $F^2 > 2$sigma(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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5253 (4)	0.4411 (3)	0.5808 (2)	0.0934 (11)
N1	0.6804 (4)	0.1340 (3)	0.5653 (2)	0.0659 (9)
N2	0.6078 (3)	0.4744 (3)	0.8480 (2)	0.0551 (8)
N3	0.3789 (3)	0.2540 (3)	0.7351 (2)	0.0628 (9)
H3N	0.3971	0.1851	0.7076	0.075*
C1	0.7052 (5)	0.0421 (5)	0.5109 (3)	0.0828 (14)
H1	0.7626	−0.0277	0.5260	0.099*
C2	0.6530 (5)	0.0426 (5)	0.4350 (3)	0.0841 (14)
H2	0.6734	−0.0252	0.3997	0.101*
C3	0.5700 (5)	0.1446 (5)	0.4115 (3)	0.0754 (13)
H3	0.5324	0.1480	0.3598	0.090*
C4	0.5428 (5)	0.2424 (4)	0.4655 (3)	0.0693 (12)
H4	0.4868	0.3135	0.4508	0.083*
C5	0.5998 (4)	0.2338 (3)	0.5418 (2)	0.0531 (10)
C6	0.5753 (4)	0.3385 (4)	0.6019 (3)	0.0612 (11)
C7	0.6147 (4)	0.3152 (4)	0.6885 (2)	0.0614 (11)
H7A	0.6977	0.3677	0.7021	0.074*
H7B	0.6405	0.2245	0.6956	0.074*
C8	0.4910 (4)	0.3490 (3)	0.7454 (2)	0.0512 (10)
H8	0.4527	0.4340	0.7293	0.061*
C9	0.5452 (4)	0.3607 (3)	0.8309 (2)	0.0493 (9)
C10	0.5356 (4)	0.2627 (4)	0.8858 (3)	0.0660 (11)
H10	0.4912	0.1846	0.8726	0.079*
C11	0.5924 (5)	0.2811 (5)	0.9609 (3)	0.0828 (14)
H11A	0.5857	0.2156	0.9992	0.099*

C12	0.6590 (5)	0.3961 (6)	0.9794 (3)	0.0842 (15)
H12	0.7000	0.4101	1.0296	0.101*
C13	0.6627 (4)	0.4886 (4)	0.9215 (3)	0.0701 (13)
H13	0.7065	0.5674	0.9339	0.084*
C14	0.2431 (4)	0.2689 (3)	0.7675 (2)	0.0504 (9)
C15	0.1444 (4)	0.1682 (3)	0.7591 (2)	0.0552 (10)
H15	0.1720	0.0907	0.7348	0.066*
C16	0.0075 (5)	0.1827 (4)	0.7863 (3)	0.0652 (11)
H16	−0.0574	0.1149	0.7794	0.078*
C17	−0.0373 (4)	0.2942 (4)	0.8235 (3)	0.0654 (11)
H17	−0.1312	0.3027	0.8417	0.078*
C18	0.0597 (4)	0.3928 (4)	0.8330 (3)	0.0611 (11)
H18	0.0312	0.4690	0.8586	0.073*
C19	0.2000 (4)	0.3816 (3)	0.8054 (2)	0.0545 (10)
H19	0.2644	0.4498	0.8124	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.135 (3)	0.0631 (17)	0.082 (2)	0.0240 (19)	−0.001 (2)	0.0020 (17)
N1	0.071 (2)	0.067 (2)	0.060 (2)	0.0110 (19)	−0.0054 (18)	−0.008 (2)
N2	0.0545 (19)	0.0534 (19)	0.057 (2)	−0.0029 (16)	0.0051 (16)	−0.0056 (17)
N3	0.0509 (19)	0.0561 (19)	0.082 (2)	−0.0027 (16)	0.0062 (17)	−0.0321 (19)
C1	0.098 (4)	0.081 (3)	0.069 (3)	0.023 (3)	−0.011 (3)	−0.011 (3)
C2	0.096 (4)	0.083 (3)	0.073 (3)	0.008 (3)	0.002 (3)	−0.020 (3)
C3	0.094 (4)	0.082 (3)	0.050 (3)	−0.007 (3)	0.007 (2)	0.000 (3)
C4	0.084 (3)	0.066 (3)	0.057 (3)	−0.002 (2)	0.002 (2)	0.019 (2)
C5	0.055 (2)	0.047 (2)	0.057 (3)	−0.005 (2)	0.0043 (19)	0.008 (2)
C6	0.066 (3)	0.056 (2)	0.062 (3)	0.001 (2)	0.006 (2)	0.002 (2)
C7	0.064 (3)	0.065 (2)	0.055 (3)	0.002 (2)	0.005 (2)	−0.009 (2)
C8	0.050 (2)	0.0438 (19)	0.060 (3)	−0.0032 (18)	0.0016 (18)	−0.0048 (19)
C9	0.046 (2)	0.0425 (19)	0.060 (2)	0.0001 (18)	0.0037 (19)	−0.002 (2)
C10	0.068 (3)	0.052 (2)	0.077 (3)	0.001 (2)	0.009 (2)	0.012 (2)
C11	0.091 (4)	0.087 (3)	0.069 (3)	0.028 (3)	0.015 (3)	0.028 (3)
C12	0.084 (4)	0.110 (4)	0.058 (3)	0.025 (3)	−0.007 (3)	−0.008 (3)
C13	0.063 (3)	0.076 (3)	0.071 (3)	−0.001 (2)	0.002 (2)	−0.021 (3)
C14	0.052 (2)	0.046 (2)	0.054 (2)	0.0035 (18)	−0.0036 (19)	−0.007 (2)
C15	0.060 (3)	0.048 (2)	0.058 (2)	−0.001 (2)	−0.002 (2)	−0.0083 (19)
C16	0.064 (3)	0.064 (3)	0.068 (3)	−0.013 (2)	0.005 (2)	0.001 (2)
C17	0.050 (2)	0.079 (3)	0.067 (3)	0.009 (2)	0.006 (2)	0.000 (2)
C18	0.065 (3)	0.059 (2)	0.059 (3)	0.015 (2)	−0.003 (2)	−0.005 (2)
C19	0.055 (3)	0.051 (2)	0.058 (2)	0.0027 (18)	−0.006 (2)	−0.0078 (19)

Geometric parameters (Å, º)

O1—C6	1.205 (4)	C8—C9	1.516 (5)
N1—C5	1.329 (4)	C8—H8	0.9800
N1—C1	1.330 (5)	C9—C10	1.363 (5)

N2—C13	1.335 (5)	C10—C11	1.370 (7)
N2—C9	1.336 (4)	C10—H10	0.9300
N3—C14	1.384 (4)	C11—C12	1.370 (6)
N3—C8	1.440 (4)	C11—H11A	0.9300
N3—H3N	0.8600	C12—C13	1.354 (6)
C1—C2	1.354 (7)	C12—H12	0.9300
C1—H1	0.9300	C13—H13	0.9300
C2—C3	1.359 (6)	C14—C19	1.379 (5)
C2—H2	0.9300	C14—C15	1.390 (5)
C3—C4	1.372 (6)	C15—C16	1.362 (5)
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.380 (6)	C16—C17	1.367 (5)
C4—H4	0.9300	C16—H16	0.9300
C5—C6	1.487 (5)	C17—C18	1.366 (5)
C6—C7	1.506 (6)	C17—H17	0.9300
C7—C8	1.531 (5)	C18—C19	1.391 (5)
C7—H7A	0.9700	C18—H18	0.9300
C7—H7B	0.9700	C19—H19	0.9300

C5—N1—C1	116.4 (4)	C7—C8—H8	107.9
C13—N2—C9	117.2 (4)	N2—C9—C10	122.1 (4)
C14—N3—C8	122.8 (3)	N2—C9—C8	114.5 (3)
C14—N3—H3N	118.6	C10—C9—C8	123.4 (3)
C8—N3—H3N	118.6	C9—C10—C11	119.0 (4)
N1—C1—C2	124.8 (5)	C9—C10—H10	120.5
N1—C1—H1	117.6	C11—C10—H10	120.5
C2—C1—H1	117.6	C12—C11—C10	119.9 (4)
C1—C2—C3	118.4 (5)	C12—C11—H11A	120.0
C1—C2—H2	120.8	C10—C11—H11A	120.0
C3—C2—H2	120.8	C13—C12—C11	117.2 (4)
C2—C3—C4	118.8 (4)	C13—C12—H12	121.4
C2—C3—H3	120.6	C11—C12—H12	121.4
C4—C3—H3	120.6	N2—C13—C12	124.5 (4)
C3—C4—C5	119.0 (4)	N2—C13—H13	117.7
C3—C4—H4	120.5	C12—C13—H13	117.7
C5—C4—H4	120.5	C19—C14—N3	122.5 (3)
N1—C5—C4	122.5 (4)	C19—C14—C15	118.6 (3)
N1—C5—C6	116.5 (4)	N3—C14—C15	118.9 (3)
C4—C5—C6	121.0 (4)	C16—C15—C14	120.3 (3)
O1—C6—C5	119.8 (4)	C16—C15—H15	119.9
O1—C6—C7	120.8 (4)	C14—C15—H15	119.9
C5—C6—C7	119.5 (3)	C15—C16—C17	121.9 (4)
C6—C7—C8	111.9 (3)	C15—C16—H16	119.0
C6—C7—H7A	109.2	C17—C16—H16	119.0
C8—C7—H7A	109.2	C18—C17—C16	118.1 (4)
C6—C7—H7B	109.2	C18—C17—H17	120.9
C8—C7—H7B	109.2	C16—C17—H17	120.9
H7A—C7—H7B	107.9	C17—C18—C19	121.5 (4)

N3—C8—C9	114.0 (3)	C17—C18—H18	119.3
N3—C8—C7	108.5 (3)	C19—C18—H18	119.3
C9—C8—C7	110.4 (3)	C14—C19—C18	119.6 (4)
N3—C8—H8	107.9	C14—C19—H19	120.2
C9—C8—H8	107.9	C18—C19—H19	120.2
C5—N1—C1—C2	−1.0 (7)	N3—C8—C9—N2	−157.1 (3)
N1—C1—C2—C3	0.7 (8)	C7—C8—C9—N2	80.3 (4)
C1—C2—C3—C4	0.0 (7)	N3—C8—C9—C10	24.8 (5)
C2—C3—C4—C5	−0.3 (7)	C7—C8—C9—C10	−97.7 (4)
C1—N1—C5—C4	0.7 (6)	N2—C9—C10—C11	−0.2 (6)
C1—N1—C5—C6	−178.4 (4)	C8—C9—C10—C11	177.7 (4)
C3—C4—C5—N1	−0.1 (6)	C9—C10—C11—C12	−0.8 (7)
C3—C4—C5—C6	179.0 (4)	C10—C11—C12—C13	1.3 (7)
N1—C5—C6—O1	167.1 (4)	C9—N2—C13—C12	0.0 (6)
C4—C5—C6—O1	−12.0 (6)	C11—C12—C13—N2	−0.9 (7)
N1—C5—C6—C7	−12.3 (5)	C8—N3—C14—C19	6.6 (5)
C4—C5—C6—C7	168.6 (4)	C8—N3—C14—C15	−175.7 (3)
O1—C6—C7—C8	51.2 (5)	C19—C14—C15—C16	1.5 (6)
C5—C6—C7—C8	−129.4 (3)	N3—C14—C15—C16	−176.3 (4)
C14—N3—C8—C9	67.7 (4)	C14—C15—C16—C17	−1.0 (6)
C14—N3—C8—C7	−168.8 (3)	C15—C16—C17—C18	0.0 (6)
C6—C7—C8—N3	69.9 (4)	C16—C17—C18—C19	0.6 (6)
C6—C7—C8—C9	−164.4 (3)	N3—C14—C19—C18	176.7 (4)
C13—N2—C9—C10	0.6 (5)	C15—C14—C19—C18	−0.9 (5)
C13—N2—C9—C8	−177.4 (3)	C17—C18—C19—C14	−0.1 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···N2^{i}	0.86	2.35	3.191 (4)	164
C10—H10···O1^{i}	0.93	2.62	3.398 (5)	141
C3—H3···*Cg*^{ii}	0.93	2.77	3.548 (5)	142

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