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2-[3-Methoxy-5-(pyrimidin-2-yl)phenyl]-pyrimidine

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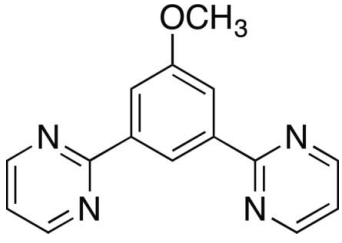
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.123; data-to-parameter ratio = 13.5.

The title compound, $C_{15}H_{12}N_4O$, was synthesized by a standard Suzuki cross-coupling reaction. The terminal pyrimidine rings are rotated at dihedral angles of 12.06 (4) and −13.13 (4)° with respect to the central benzene ring. In the crystal, the molecules are connected by two kinds of C—H···N hydrogen bonds, forming zigzag chains along the c axis. Weak π–π interactions between the benzene and one of the pyrimidine rings are also found and stack the molecules along the b axis [centroid–centroid distance = 4.112 (3) Å].

Related literature

For general background to the chemistry of tridentate NCN ligands, see: Pugh & Danopoulos (2007); Wu *et al.* (2009, 2012); Williams (2009); Wang *et al.* (2010). For the synthesis of the title compound, see: Avitia *et al.* (2011); Wakioka *et al.* (2010); Cardenas & Echavarren (1999).



Experimental

Crystal data

$C_{15}H_{12}N_4O$
M_r = 264.29
Orthorhombic, *Pbca*
a = 11.3779 (8) Å
b = 8.0954 (6) Å
c = 27.3371 (18) Å
V = 2518.0 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.28 × 0.21 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.800, T_{max} = 1.000
14088 measured reflections
2461 independent reflections
2059 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.123
S = 1.04
2461 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···N4^{i}	0.93	2.69	3.597 (2)	166
C15—H15B···N3^{ii}	0.96	2.69	3.547 (2)	149

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*.

This work was supported by STCSM (12JC1400200).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5297).

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2-[3-Methoxy-5-(pyrimidin-2-yl)phenyl]pyrimidine

Jiena Hu, Yang Wu, Dengqing Zhang, Xianying Li and Wusong Jin

S1. Comment

Pincer compounds have extensive applications in the coordination chemistry and material science (Pugh & Danopoulos, 2007). The most widely-used pincer ligand is terpyridine. Meanwhile, some tridentate NCN ligands with similar structures to terpyridine have also been reported (Williams, 2009; Wang *et al.*, 2010; Wu *et al.*, 2009). As part of our ongoing studies in this field (Wu *et al.*, 2012), we report here the crystal structure of a new pincer compound 1,3-di(pyrimin-2-yl)-5-methoxybenzene.

The molecular structure of the title compound is shown in Figure 1. The three aryl rings of the title compound are not coplanar, but dihedral angles of 12.06 (4)° and -13.13 (4)° are found between the terminal pyrimidine rings and central phenyl ring, respectively. In the crystal, intermolecular C9–H8···N4 and C15–H15B···N3 hydrogen bonds (Table 1) form zig-zag chains along *c*. The molecules stack along the *b* axis in such a way that the same pyrimidine-phenyl segment of two neighboring molecules overlap in opposite directions with a centroid-centroid distance of 4.112 (3) Å.

S2. Experimental

The compound was prepared using methods described in the literature (Avitia *et al.*, 2011; Wakioka *et al.*, 2010; Cardenas & Echavarren, 1999). A mixture of 1,3-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-5-methoxybenzene (600 mg, 1.67 mmol), 2-bromopyrimidine (800 mg, 5.03 mmol), K_2CO_3 aq (2.0 *M*, 8.40 ml) and $Pd(PPh_3)_4$ (390 mg, 0.251 mmol) in toluene (30 ml) was stirred at 110°C for 48 h under an argon atmosphere. After cooling to room temperature, the reaction mixture was poured into water, and extracted with CH_2Cl_2. The combined organic phase was washed with water and dried over anhydrous magnesium sulfate. After filtration and evaporation, the residue was purified with flash column chromatography (SiO_2, eluted with CH_2Cl_2/petroleum ether = 1:5) to obtain the 1,3-di(pyrimidin-2-yl)-5-methoxybenzene as a white powder (353 mg) in 80% yield**.** ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.16 (s, 1H), 8.83 (d, J = 4.8 Hz, 4H), 8.14 (d, J = 1.5 Hz, 2H), 7.20 (t, J = 4.8 Hz, 2H), 3.99 (s, 3H). ^{13}C NMR(100 MHz, $CDCl_3$) δ 164.30, 160.51, 157.23, 139.36, 120.85, 119.32, 115.95, 55.74. MS(EI): m/*z*= 263.95. A single-crystal was obtained by slow evaporation of an acetonitrile solution of the title compound.

S3. Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms with C—H = 0.96 (methyl) Å [U iso (H) = 1.5U eq (C)], and C—H = 0.93 (aromatic) Å [U iso (H) = 1.2U eq (C)].

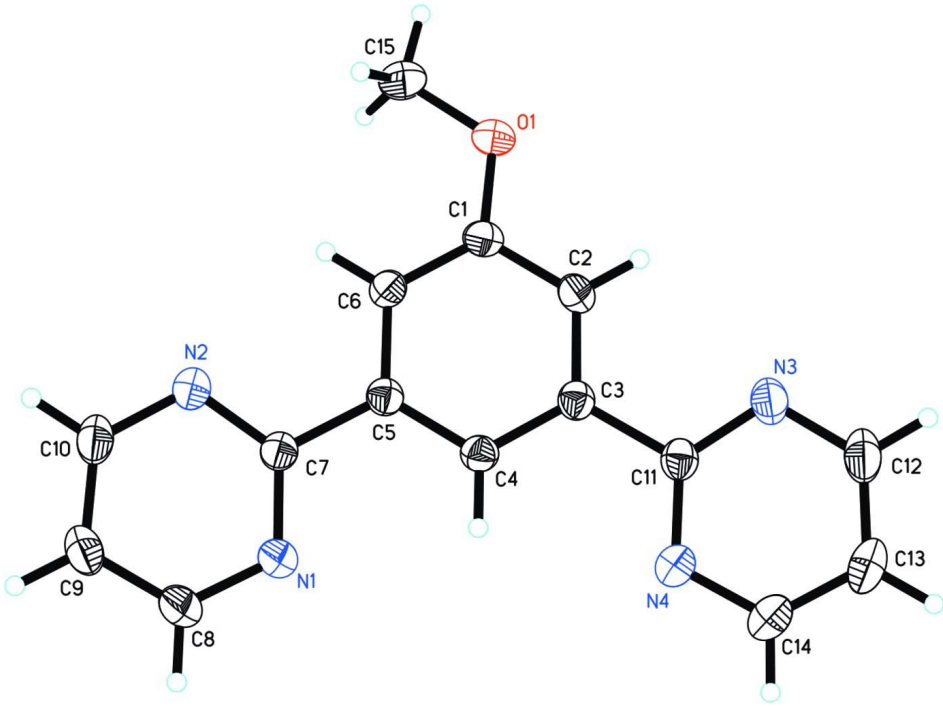

Figure 1

A perspective view of the title compound with displacement ellipsoids drawn at the 30% probability level.

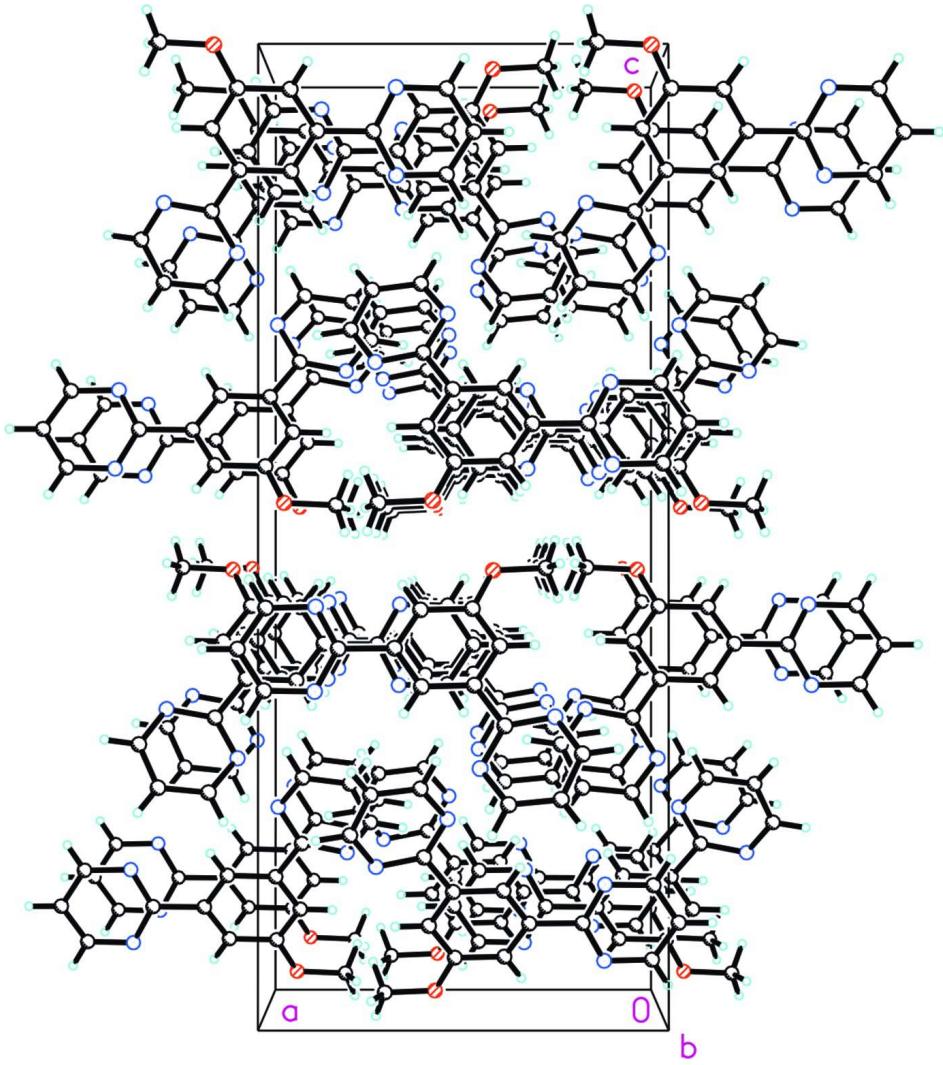

Figure 2

A view of the unit-cell contents of the title compound along the *b* axis.

2-[3-Methoxy-5-(pyrimidin-2-yl)phenyl]pyrimidine

Crystal data

$C_{15}H_{12}N_4O$
$M_r = 264.29$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 11.3779\ (8)$ Å
$b = 8.0954\ (6)$ Å
$c = 27.3371\ (18)$ Å
$V = 2518.0\ (3)$ Å^3
$Z = 8$
$F(000) = 1104$
$D_x = 1.394$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4404 reflections
$\theta = 4.7–56.5°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Prismatic, colourless
0.28 × 0.21 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.800, T_{max} = 1.000
14088 measured reflections
2461 independent reflections
2059 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 26.0°, θ_{min} = 1.5°
h = −14→12
k = −8→9
l = −33→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.123
S = 1.04
2461 reflections
182 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
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2 + 0.4657P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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.

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 $F^2 > \sigma(F^2)$ 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}
N1	1.03793 (11)	0.23473 (17)	0.28017 (4)	0.0556 (4)
N2	1.20879 (9)	0.20037 (16)	0.32842 (4)	0.0508 (3)
N3	0.65549 (10)	−0.04972 (17)	0.43132 (4)	0.0550 (3)
N4	0.65653 (10)	0.05619 (16)	0.35048 (4)	0.0519 (3)
O1	1.07030 (9)	−0.16817 (14)	0.46649 (3)	0.0572 (3)
C1	1.02113 (12)	−0.07963 (17)	0.42928 (4)	0.0434 (3)
C2	0.89954 (12)	−0.07835 (17)	0.42817 (5)	0.0444 (3)
H2	0.8574	−0.1340	0.4522	0.053*
C3	0.84023 (11)	0.00505 (16)	0.39159 (4)	0.0394 (3)
C4	0.90336 (11)	0.08851 (16)	0.35561 (4)	0.0387 (3)
H4	0.8639	0.1440	0.3308	0.046*
C5	1.02519 (11)	0.08866 (16)	0.35693 (4)	0.0380 (3)
C6	1.08424 (11)	0.00412 (16)	0.39390 (4)	0.0415 (3)
H6	1.1660	0.0040	0.3948	0.050*
C7	1.09451 (11)	0.17957 (16)	0.31955 (4)	0.0392 (3)
C8	1.10177 (14)	0.3194 (2)	0.24815 (6)	0.0609 (4)

H8	1.0644	0.3616	0.2206	0.073*
C9	1.21922 (13)	0.3476 (2)	0.25355 (6)	0.0554 (4)
H9	1.2626	0.4064	0.2306	0.067*
C10	1.26891 (12)	0.2841 (2)	0.29482 (6)	0.0567 (4)
H10	1.3490	0.3001	0.2998	0.068*
C11	0.70953 (11)	0.00467 (17)	0.39113 (4)	0.0417 (3)
C12	0.53848 (13)	−0.0495 (2)	0.43016 (6)	0.0627 (5)
H12	0.4977	−0.0875	0.4574	0.075*
C13	0.47587 (13)	0.0040 (2)	0.39070 (6)	0.0609 (4)
H13	0.3941	0.0056	0.3907	0.073*
C14	0.53930 (13)	0.0549 (2)	0.35126 (6)	0.0589 (4)
H14	0.4991	0.0905	0.3236	0.071*
C15	1.19460 (14)	−0.1760 (2)	0.46883 (6)	0.0639 (5)
H15A	1.2246	−0.2221	0.4390	0.096*
H15B	1.2177	−0.2443	0.4959	0.096*
H15C	1.2258	−0.0668	0.4732	0.096*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0475 (6)	0.0735 (9)	0.0457 (6)	−0.0099 (6)	−0.0043 (5)	0.0165 (6)
N2	0.0395 (6)	0.0582 (8)	0.0548 (7)	−0.0046 (5)	−0.0003 (5)	0.0092 (6)
N3	0.0450 (7)	0.0736 (9)	0.0466 (7)	−0.0109 (6)	0.0072 (5)	−0.0010 (6)
N4	0.0403 (6)	0.0610 (8)	0.0542 (7)	0.0031 (5)	0.0002 (5)	0.0046 (6)
O1	0.0531 (6)	0.0696 (7)	0.0491 (6)	0.0027 (5)	−0.0065 (4)	0.0201 (5)
C1	0.0468 (7)	0.0460 (8)	0.0375 (6)	0.0020 (6)	−0.0027 (5)	0.0030 (6)
C2	0.0463 (8)	0.0490 (8)	0.0378 (6)	−0.0033 (6)	0.0050 (5)	0.0046 (6)
C3	0.0387 (7)	0.0411 (7)	0.0383 (6)	−0.0012 (5)	0.0021 (5)	−0.0037 (5)
C4	0.0395 (7)	0.0415 (7)	0.0352 (6)	0.0008 (5)	−0.0013 (5)	0.0001 (5)
C5	0.0387 (7)	0.0395 (7)	0.0357 (6)	−0.0010 (5)	0.0012 (5)	−0.0027 (5)
C6	0.0359 (6)	0.0472 (8)	0.0414 (7)	0.0001 (5)	−0.0014 (5)	−0.0003 (6)
C7	0.0380 (6)	0.0410 (7)	0.0385 (6)	−0.0010 (5)	0.0006 (5)	−0.0021 (5)
C8	0.0611 (9)	0.0768 (11)	0.0449 (8)	−0.0123 (8)	−0.0027 (7)	0.0170 (8)
C9	0.0587 (9)	0.0581 (9)	0.0495 (8)	−0.0128 (7)	0.0122 (6)	0.0053 (7)
C10	0.0397 (7)	0.0626 (10)	0.0677 (10)	−0.0083 (7)	0.0078 (6)	0.0072 (8)
C11	0.0400 (7)	0.0425 (7)	0.0426 (7)	−0.0017 (5)	0.0036 (5)	−0.0050 (6)
C12	0.0465 (8)	0.0834 (12)	0.0582 (9)	−0.0140 (8)	0.0140 (7)	−0.0097 (8)
C13	0.0356 (7)	0.0702 (11)	0.0768 (11)	−0.0021 (7)	0.0063 (7)	−0.0170 (9)
C14	0.0416 (8)	0.0652 (10)	0.0699 (10)	0.0051 (7)	−0.0055 (7)	−0.0001 (8)
C15	0.0553 (9)	0.0735 (11)	0.0628 (9)	0.0086 (8)	−0.0128 (7)	0.0178 (8)

Geometric parameters (Å, º)

N1—C8	1.3280 (18)	C4—H4	0.9300
N1—C7	1.3316 (17)	C5—C6	1.3934 (17)
N2—C10	1.3310 (18)	C5—C7	1.4859 (17)
N2—C7	1.3333 (16)	C6—H6	0.9300
N3—C12	1.3317 (19)	C8—C9	1.364 (2)

N3—C11	1.3338 (16)	C8—H8	0.9300
N4—C11	1.3311 (17)	C9—C10	1.363 (2)
N4—C14	1.3340 (18)	C9—H9	0.9300
O1—C1	1.3643 (15)	C10—H10	0.9300
O1—C15	1.4171 (18)	C12—C13	1.363 (2)
C1—C6	1.3821 (18)	C12—H12	0.9300
C1—C2	1.3838 (18)	C13—C14	1.361 (2)
C2—C3	1.3826 (18)	C13—H13	0.9300
C2—H2	0.9300	C14—H14	0.9300
C3—C4	1.3928 (17)	C15—H15A	0.9600
C3—C11	1.4872 (18)	C15—H15B	0.9600
C4—C5	1.3867 (17)	C15—H15C	0.9600

C8—N1—C7	116.21 (12)	N1—C8—H8	118.3
C10—N2—C7	116.10 (12)	C9—C8—H8	118.3
C12—N3—C11	116.16 (13)	C10—C9—C8	115.66 (13)
C11—N4—C14	115.93 (12)	C10—C9—H9	122.2
C1—O1—C15	117.79 (11)	C8—C9—H9	122.2
O1—C1—C6	124.49 (12)	N2—C10—C9	123.39 (13)
O1—C1—C2	115.47 (11)	N2—C10—H10	118.3
C6—C1—C2	120.03 (11)	C9—C10—H10	118.3
C3—C2—C1	120.49 (12)	N4—C11—N3	125.60 (12)
C3—C2—H2	119.8	N4—C11—C3	117.35 (11)
C1—C2—H2	119.8	N3—C11—C3	117.04 (11)
C2—C3—C4	119.74 (12)	N3—C12—C13	122.78 (14)
C2—C3—C11	119.55 (11)	N3—C12—H12	118.6
C4—C3—C11	120.71 (11)	C13—C12—H12	118.6
C5—C4—C3	119.83 (11)	C14—C13—C12	116.48 (14)
C5—C4—H4	120.1	C14—C13—H13	121.8
C3—C4—H4	120.1	C12—C13—H13	121.8
C4—C5—C6	120.04 (11)	N4—C14—C13	123.02 (15)
C4—C5—C7	120.87 (11)	N4—C14—H14	118.5
C6—C5—C7	119.09 (11)	C13—C14—H14	118.5
C1—C6—C5	119.87 (12)	O1—C15—H15A	109.5
C1—C6—H6	120.1	O1—C15—H15B	109.5
C5—C6—H6	120.1	H15A—C15—H15B	109.5
N1—C7—N2	125.18 (12)	O1—C15—H15C	109.5
N1—C7—C5	117.74 (11)	H15A—C15—H15C	109.5
N2—C7—C5	117.07 (11)	H15B—C15—H15C	109.5
N1—C8—C9	123.44 (14)		

C15—O1—C1—C6	−0.5 (2)	C6—C5—C7—N1	168.58 (12)
C15—O1—C1—C2	178.92 (13)	C4—C5—C7—N2	167.50 (12)
O1—C1—C2—C3	−178.88 (12)	C6—C5—C7—N2	−11.89 (18)
C6—C1—C2—C3	0.6 (2)	C7—N1—C8—C9	1.4 (2)
C1—C2—C3—C4	−0.05 (19)	N1—C8—C9—C10	−0.6 (3)
C1—C2—C3—C11	179.97 (12)	C7—N2—C10—C9	0.5 (2)
C2—C3—C4—C5	−0.56 (18)	C8—C9—C10—N2	−0.4 (2)

C11—C3—C4—C5	179.41 (11)	C14—N4—C11—N3	1.3 (2)
C3—C4—C5—C6	0.63 (18)	C14—N4—C11—C3	−179.78 (13)
C3—C4—C5—C7	−178.76 (11)	C12—N3—C11—N4	−0.9 (2)
O1—C1—C6—C5	178.91 (12)	C12—N3—C11—C3	−179.84 (13)
C2—C1—C6—C5	−0.52 (19)	C2—C3—C11—N4	−166.49 (13)
C4—C5—C6—C1	−0.10 (18)	C4—C3—C11—N4	13.54 (18)
C7—C5—C6—C1	179.31 (12)	C2—C3—C11—N3	12.55 (18)
C8—N1—C7—N2	−1.3 (2)	C4—C3—C11—N3	−167.43 (12)
C8—N1—C7—C5	178.20 (13)	C11—N3—C12—C13	−0.5 (2)
C10—N2—C7—N1	0.4 (2)	N3—C12—C13—C14	1.4 (3)
C10—N2—C7—C5	−179.09 (13)	C11—N4—C14—C13	−0.2 (2)
C4—C5—C7—N1	−12.03 (18)	C12—C13—C14—N4	−1.0 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···N4^{i}	0.93	2.69	3.597 (2)	166
C15—H15*B*···N3^{ii}	0.96	2.69	3.547 (2)	149

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