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2-Chloro-*N*-ethyl-9-isopropyl-9*H*-purin-6-amine

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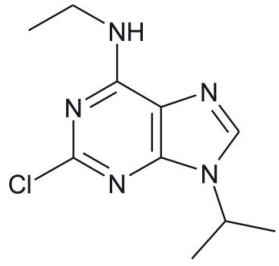
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.025; wR factor = 0.069; data-to-parameter ratio = 13.4.

In the title compound, $C_{10}H_{14}ClN_5$, the purine ring system is essentially planar, with an r.m.s. deviation from the least-squares plane defined by the nine constituent atoms of 0.0063 (11) Å. In the crystal, molecules are linked by weak N—H···N and C—H···π interactions.

Related literature

For the synthesis, see: Fiorini & Abel (1998). For related structures, see: Kubicki & Codding (2001); Rouchal *et al.* (2009*a*,*b*, 2010). For other related literature, see: Legraverend & Grierson (2006).



Experimental

Crystal data

$C_{10}H_{14}ClN_5$
M_r = 239.71
Monoclinic, $P2_1/n$
a = 8.1385 (2) Å
b = 9.6245 (2) Å
c = 14.8388 (3) Å
β = 92.997 (2)°
V = 1160.72 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 120 K
0.40 × 0.40 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire2 (large Be window) detector
Absorption correction: multi-scan *CrysAlis RED* (Oxford Diffraction, 2009)
T_{min} = 0.973, T_{max} = 1.000
13543 measured reflections
2041 independent reflections
1793 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.069
S = 1.07
2041 reflections
152 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

*Cg*1 is the center of gravity of the pyridine ring (C1/N1/C2–C4/ N2).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N4^{i}	0.837 (16)	2.205 (17)	2.9979 (16)	158.2 (15)
C8—H8···*Cg*1^{ii}	1.00 (1)	2.90 (1)	3.6403 (13)	131 (1)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2254).

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supporting information

Acta Cryst. (2012). E**68**, o2759 [doi:10.1107/S1600536812035933]

2-Chloro-*N*-ethyl-9-isopropyl-9*H*-purin-6-amine

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S1. Comment

The wide range of biological activities of di-, tri-, or tetrasubstituted purines is closely associated with essentially unlimited number of substituents that can be combined in the C2, C6, C8 and N9 positions of the purine ring (Legraverend & Grierson, 2006). The title molecule has been prepared as a part of our ongoing study of novel 2,6,9-trisubstituted purine series (Rouchal *et al.*, 2009*a*,*b*, 2010). To the best of our knowledge, the title compound has not been described in the literature so far.

In the title molecule (Fig. 1), the purine unit is essentially planar, with a r.m.s. deviation of 0.0063 (11) Å from the least-squares plane defined by the nine constituent atoms. Although all pyrimidine atoms lie essentially in plane, the ring is markedly deformed from regular hexagon geometry with N1–C2–N3 angle of 132.0 (11)°. In the crystal structure (Fig. 2), molecules are connected by weak intermolecular N—H···N and C—H···π interactions (Table 1).

S2. Experimental

The title compound was prepared according to slightly modified literature procedure (Fiorini & Abel, 1998). 2,6-Dichloro-9-isopropyl-9*H*-purine (100 mg, 0.43 mmol) and ethylamine hydrochloride (37 mg, 0.45 mmol) were dissolved in the mixture of DMF (3 cm^3) and triethylamine (87 mg, 0.86 mmol). The resulting solution was stirred at 90 °C for 3 h. Subsequently, the mixture was diluted with water and extracted with ethyl acetate (6 × 10 cm^3). Combined organic layers were washed twice with brine, dried over Na_2SO_4 and evaporated in vacuum. Crystallization of the crude product from diethyl ether at room temperature provided desired compound as colorless crystals (67 mg, 65%, mp 390–393 K) suitable for X-ray diffraction analysis.

S3. Refinement

All carbon bound H atoms were placed at calculated positions and were refined as riding with their U_{iso} set to either 1.2U_{eq} or 1.5U_{eq} (methyl) of the respective carrier atoms. The positions of methyl H atoms were optimized rotationally. Nitrogen bound H atom was located in a difference Fourier map and refined freely.

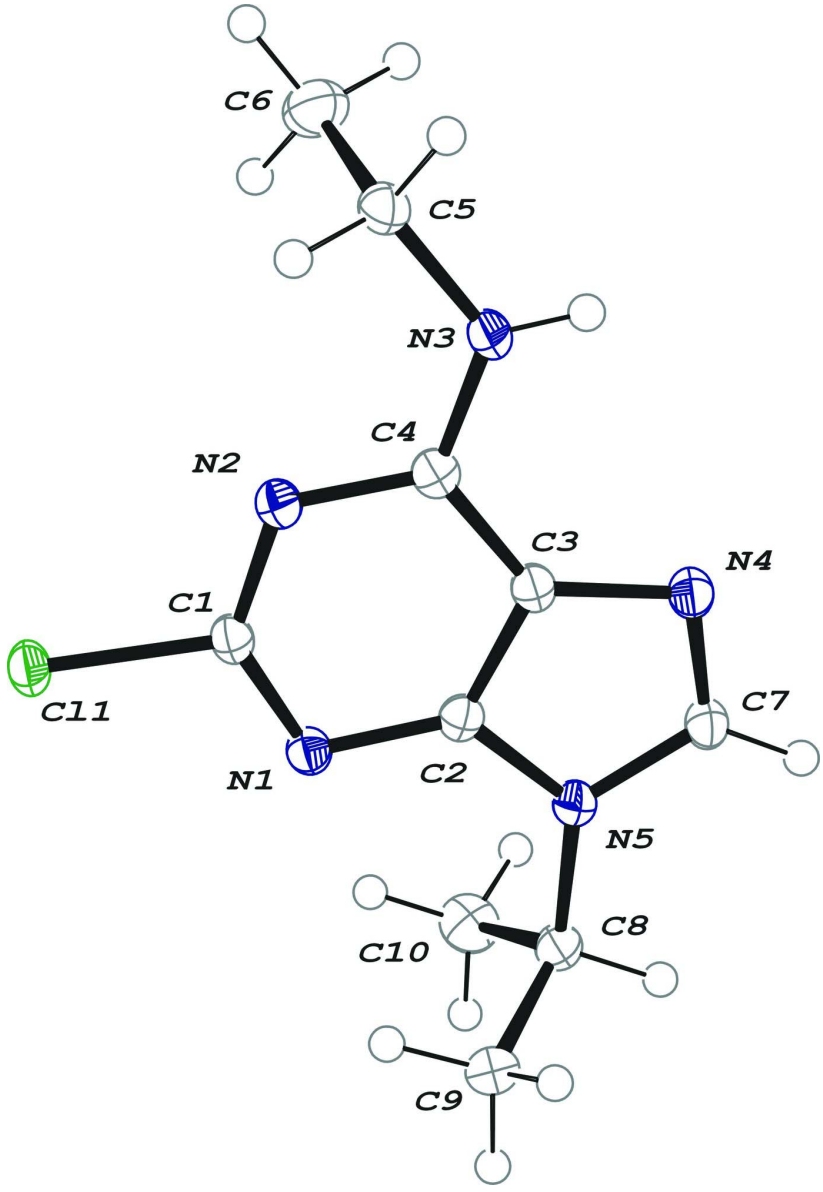

Figure 1

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.

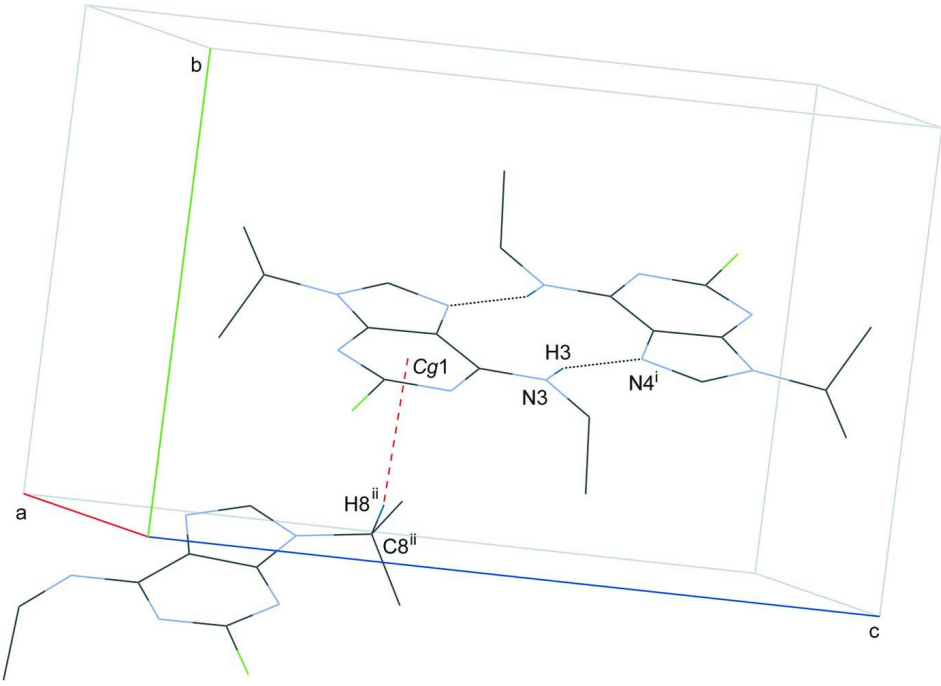

Figure 2

A view of the N—H···N (black dotted lines) and C—H···π (red dashed lines) interactions in the crystal structure of the title compound. H atoms non-participating in hydrogen-bonding were omitted for clarity. Symmetry codes: (i) -*x*, -*y* + 1, -*z* + 1; (ii) -*x* + 0.5, *y* - 0.5, -*z* + 0.5.

2-chloro-*N*-ethyl-9-isopropyl-9*H*-purine-6-amine

Crystal data

$C_{10}H_{14}ClN_5$
M_r = 239.71
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 8.1385 (2) Å
b = 9.6245 (2) Å
c = 14.8388 (3) Å
β = 92.997 (2)°
V = 1160.72 (4) Å^3
Z = 4
$F(000)$ = 504
D_x = 1.372 Mg m^{-3}
Melting point: 392 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9070 reflections
θ = 2.9–27.2°
μ = 0.31 mm^{-1}
T = 120 K
Block, colourless
0.40 × 0.40 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire2 (large Be window) detector
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 8.4353 pixels mm^{-1}
ω scan
Absorption correction: multi-scan *CrysAlis RED* (Oxford Diffraction, 2009)
T_{min} = 0.973, T_{max} = 1.000
13543 measured reflections
2041 independent reflections
1793 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.0°, θ_{min} = 2.9°
h = −9→9
k = −10→11
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.069$
$S = 1.07$
2041 reflections
152 parameters
0 restraints

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0372P)^2 + 0.3781P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Experimental. Spectral properties of title compound:1H NMR ($CDCl_3$): 1.30 (t, J = 7.3 Hz, 3H, $NHCH_2C\mathbf{H}_3$), 1.57 (d, J = 6.6 Hz, 6H, $CH(C\mathbf{H}_3)_2$), 3.72 (m, 2H, $NHC\mathbf{H}_2CH_3$), 4.80 (septet, J = 6.9 Hz, 1H, $C\mathbf{H}(CH_3)_2$), 5.84 (bs, 1H, $N\mathbf{H}CH_2CH_3$), 7.76 (s, 1H, NC8HN) p.p.m.. 13 C NMR ($CDCl_3$): 15.1(CH_3), 23.0(CH_3), 36.0(CH_2), 47.1(CH), 119.0(C), 137.6(CH), 154.5(C), 155.5(C) p.p.m.. IR (KBr): 3103(*m*), 2974(*m*), 2933(w), 1726(w), 1605(*s*), 1560(*m*), 1481(w), 1454(w), 1435(w), 1404(*m*), 1362(*m*), 1309(*s*), 1227(*s*), 1188(w), 130(w), 1034(*s*), 941(*m*), 874(*m*), 785(*m*), 657(*m*), 609(w) cm^{-1}. GC—EI—MS (200 °C, 70 eV): 241(M^+(^{37}Cl), 30), 240 (13), 239(M^+(^{35}Cl), 95), 226 (20), 225 (7), 224 (62), 213 (10), 211 (32), 199 (9), 198 (18), 197 (27), 196 (45), 184 (32), 183 (9), 182 (100), 171 (17), 169 (54), 161 (27), 160 (17), 156 (6), 155 (10), 154 (19), 153 (8), 146 (20), 134 (43), 133 (22), 120 (6), 119 (47), 118 (5), 108 (9), 107 (12), 106 (7), 93 (8), 92 (21), 80 (7), 68 (5), 67 (9), 66 (11), 65 (6), 55 (6), 54 (12), 53 (9), 44 (74), 43 (34), 42 (11), 41 (34), 40 (6) m/*z*(%). Elemental analysis calc for $C_{10}H_{14}ClN_4$ (239.70) C 50.11, H 5.89, N 29.22; found C 50.16, H 5.92, N 29.02.

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 $F^2 > 2\sigma(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.71286 (4)	0.25662 (4)	0.37828 (2)	0.02326 (12)
N1	0.46503 (13)	0.39081 (11)	0.30528 (7)	0.0176 (2)
N2	0.45822 (13)	0.33371 (11)	0.46370 (7)	0.0172 (2)
N3	0.24124 (14)	0.38987 (12)	0.55160 (7)	0.0195 (3)
H3	0.147 (2)	0.4230 (17)	0.5565 (11)	0.027 (4)*
N4	0.08494 (13)	0.52739 (11)	0.38017 (7)	0.0194 (3)
N5	0.21933 (13)	0.51796 (11)	0.25145 (7)	0.0180 (2)
C1	0.52134 (15)	0.33857 (13)	0.38306 (8)	0.0169 (3)
C2	0.31722 (15)	0.45181 (13)	0.31630 (8)	0.0164 (3)
C3	0.23370 (15)	0.45883 (13)	0.39493 (9)	0.0164 (3)
C4	0.30943 (15)	0.39438 (13)	0.47204 (8)	0.0166 (3)
C5	0.31812 (16)	0.31996 (15)	0.63006 (8)	0.0211 (3)
H5A	0.2794	0.3635	0.6855	0.025*
H5B	0.4388	0.3329	0.6301	0.025*
C6	0.27990 (19)	0.16560 (16)	0.63171 (10)	0.0312 (4)
H6A	0.3327	0.1238	0.6861	0.047*
H6B	0.3218	0.1212	0.5781	0.047*

H6C	0.1606	0.1521	0.6321	0.047*
C7	0.08323 (16)	0.56029 (14)	0.29428 (9)	0.0199 (3)
H7	−0.0051	0.6094	0.2644	0.024*
C8	0.25198 (17)	0.53989 (14)	0.15553 (9)	0.0210 (3)
H8	0.1594	0.5962	0.1277	0.025*
C9	0.40995 (17)	0.62252 (15)	0.14745 (9)	0.0250 (3)
H9A	0.4035	0.7089	0.1820	0.038*
H9B	0.4244	0.6445	0.0839	0.038*
H9C	0.5037	0.5674	0.1712	0.038*
C10	0.25535 (19)	0.40217 (16)	0.10584 (10)	0.0285 (3)
H10A	0.1506	0.3537	0.1119	0.043*
H10B	0.3455	0.3448	0.1317	0.043*
H10C	0.2722	0.4190	0.0418	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01873 (19)	0.0305 (2)	0.02086 (19)	0.00947 (13)	0.00355 (13)	0.00232 (13)
N1	0.0166 (6)	0.0174 (6)	0.0189 (6)	0.0010 (4)	0.0019 (4)	−0.0002 (4)
N2	0.0164 (5)	0.0172 (6)	0.0181 (5)	0.0006 (4)	0.0015 (4)	−0.0013 (4)
N3	0.0169 (6)	0.0237 (6)	0.0181 (6)	0.0048 (5)	0.0037 (4)	0.0010 (4)
N4	0.0170 (6)	0.0193 (6)	0.0220 (6)	0.0021 (4)	0.0018 (4)	0.0002 (5)
N5	0.0164 (6)	0.0199 (6)	0.0179 (5)	0.0021 (4)	0.0012 (4)	0.0019 (5)
C1	0.0148 (6)	0.0156 (7)	0.0203 (6)	0.0007 (5)	0.0019 (5)	−0.0015 (5)
C2	0.0158 (6)	0.0141 (7)	0.0191 (6)	−0.0012 (5)	−0.0004 (5)	−0.0003 (5)
C3	0.0151 (6)	0.0143 (7)	0.0200 (6)	−0.0007 (5)	0.0014 (5)	−0.0015 (5)
C4	0.0169 (6)	0.0136 (6)	0.0192 (6)	−0.0023 (5)	0.0010 (5)	−0.0027 (5)
C5	0.0202 (7)	0.0265 (8)	0.0166 (6)	0.0029 (6)	0.0020 (5)	0.0003 (5)
C6	0.0311 (8)	0.0295 (9)	0.0329 (8)	−0.0026 (6)	0.0016 (6)	0.0085 (6)
C7	0.0159 (6)	0.0200 (7)	0.0238 (7)	0.0025 (5)	0.0014 (5)	0.0015 (6)
C8	0.0202 (7)	0.0261 (8)	0.0169 (6)	0.0038 (5)	0.0008 (5)	0.0044 (5)
C9	0.0255 (7)	0.0241 (8)	0.0258 (7)	0.0015 (6)	0.0046 (6)	0.0065 (6)
C10	0.0300 (8)	0.0333 (9)	0.0220 (7)	−0.0012 (6)	−0.0001 (6)	−0.0046 (6)

Geometric parameters (Å, º)

Cl1—C1	1.7516 (13)	C5—H5A	0.9900
N1—C1	1.3189 (16)	C5—H5B	0.9900
N1—C2	1.3561 (16)	C6—H6A	0.9800
N2—C1	1.3274 (16)	C6—H6B	0.9800
N2—C4	1.3558 (16)	C6—H6C	0.9800
N3—C4	1.3312 (17)	C7—H7	0.9500
N3—C5	1.4572 (17)	C8—C10	1.518 (2)
N3—H3	0.837 (17)	C8—C9	1.5217 (19)
N4—C7	1.3126 (17)	C8—H8	1.0000
N4—C3	1.3861 (17)	C9—H9A	0.9800
N5—C7	1.3677 (17)	C9—H9B	0.9800
N5—C2	1.3733 (16)	C9—H9C	0.9800

N5—C8	1.4766 (16)	C10—H10A	0.9800
C2—C3	1.3824 (18)	C10—H10B	0.9800
C3—C4	1.4143 (18)	C10—H10C	0.9800
C5—C6	1.518 (2)		

C1—N1—C2	109.23 (11)	C5—C6—H6A	109.5
C1—N2—C4	117.17 (11)	C5—C6—H6B	109.5
C4—N3—C5	122.83 (12)	H6A—C6—H6B	109.5
C4—N3—H3	119.4 (11)	C5—C6—H6C	109.5
C5—N3—H3	117.6 (11)	H6A—C6—H6C	109.5
C7—N4—C3	103.48 (10)	H6B—C6—H6C	109.5
C7—N5—C2	105.47 (10)	N4—C7—N5	114.31 (12)
C7—N5—C8	126.60 (11)	N4—C7—H7	122.8
C2—N5—C8	127.93 (11)	N5—C7—H7	122.8
N1—C1—N2	132.00 (12)	N5—C8—C10	110.62 (11)
N1—C1—Cl1	113.92 (9)	N5—C8—C9	110.24 (11)
N2—C1—Cl1	114.06 (9)	C10—C8—C9	112.42 (12)
N1—C2—N5	127.02 (11)	N5—C8—H8	107.8
N1—C2—C3	126.96 (12)	C10—C8—H8	107.8
N5—C2—C3	106.02 (11)	C9—C8—H8	107.8
C2—C3—N4	110.72 (11)	C8—C9—H9A	109.5
C2—C3—C4	116.66 (12)	C8—C9—H9B	109.5
N4—C3—C4	132.59 (12)	H9A—C9—H9B	109.5
N3—C4—N2	118.86 (12)	C8—C9—H9C	109.5
N3—C4—C3	123.20 (12)	H9A—C9—H9C	109.5
N2—C4—C3	117.94 (11)	H9B—C9—H9C	109.5
N3—C5—C6	112.64 (11)	C8—C10—H10A	109.5
N3—C5—H5A	109.1	C8—C10—H10B	109.5
C6—C5—H5A	109.1	H10A—C10—H10B	109.5
N3—C5—H5B	109.1	C8—C10—H10C	109.5
C6—C5—H5B	109.1	H10A—C10—H10C	109.5
H5A—C5—H5B	107.8	H10B—C10—H10C	109.5

C2—N1—C1—N2	2.0 (2)	C5—N3—C4—N2	−1.24 (19)
C2—N1—C1—Cl1	−179.87 (9)	C5—N3—C4—C3	178.29 (12)
C4—N2—C1—N1	−1.4 (2)	C1—N2—C4—N3	179.07 (11)
C4—N2—C1—Cl1	−179.56 (9)	C1—N2—C4—C3	−0.48 (17)
C1—N1—C2—N5	179.90 (12)	C2—C3—C4—N3	−178.20 (12)
C1—N1—C2—C3	−0.80 (18)	N4—C3—C4—N3	−0.3 (2)
C7—N5—C2—N1	179.21 (12)	C2—C3—C4—N2	1.33 (17)
C8—N5—C2—N1	−0.8 (2)	N4—C3—C4—N2	179.24 (13)
C7—N5—C2—C3	−0.21 (14)	C4—N3—C5—C6	−85.04 (16)
C8—N5—C2—C3	179.74 (12)	C3—N4—C7—N5	0.27 (15)
N1—C2—C3—N4	−179.04 (12)	C2—N5—C7—N4	−0.05 (15)
N5—C2—C3—N4	0.38 (14)	C8—N5—C7—N4	−179.99 (12)
N1—C2—C3—C4	−0.68 (19)	C7—N5—C8—C10	−114.50 (14)
N5—C2—C3—C4	178.74 (11)	C2—N5—C8—C10	65.56 (16)
C7—N4—C3—C2	−0.40 (14)	C7—N5—C8—C9	120.53 (14)

C7—N4—C3—C4	−178.41 (14)	C2—N5—C8—C9	−59.40 (17)

Hydrogen-bond geometry (Å, º)

*Cg*1 is the center of gravity of the pyridine ring (C1, N1, C2, C3, C4, N2).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···N4^{i}	0.837 (16)	2.205 (17)	2.9979 (16)	158.2 (15)
C8—H8···*Cg*1^{ii}	1.00 (1)	2.90 (1)	3.6403 (13)	131 (1)

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