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2-Amino-4-(2-chlorophenyl)-6-(naphthalen-1-yl)pyridine-3-carbonitrile

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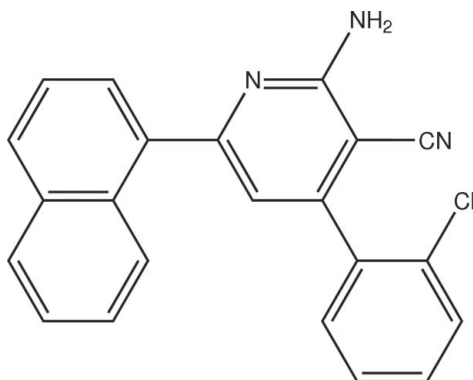
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.069; wR factor = 0.185; data-to-parameter ratio = 13.8.

In the title compound, $\text{C}_{22}\text{H}_{14}\text{ClN}_3$, prepared by a one-pot reaction under microwave irradiation, the dihedral angles between the central pyridine ring and the pendant naphthyl and chlorobenzene ring systems are $49.2(2)$ and $58.2(3)^\circ$, respectively. In the crystal, inversion dimers linked by pairs of $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds generate $R_2^2(8)$ loops. The pyridine N atom is the acceptor.

Related literature

For the use of 2-amino-3-cyanopyridines as intermediates in the preparation of heterocyclic compounds, see: Shishoo *et al.* (1983). For the synthesis, see: Mantri *et al.* (2008). For related structures, see: Mkhaliid *et al.* (2006).



Experimental

Crystal data

 $\text{C}_{22}\text{H}_{14}\text{ClN}_3$
 $M_r = 355.81$

 Monoclinic, $P2_1/n$
 $a = 12.275(3)$ Å
 $b = 4.6490(9)$ Å
 $c = 30.887(6)$ Å
 $\beta = 90.18(3)^\circ$
 $V = 1762.6(6)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.23$ mm⁻¹
 $T = 293$ K
 $0.20 \times 0.10 \times 0.10$ mm

Data collection

 Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.956$, $T_{\max} = 0.978$
 3397 measured reflections

 3236 independent reflections
 1558 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.057$
 3 standard reflections every 200 reflections
 intensity decay: 1%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.069$
 $wR(F^2) = 0.185$
 $S = 1.00$
 3236 reflections

 235 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2A}\cdots\text{N1}^i$	0.86	2.23	3.086 (5)	176

 Symmetry code: (i) $-x + 1, -y + 1, -z$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6750).

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supporting information

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2-Amino-4-(2-chlorophenyl)-6-(naphthalen-1-yl)pyridine-3-carbonitrile**Hong-Xia Wei, Jing Zhu, Ming Li, Jian-qiang Wang and Cheng Guo****S1. Comment**

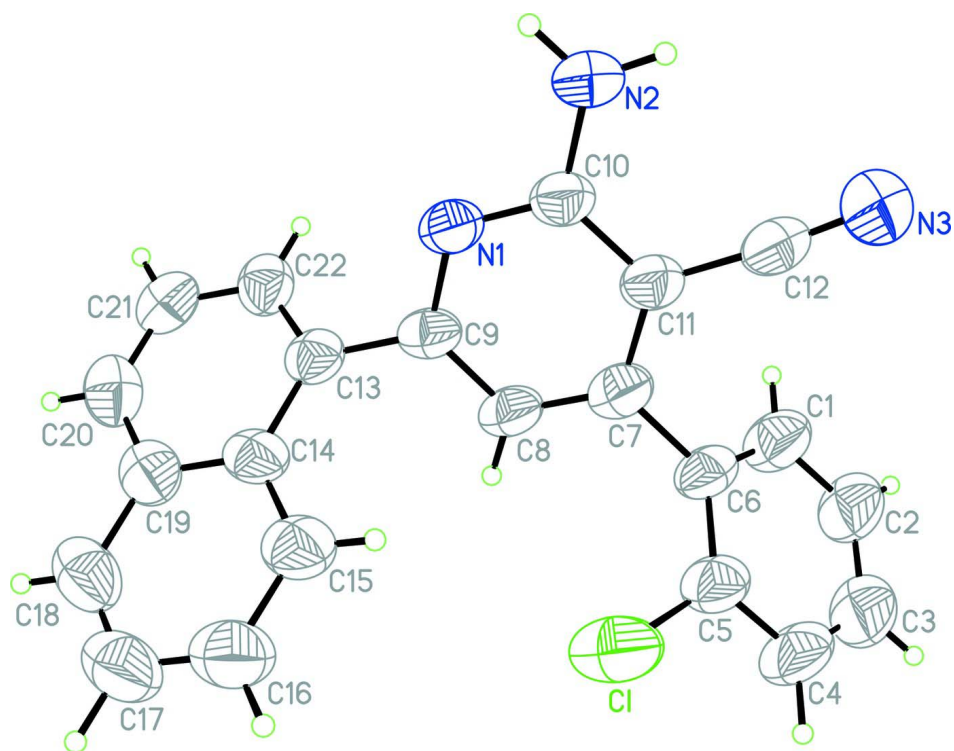
The title compound, C₂₂H₁₄ClN₃(1), is an intermediate in the synthesis of biologically active molecules (Shishoo *et al.*, 1983; Mantri *et al.* 2008). Herein we report its crystal structure. The molecular structure of (I) is shown in Fig. 1, and the selected geometric parameters are given in Table 1. In the molecules, the naphthyl (C13—C22) and phenyl ring planes (C1—C6) form torsion angles 49.2 and 58.2 °, respectively, with the middle pyridyl ring plane. In the crystal, there are N—H···N hydrogen bonds, which connect the independent molecules into dimers (Fig. 2 and Tab. 1).

S2. Experimental

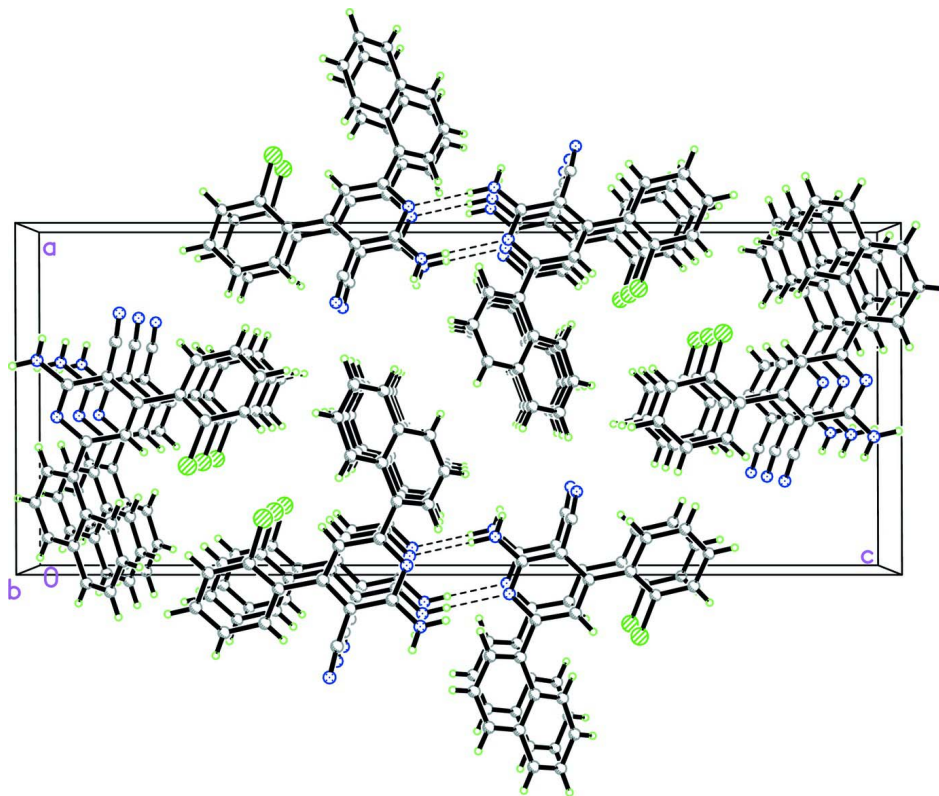
For the preparation of the title compound (1), a mixture of 2-chlorobenzaldehyde (2 mmol), malononitrile (2 mmol), 1-naphthaldehyde (2 mmol) and ammonium acetate (16 mmol) was refluxed under microwave irradiation (6 min, WF-4000M microwave reaction system). After cooling to room temperature, the resulting solid product was filtered off and recrystallized from methanol to give the title compound. Colourless needles were obtained by dissolving the title compound (0.5 g) in methanol (20 ml) and slowly evaporating the solvent at room temperature for a period of about two weeks.

S3. Refinement

All H atoms were positioned geometrically, with C—H = 0.86 Å and N—H = 0.93 Å for aromatic and amino H, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

**Figure 1**

The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

**Figure 2**

A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

2-Amino-4-(2-chlorophenyl)-6-(naphthalen-1-yl)pyridine-3-carbonitrile

Crystal data

$C_{22}H_{14}ClN_3$

$M_r = 355.81$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 12.275\ (3)\ \text{\AA}$

$b = 4.6490\ (9)\ \text{\AA}$

$c = 30.887\ (6)\ \text{\AA}$

$\beta = 90.18\ (3)^\circ$

$V = 1762.6\ (6)\ \text{\AA}^3$

$Z = 4$

$F(000) = 736$

$D_x = 1.341\ \text{Mg m}^{-3}$

Melting point: 421 K

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 25 reflections

$\theta = 9\text{--}12^\circ$

$\mu = 0.23\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Needle, colourless

$0.20 \times 0.10 \times 0.10\ \text{mm}$

Data collection

Enraf–Nonius CAD-4

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega/2\theta$ scans

Absorption correction: ψ scan

(North *et al.*, 1968)

$T_{\min} = 0.956$, $T_{\max} = 0.978$

3397 measured reflections

3236 independent reflections

1558 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.057$

$\theta_{\max} = 25.5^\circ$, $\theta_{\min} = 1.3^\circ$

$h = -14 \rightarrow 0$

$k = 0 \rightarrow 5$

$l = -37 \rightarrow 37$

3 standard reflections every 200 reflections

intensity decay: 1%

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.069$
 $wR(F^2) = 0.185$
 $S = 1.00$
 3236 reflections
 235 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
 $w = 1/[\sigma^2(F_o^2) + (0.050P)^2 + 1.9P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl	0.31710 (12)	0.7045 (4)	0.20602 (4)	0.0872 (5)
N1	0.4498 (3)	0.3780 (9)	0.05627 (11)	0.0550 (10)
C1	0.6080 (4)	0.3185 (13)	0.20923 (15)	0.0773 (16)
H1B	0.6571	0.2335	0.1903	0.093*
N2	0.6054 (3)	0.6314 (10)	0.04127 (11)	0.0763 (14)
H2A	0.5897	0.6189	0.0142	0.092*
H2B	0.6635	0.7198	0.0494	0.092*
C2	0.6330 (5)	0.3305 (14)	0.25299 (16)	0.0863 (18)
H2C	0.6981	0.2532	0.2632	0.104*
N3	0.7314 (4)	0.8313 (12)	0.13555 (14)	0.0968 (17)
C3	0.5613 (5)	0.4568 (15)	0.28131 (17)	0.090 (2)
H3A	0.5779	0.4680	0.3107	0.109*
C4	0.4654 (5)	0.5656 (13)	0.26593 (15)	0.0814 (17)
H4A	0.4162	0.6475	0.2851	0.098*
C5	0.4402 (4)	0.5563 (11)	0.22255 (14)	0.0634 (13)
C6	0.5115 (4)	0.4305 (11)	0.19312 (13)	0.0578 (13)
C7	0.4887 (4)	0.4158 (11)	0.14580 (14)	0.0579 (13)
C8	0.3990 (4)	0.2782 (11)	0.12958 (13)	0.0597 (13)
H8A	0.3499	0.1926	0.1485	0.072*
C9	0.3800 (3)	0.2645 (10)	0.08536 (13)	0.0507 (11)
C10	0.5383 (4)	0.5115 (11)	0.07123 (14)	0.0542 (12)
C11	0.5606 (3)	0.5421 (11)	0.11589 (14)	0.0568 (12)
C12	0.6549 (4)	0.7022 (13)	0.12856 (14)	0.0673 (15)
C13	0.2812 (4)	0.1290 (10)	0.06732 (14)	0.0533 (12)
C14	0.1748 (4)	0.1886 (11)	0.08349 (14)	0.0577 (13)

C15	0.1539 (4)	0.3973 (12)	0.11570 (15)	0.0682 (14)
H15A	0.2113	0.5015	0.1276	0.082*
C16	0.0499 (5)	0.4471 (14)	0.12953 (18)	0.0842 (17)
H16A	0.0379	0.5848	0.1508	0.101*
C17	-0.0383 (5)	0.2979 (16)	0.11261 (19)	0.090 (2)
H17A	-0.1083	0.3344	0.1226	0.108*
C18	-0.0217 (4)	0.0997 (14)	0.08151 (19)	0.0807 (17)
H18A	-0.0808	-0.0007	0.0702	0.097*
C19	0.0835 (4)	0.0418 (12)	0.06576 (16)	0.0629 (13)
C20	0.1015 (4)	-0.1567 (12)	0.03181 (17)	0.0735 (15)
H20A	0.0425	-0.2559	0.0202	0.088*
C21	0.2025 (4)	-0.2061 (11)	0.01577 (15)	0.0679 (14)
H21A	0.2118	-0.3334	-0.0071	0.082*
C22	0.2927 (4)	-0.0649 (11)	0.03379 (15)	0.0613 (13)
H22A	0.3618	-0.1027	0.0229	0.074*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0919 (10)	0.1025 (12)	0.0674 (9)	0.0187 (9)	0.0206 (7)	-0.0003 (8)
N1	0.057 (2)	0.066 (3)	0.042 (2)	0.005 (2)	0.0057 (17)	0.003 (2)
C1	0.086 (4)	0.095 (4)	0.051 (3)	0.012 (3)	-0.004 (3)	-0.004 (3)
N2	0.065 (3)	0.118 (4)	0.046 (2)	-0.017 (3)	0.0128 (19)	-0.002 (3)
C2	0.100 (4)	0.106 (5)	0.052 (3)	-0.009 (4)	-0.012 (3)	0.006 (4)
N3	0.075 (3)	0.137 (5)	0.079 (3)	-0.029 (3)	0.009 (3)	-0.024 (3)
C3	0.100 (5)	0.123 (6)	0.049 (3)	-0.024 (4)	-0.007 (3)	0.008 (4)
C4	0.105 (4)	0.100 (5)	0.039 (3)	-0.011 (4)	0.013 (3)	-0.007 (3)
C5	0.073 (3)	0.072 (4)	0.045 (3)	-0.006 (3)	0.011 (2)	0.002 (3)
C6	0.073 (3)	0.064 (3)	0.036 (2)	-0.002 (3)	0.006 (2)	0.001 (2)
C7	0.064 (3)	0.066 (3)	0.043 (3)	0.009 (3)	0.003 (2)	0.000 (2)
C8	0.069 (3)	0.072 (3)	0.038 (2)	-0.004 (3)	0.009 (2)	0.002 (3)
C9	0.057 (3)	0.053 (3)	0.042 (2)	0.006 (2)	0.009 (2)	0.008 (2)
C10	0.050 (3)	0.069 (3)	0.044 (3)	0.006 (3)	0.007 (2)	0.000 (2)
C11	0.051 (3)	0.074 (3)	0.046 (3)	0.006 (3)	0.006 (2)	-0.008 (3)
C12	0.066 (3)	0.094 (4)	0.041 (3)	-0.001 (3)	0.007 (2)	-0.012 (3)
C13	0.060 (3)	0.057 (3)	0.042 (2)	0.004 (3)	0.000 (2)	0.011 (2)
C14	0.057 (3)	0.066 (3)	0.050 (3)	0.005 (3)	0.011 (2)	0.021 (3)
C15	0.069 (3)	0.080 (4)	0.056 (3)	0.003 (3)	0.010 (2)	0.015 (3)
C16	0.083 (4)	0.100 (5)	0.069 (4)	0.020 (4)	0.016 (3)	0.009 (3)
C17	0.067 (4)	0.121 (6)	0.083 (4)	0.021 (4)	0.019 (3)	0.031 (4)
C18	0.058 (3)	0.092 (5)	0.092 (4)	0.000 (3)	0.004 (3)	0.027 (4)
C19	0.062 (3)	0.062 (3)	0.064 (3)	-0.001 (3)	-0.001 (2)	0.018 (3)
C20	0.070 (3)	0.071 (4)	0.080 (4)	-0.001 (3)	-0.014 (3)	0.007 (3)
C21	0.090 (4)	0.063 (3)	0.051 (3)	0.007 (3)	-0.004 (3)	-0.003 (3)
C22	0.063 (3)	0.063 (3)	0.058 (3)	0.001 (3)	-0.007 (2)	0.007 (3)

Geometric parameters (Å, °)

C1—C5	1.736 (5)	C9—C13	1.475 (6)
N1—C10	1.332 (5)	C10—C11	1.413 (6)
N1—C9	1.351 (5)	C11—C12	1.429 (7)
C1—C6	1.385 (6)	C13—C22	1.380 (6)
C1—C2	1.386 (6)	C13—C14	1.427 (6)
C1—H1B	0.9300	C14—C15	1.414 (7)
N2—C10	1.360 (5)	C14—C19	1.421 (6)
N2—H2A	0.8600	C15—C16	1.367 (6)
N2—H2B	0.8600	C15—H15A	0.9300
C2—C3	1.375 (7)	C16—C17	1.386 (8)
C2—H2C	0.9300	C16—H16A	0.9300
N3—C12	1.134 (6)	C17—C18	1.347 (8)
C3—C4	1.365 (7)	C17—H17A	0.9300
C3—H3A	0.9300	C18—C19	1.407 (6)
C4—C5	1.375 (6)	C18—H18A	0.9300
C4—H4A	0.9300	C19—C20	1.414 (7)
C5—C6	1.392 (6)	C20—C21	1.356 (6)
C6—C7	1.489 (6)	C20—H20A	0.9300
C7—C8	1.367 (6)	C21—C22	1.401 (6)
C7—C11	1.408 (6)	C21—H21A	0.9300
C8—C9	1.386 (5)	C22—H22A	0.9300
C8—H8A	0.9300		
C10—N1—C9	118.0 (4)	C7—C11—C10	118.6 (4)
C6—C1—C2	121.4 (5)	C7—C11—C12	123.1 (4)
C6—C1—H1B	119.3	C10—C11—C12	118.3 (4)
C2—C1—H1B	119.3	N3—C12—C11	175.1 (5)
C10—N2—H2A	120.0	C22—C13—C14	119.0 (4)
C10—N2—H2B	120.0	C22—C13—C9	118.5 (4)
H2A—N2—H2B	120.0	C14—C13—C9	122.5 (4)
C3—C2—C1	119.8 (5)	C15—C14—C19	117.1 (4)
C3—C2—H2C	120.1	C15—C14—C13	123.2 (5)
C1—C2—H2C	120.1	C19—C14—C13	119.6 (5)
C4—C3—C2	119.3 (5)	C16—C15—C14	120.5 (5)
C4—C3—H3A	120.3	C16—C15—H15A	119.7
C2—C3—H3A	120.3	C14—C15—H15A	119.7
C3—C4—C5	121.3 (5)	C15—C16—C17	121.7 (6)
C3—C4—H4A	119.4	C15—C16—H16A	119.1
C5—C4—H4A	119.4	C17—C16—H16A	119.1
C4—C5—C6	120.6 (5)	C18—C17—C16	119.4 (5)
C4—C5—C1	117.8 (4)	C18—C17—H17A	120.3
C6—C5—C1	121.5 (4)	C16—C17—H17A	120.3
C1—C6—C5	117.5 (4)	C17—C18—C19	121.2 (6)
C1—C6—C7	119.5 (4)	C17—C18—H18A	119.4
C5—C6—C7	122.9 (4)	C19—C18—H18A	119.4
C8—C7—C11	117.4 (4)	C18—C19—C20	121.8 (5)

C8—C7—C6	122.0 (4)	C18—C19—C14	119.9 (5)
C11—C7—C6	120.6 (4)	C20—C19—C14	118.3 (5)
C7—C8—C9	121.0 (4)	C21—C20—C19	121.8 (5)
C7—C8—H8A	119.5	C21—C20—H20A	119.1
C9—C8—H8A	119.5	C19—C20—H20A	119.1
N1—C9—C8	122.2 (4)	C20—C21—C22	119.9 (5)
N1—C9—C13	116.0 (4)	C20—C21—H21A	120.1
C8—C9—C13	121.8 (4)	C22—C21—H21A	120.1
N1—C10—N2	116.7 (4)	C13—C22—C21	121.4 (5)
N1—C10—C11	122.8 (4)	C13—C22—H22A	119.3
N2—C10—C11	120.5 (4)	C21—C22—H22A	119.3
C6—C1—C2—C3	-0.3 (9)	N1—C10—C11—C12	-176.8 (5)
C1—C2—C3—C4	0.9 (9)	N2—C10—C11—C12	0.1 (7)
C2—C3—C4—C5	-1.3 (10)	C7—C11—C12—N3	178 (7)
C3—C4—C5—C6	1.1 (9)	C10—C11—C12—N3	-3 (7)
C3—C4—C5—C1	-179.2 (5)	N1—C9—C13—C22	48.9 (6)
C2—C1—C6—C5	0.1 (8)	C8—C9—C13—C22	-132.1 (5)
C2—C1—C6—C7	179.3 (5)	N1—C9—C13—C14	-131.7 (4)
C4—C5—C6—C1	-0.5 (8)	C8—C9—C13—C14	47.3 (6)
C1—C5—C6—C1	179.8 (4)	C22—C13—C14—C15	-175.7 (4)
C4—C5—C6—C7	-179.6 (5)	C9—C13—C14—C15	5.0 (7)
C1—C5—C6—C7	0.7 (7)	C22—C13—C14—C19	1.8 (6)
C1—C6—C7—C8	121.2 (6)	C9—C13—C14—C19	-177.5 (4)
C5—C6—C7—C8	-59.7 (7)	C19—C14—C15—C16	1.6 (7)
C1—C6—C7—C11	-58.1 (7)	C13—C14—C15—C16	179.2 (4)
C5—C6—C7—C11	121.1 (5)	C14—C15—C16—C17	-0.3 (8)
C11—C7—C8—C9	0.0 (7)	C15—C16—C17—C18	-0.5 (9)
C6—C7—C8—C9	-179.3 (4)	C16—C17—C18—C19	-0.2 (9)
C10—N1—C9—C8	-1.3 (7)	C17—C18—C19—C20	-177.2 (5)
C10—N1—C9—C13	177.7 (4)	C17—C18—C19—C14	1.6 (8)
C7—C8—C9—N1	1.8 (7)	C15—C14—C19—C18	-2.2 (7)
C7—C8—C9—C13	-177.1 (5)	C13—C14—C19—C18	-179.9 (4)
C9—N1—C10—N2	-178.0 (4)	C15—C14—C19—C20	176.6 (4)
C9—N1—C10—C11	-1.0 (7)	C13—C14—C19—C20	-1.1 (7)
C8—C7—C11—C10	-2.1 (7)	C18—C19—C20—C21	178.0 (5)
C6—C7—C11—C10	177.1 (4)	C14—C19—C20—C21	-0.8 (7)
C8—C7—C11—C12	177.4 (5)	C19—C20—C21—C22	1.9 (8)
C6—C7—C11—C12	-3.4 (7)	C14—C13—C22—C21	-0.7 (7)
N1—C10—C11—C7	2.7 (7)	C9—C13—C22—C21	178.6 (4)
N2—C10—C11—C7	179.6 (4)	C20—C21—C22—C13	-1.1 (7)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2A \cdots N1 ⁱ	0.86	2.23	3.086 (5)	176

Symmetry code: (i) $-x+1, -y+1, -z$.