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3-(1*H*-Imidazo[4,5-*f*][1,10]-phenanthrolin-2-yl)benzonitrile methanol solvate

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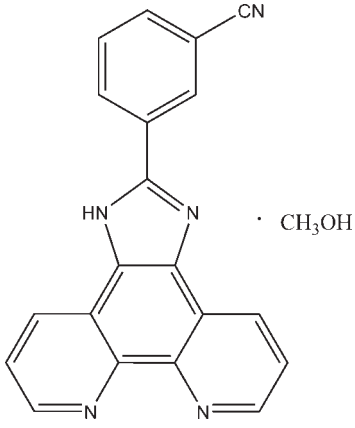
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.072; wR factor = 0.179; data-to-parameter ratio = 14.0.

In the title compound, $C_{20}H_{11}N_5\cdot CH_3OH$, the benzene ring is twisted by a small dihedral angle of 1.89 (11)° with respect to the imidazo[4,5-*f*][1,10]phenanthroline ring system. N—H···O and O—H···N hydrogen bonding is present in the crystal structure.

Related literature

For related structures, see: Sun *et al.* (2007); Yin (2008); Zhang *et al.* (2008).



Experimental

Crystal data

$C_{20}H_{11}N_5\cdot CH_4O$
M_r = 353.38
Monoclinic, $P2_1/n$
a = 7.115 (1) Å
b = 18.385 (2) Å
c = 13.5576 (12) Å
β = 99.956 (19)°
V = 1746.7 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.30 × 0.28 × 0.26 mm

Data collection

Rigaku, SCXmini diffractometer
15971 measured reflections
3432 independent reflections
2018 reflections with $I > 2\sigma(I)$
R_{int} = 0.099

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.179
S = 1.04
3432 reflections
245 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···O1	0.86	1.95	2.803 (3)	174
O1—H1E···N5^{i}	0.98	1.89	2.857 (3)	168

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

This work was supported by a start-up grant from Beihua University, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2700).

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supporting information

Acta Cryst. (2010). E**66**, o55 [doi:10.1107/S1600536809051472]

3-(1*H*-Imidazo[4,5-*f*][1,10]phenanthrolin-2-yl)benzonitrile methanol solvate

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S1. Comment

1,10-Phenanthroline and its derivatives are commonly used as ligands in metal-organic coordination polymers (Sun *et al.*, 2007; Yin, 2008; Zhang *et al.*, 2008). The title compound was synthesized from 1,10-phenanthroline-5,6-dione.

The asymmetric unit of the title compound, $C_{20}H_{11}N_5.CH_3OH$, contains one organic molecule and one solvent methanol molecule (Fig.1). The molecules are connected by N—H···O and O—H···N hydrogen bonding to form a one-dimensional chain (Fig. 2). The organic molecule is essentially planar.

S2. Experimental

1,10-Phenanthroline-5,6-dione (1.5 mmol) and 3-cyanobenzaldehyde (1.5 mmol) were dissolved in CH_3COOH and CH_3COONH_4 (1:1) solution (30 ml). The mixture was refluxed for 1.5 h under argon, after cooling this mixture was diluted with water and neutralized with concentrated aqueous ammonia, immediately resulting a yellow precipitate, which was washed with water, acetone and diethyl ether respectively. Crystals of the title compound were obtained by recrystallization from dichloromethane-methanol solution.

S3. Refinement

Methanol H atom was located in a difference Fourier map and refined as riding in as-found relative position, the thermal parameter was refined. Other H atoms were placed in calculated positions with C—H = 0.93 Å (aromatic), 0.96 Å (methyl) and N—H = 0.86 Å, and refined using a riding model, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H and 1.2U_{eq}(C,N) for the others.

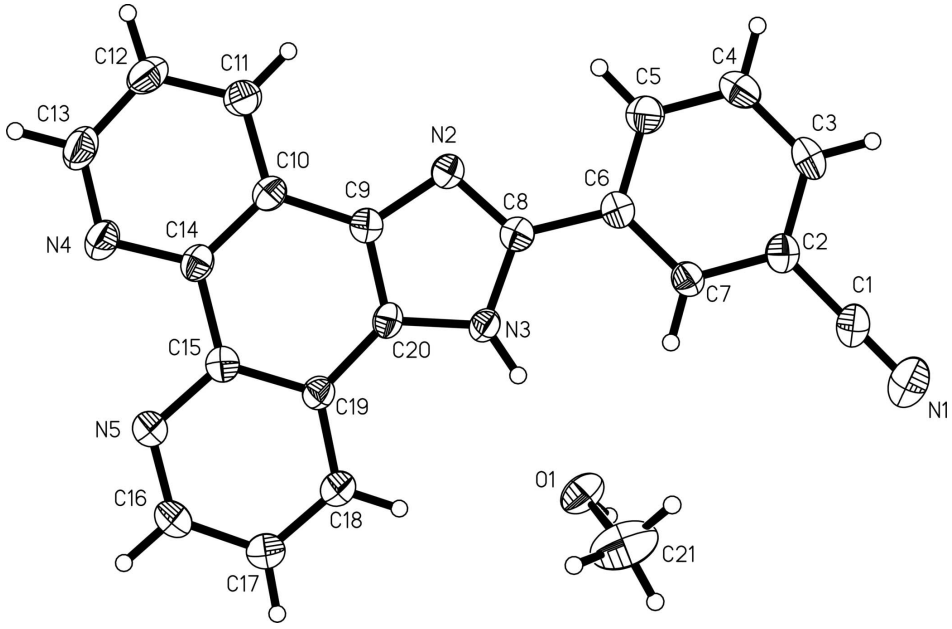

Figure 1

The asymmetric unit of the title compound with atom labels. Displacement ellipsoids were drawn at the 30% probability level.

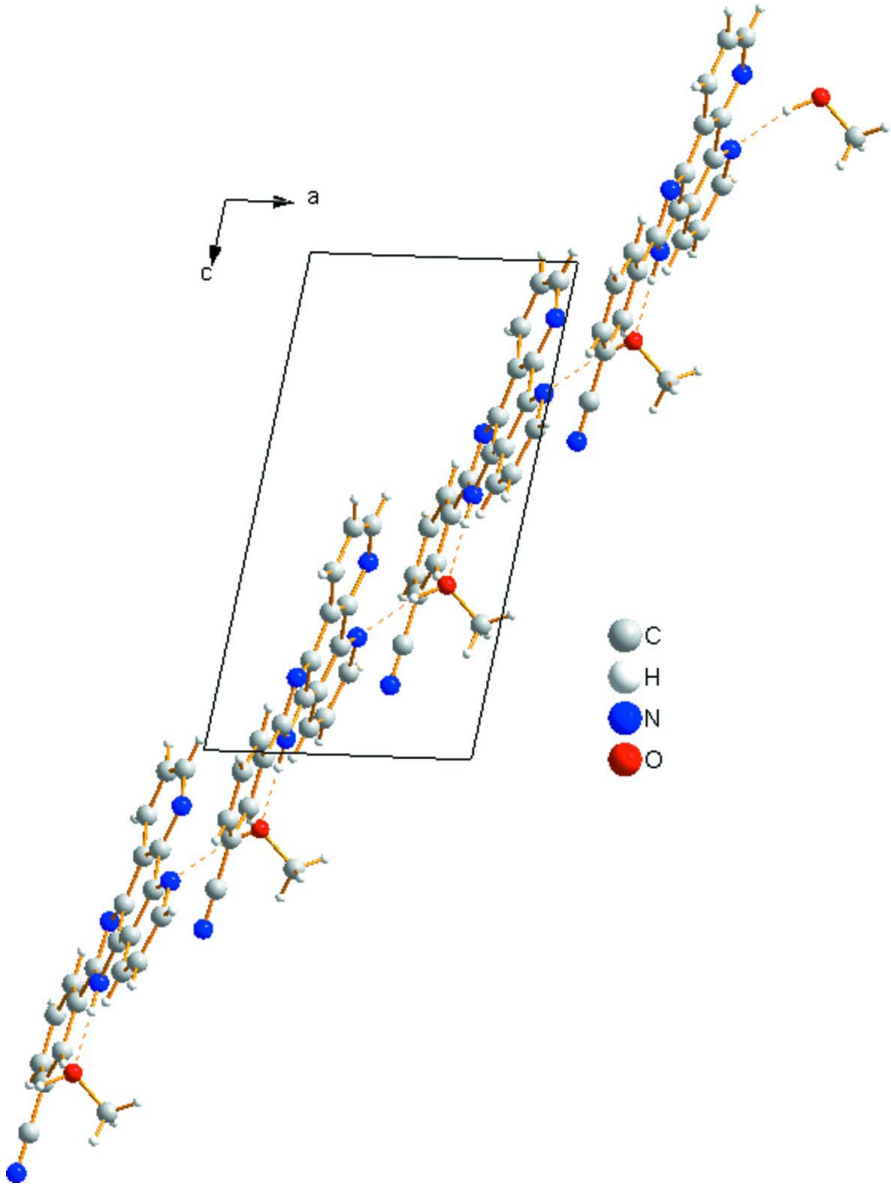

Figure 2

The packing viewed along the *b* axis. Hydrogen bonds are drawn as dashed lines

3-(1*H*-Imidazo[4,5-*f*][1,10]phenanthrolin-2-yl)benzonitrile methanol solvate

Crystal data

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$M_r = 353.38$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.115\ (1)$ Å
$b = 18.385\ (2)$ Å
$c = 13.5576\ (12)$ Å
$\beta = 99.956\ (19)°$
$V = 1746.7\ (4)$ Å^3
$Z = 4$
$F(000) = 736$
$D_x = 1.344$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2018 reflections
$\theta = 3.0–26.0°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, colorless
0.30 × 0.28 × 0.26 mm

Data collection

Rigaku, SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scan
15971 measured reflections
3432 independent reflections
2018 reflections with $I > 2\sigma(I)$
$R_{int} = 0.099$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.0°$
$h = -8 \rightarrow 8$
$k = -22 \rightarrow 22$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.072$
$wR(F^2) = 0.179$
$S = 1.04$
3432 reflections
245 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
$w = 1/[\sigma^2(F_o^2) + (0.0784P)^2 + 0.0647P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.6541 (5)	−0.05805 (18)	0.7761 (3)	0.0518 (8)
C2	0.6702 (4)	−0.07430 (16)	0.6737 (2)	0.0430 (7)
C3	0.6441 (4)	−0.14493 (17)	0.6377 (3)	0.0510 (8)
H3A	0.6173	−0.1822	0.6795	0.061*
C4	0.6581 (5)	−0.15944 (17)	0.5396 (3)	0.0538 (9)
H4A	0.6398	−0.2066	0.5152	0.065*
C5	0.6993 (4)	−0.10430 (17)	0.4769 (2)	0.0501 (8)
H5A	0.7079	−0.1149	0.4107	0.060*
C6	0.7281 (4)	−0.03325 (15)	0.5117 (2)	0.0386 (7)
C7	0.7123 (4)	−0.01864 (16)	0.6113 (2)	0.0407 (7)
H7A	0.7300	0.0285	0.6360	0.049*
C8	0.7695 (4)	0.02402 (15)	0.4435 (2)	0.0391 (7)
C9	0.8262 (4)	0.08115 (15)	0.3144 (2)	0.0388 (7)
C10	0.8600 (4)	0.10077 (16)	0.2162 (2)	0.0391 (7)
C11	0.8394 (4)	0.05275 (17)	0.1346 (2)	0.0490 (8)
H11A	0.7998	0.0051	0.1413	0.059*
C12	0.8787 (5)	0.07721 (19)	0.0448 (2)	0.0567 (9)

H12A	0.8629	0.0470	−0.0110	0.068*
C13	0.9430 (5)	0.14836 (19)	0.0393 (2)	0.0596 (10)
H13A	0.9758	0.1633	−0.0210	0.072*
C14	0.9157 (4)	0.17313 (16)	0.2013 (2)	0.0411 (7)
C15	0.9232 (4)	0.22708 (16)	0.2820 (2)	0.0407 (7)
C16	0.9749 (5)	0.34573 (18)	0.3349 (3)	0.0622 (10)
H16A	1.0103	0.3930	0.3221	0.075*
C17	0.9213 (5)	0.33164 (18)	0.4271 (2)	0.0592 (9)
H17A	0.9197	0.3687	0.4737	0.071*
C18	0.8714 (5)	0.26248 (17)	0.4477 (2)	0.0511 (8)
H18A	0.8365	0.2515	0.5090	0.061*
C19	0.8732 (4)	0.20771 (15)	0.3752 (2)	0.0388 (7)
C20	0.8320 (4)	0.13278 (15)	0.3885 (2)	0.0371 (7)
C21	0.9291 (6)	0.1623 (3)	0.7300 (3)	0.1045 (16)
H21A	0.9003	0.1877	0.7875	0.157*
H21B	1.0353	0.1853	0.7075	0.157*
H21C	0.9610	0.1127	0.7478	0.157*
N1	0.6392 (5)	−0.04524 (19)	0.8570 (2)	0.0749 (9)
N2	0.7870 (3)	0.01301 (12)	0.34899 (16)	0.0406 (6)
N3	0.7950 (3)	0.09555 (12)	0.47126 (17)	0.0390 (6)
H3B	0.7891	0.1137	0.5291	0.047*
N4	0.9610 (4)	0.19635 (14)	0.11301 (19)	0.0550 (7)
N5	0.9787 (4)	0.29620 (13)	0.26399 (19)	0.0513 (7)
O1	0.7700 (4)	0.16399 (13)	0.65327 (16)	0.0640 (7)
H1E	0.6578	0.1781	0.6823	0.14 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.050 (2)	0.061 (2)	0.044 (2)	−0.0024 (16)	0.0072 (16)	0.0113 (16)
C2	0.0349 (16)	0.050 (2)	0.0432 (18)	0.0017 (13)	0.0034 (14)	0.0069 (14)
C3	0.051 (2)	0.0400 (19)	0.063 (2)	−0.0021 (14)	0.0118 (17)	0.0147 (16)
C4	0.055 (2)	0.0383 (19)	0.070 (2)	−0.0027 (14)	0.0155 (18)	−0.0017 (17)
C5	0.0488 (19)	0.050 (2)	0.052 (2)	−0.0018 (15)	0.0117 (16)	−0.0018 (16)
C6	0.0300 (16)	0.0410 (17)	0.0446 (17)	0.0024 (12)	0.0063 (13)	0.0044 (13)
C7	0.0409 (17)	0.0381 (17)	0.0426 (18)	−0.0026 (13)	0.0060 (14)	0.0031 (13)
C8	0.0345 (16)	0.0416 (17)	0.0414 (17)	0.0021 (12)	0.0074 (13)	0.0001 (13)
C9	0.0359 (16)	0.0419 (18)	0.0382 (16)	−0.0003 (12)	0.0051 (13)	0.0016 (13)
C10	0.0351 (16)	0.0460 (18)	0.0361 (16)	0.0024 (13)	0.0062 (13)	−0.0013 (13)
C11	0.053 (2)	0.0483 (19)	0.0457 (19)	0.0018 (15)	0.0095 (16)	−0.0034 (15)
C12	0.070 (2)	0.062 (2)	0.0386 (19)	0.0111 (17)	0.0105 (17)	−0.0064 (16)
C13	0.078 (3)	0.066 (2)	0.0375 (19)	0.0088 (19)	0.0183 (17)	0.0087 (17)
C14	0.0407 (17)	0.0470 (19)	0.0357 (16)	0.0054 (13)	0.0071 (13)	0.0038 (13)
C15	0.0378 (17)	0.0422 (18)	0.0422 (17)	0.0020 (13)	0.0077 (14)	0.0027 (13)
C16	0.087 (3)	0.0392 (19)	0.063 (2)	−0.0115 (17)	0.021 (2)	−0.0003 (17)
C17	0.079 (3)	0.049 (2)	0.052 (2)	−0.0060 (17)	0.0183 (18)	−0.0075 (16)
C18	0.064 (2)	0.049 (2)	0.0429 (18)	−0.0016 (16)	0.0161 (16)	−0.0037 (15)
C19	0.0389 (17)	0.0409 (18)	0.0377 (16)	0.0005 (12)	0.0099 (13)	−0.0005 (13)

C20	0.0347 (16)	0.0436 (18)	0.0345 (16)	0.0030 (12)	0.0103 (13)	0.0041 (13)
C21	0.082 (3)	0.134 (4)	0.092 (3)	0.011 (3)	−0.002 (3)	−0.042 (3)
N1	0.071 (2)	0.100 (3)	0.053 (2)	−0.0063 (18)	0.0090 (17)	0.0078 (18)
N2	0.0423 (15)	0.0424 (15)	0.0386 (14)	−0.0004 (11)	0.0114 (11)	0.0001 (11)
N3	0.0434 (14)	0.0430 (15)	0.0322 (13)	0.0006 (11)	0.0110 (11)	−0.0003 (11)
N4	0.073 (2)	0.0573 (18)	0.0366 (15)	0.0024 (14)	0.0161 (14)	0.0035 (13)
N5	0.0651 (18)	0.0425 (16)	0.0489 (16)	−0.0064 (13)	0.0171 (13)	−0.0016 (12)
O1	0.0695 (17)	0.0778 (17)	0.0474 (14)	0.0083 (13)	0.0177 (13)	−0.0119 (12)

Geometric parameters (Å, º)

C1—N1	1.145 (4)	C12—H12A	0.9300
C1—C2	1.444 (4)	C13—N4	1.323 (4)
C2—C3	1.388 (4)	C13—H13A	0.9300
C2—C7	1.393 (4)	C14—N4	1.361 (4)
C3—C4	1.376 (4)	C14—C15	1.471 (4)
C3—H3A	0.9300	C15—N5	1.365 (4)
C4—C5	1.387 (4)	C15—C19	1.416 (4)
C4—H4A	0.9300	C16—N5	1.328 (4)
C5—C6	1.392 (4)	C16—C17	1.393 (4)
C5—H5A	0.9300	C16—H16A	0.9300
C6—C7	1.400 (4)	C17—C18	1.362 (4)
C6—C8	1.465 (4)	C17—H17A	0.9300
C7—H7A	0.9300	C18—C19	1.409 (4)
C8—N2	1.324 (3)	C18—H18A	0.9300
C8—N3	1.371 (3)	C19—C20	1.426 (4)
C9—C20	1.378 (4)	C20—N3	1.377 (3)
C9—N2	1.383 (3)	C21—O1	1.400 (4)
C9—C10	1.439 (4)	C21—H21A	0.9600
C10—C11	1.403 (4)	C21—H21B	0.9600
C10—C14	1.412 (4)	C21—H21C	0.9600
C11—C12	1.371 (4)	N3—H3B	0.8600
C11—H11A	0.9300	O1—H1E	0.9842
C12—C13	1.392 (5)		

N1—C1—C2	179.2 (4)	C12—C13—H13A	117.4
C3—C2—C7	120.2 (3)	N4—C14—C10	122.4 (3)
C3—C2—C1	120.2 (3)	N4—C14—C15	117.3 (3)
C7—C2—C1	119.5 (3)	C10—C14—C15	120.3 (3)
C4—C3—C2	119.6 (3)	N5—C15—C19	121.3 (3)
C4—C3—H3A	120.2	N5—C15—C14	118.0 (3)
C2—C3—H3A	120.2	C19—C15—C14	120.8 (3)
C3—C4—C5	120.6 (3)	N5—C16—C17	124.4 (3)
C3—C4—H4A	119.7	N5—C16—H16A	117.8
C5—C4—H4A	119.7	C17—C16—H16A	117.8
C4—C5—C6	120.8 (3)	C18—C17—C16	118.6 (3)
C4—C5—H5A	119.6	C18—C17—H17A	120.7
C6—C5—H5A	119.6	C16—C17—H17A	120.7

C5—C6—C7	118.4 (3)	C17—C18—C19	119.3 (3)
C5—C6—C8	119.6 (3)	C17—C18—H18A	120.3
C7—C6—C8	122.0 (3)	C19—C18—H18A	120.3
C2—C7—C6	120.4 (3)	C18—C19—C15	118.5 (3)
C2—C7—H7A	119.8	C18—C19—C20	125.1 (3)
C6—C7—H7A	119.8	C15—C19—C20	116.4 (3)
N2—C8—N3	112.4 (2)	N3—C20—C9	105.4 (2)
N2—C8—C6	124.3 (3)	N3—C20—C19	131.0 (3)
N3—C8—C6	123.2 (3)	C9—C20—C19	123.6 (3)
C20—C9—N2	111.0 (3)	O1—C21—H21A	109.5
C20—C9—C10	120.9 (3)	O1—C21—H21B	109.5
N2—C9—C10	128.1 (3)	H21A—C21—H21B	109.5
C11—C10—C14	118.3 (3)	O1—C21—H21C	109.5
C11—C10—C9	124.1 (3)	H21A—C21—H21C	109.5
C14—C10—C9	117.6 (3)	H21B—C21—H21C	109.5
C12—C11—C10	119.0 (3)	C8—N2—C9	104.4 (2)
C12—C11—H11A	120.5	C8—N3—C20	106.8 (2)
C10—C11—H11A	120.5	C8—N3—H3B	126.6
C11—C12—C13	118.4 (3)	C20—N3—H3B	126.6
C11—C12—H12A	120.8	C13—N4—C14	116.7 (3)
C13—C12—H12A	120.8	C16—N5—C15	117.8 (3)
N4—C13—C12	125.2 (3)	C21—O1—H1E	108.4
N4—C13—H13A	117.4		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*B*···O1	0.86	1.95	2.803 (3)	174
O1—H1*E*···N5^{i}	0.98	1.89	2.857 (3)	168

Symmetry code: (i) $x-1/2, -y+1/2, z+1/2$.