Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2,2-Dimethyl-*N*-(4-methylpyridin-2-yl)-propanamide

Gamal A. El-Hiti,[a]* Keith Smith,[b] Asim A. Balakit,[c] Amany S. Hegazy[b] and Benson M. Kariuki[b]*

[a]Department of Optometry, College of Applied Medical Sciences, King Saud University, PO Box 10219, Riyadh 11433, Saudi Arabia, [b]School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, Wales, and [c]Department of Chemistry, College of Science for Women, University of Babylon, Babylon, Iraq
Correspondence e-mail: gelhiti@ksu.edu.sa, kariukib@cardiff.ac.uk

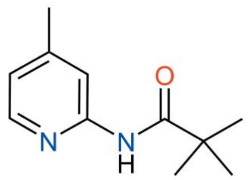
Received 6 February 2014; accepted 18 February 2014

Key indicators: single-crystal X-ray study; *T* = 296 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.046; *wR* factor = 0.154; data-to-parameter ratio = 17.1.

In the title compound, $C_{11}H_{16}N_2O$, the dihedral angle between the mean plane of the 4-methypyridine group and the plane of the amide link is 16.7 (1)°, and there is a short intramolecular C—H···O contact. Hydrogen bonding (N—H···O) between amide groups forms chains parallel to the *b* axis. Pairs of methylpyridine groups from molecules in adjacent chains are parallel but there is minimal π–π interaction.

Related literature

For biological applications of related compounds, see: de Candia *et al.* (2013); Thorat *et al.* (2013); Abdel-Megeed *et al.* (2012). For convenient routes for modifying pyridine derivatives, see: Smith *et al.* (2013); Smith *et al.* (2012); El-Hiti (2003); Joule & Mills (2000); Smith *et al.* (1994, 1995, 1999); Turner (1983). For the X-ray structures of related compounds, see: Mazik & Sicking (2004); Mazik *et al.* (2004); Hodorowicz *et al.* (2007); Koch *et al.* (2008); Liang *et al.* (2008); Seidler *et al.* (2011).



Experimental

Crystal data

$C_{11}H_{16}N_2O$	$V = 2290.35\ (10)\ \text{Å}^3$
$M_r = 192.26$	$Z = 8$
Orthorhombic, *Pbca*	Cu $K\alpha$ radiation
$a = 10.7954\ (3)$ Å	$\mu = 0.58\ \text{mm}^{-1}$
$b = 10.1809\ (2)$ Å	$T = 296$ K
$c = 20.8390\ (5)$ Å	0.27 × 0.19 × 0.14 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer	5219 measured reflections
Absorption correction: gaussian (*CrysAlis PRO*; Agilent, 2014)	2253 independent reflections
$T_{\min} = 0.930$, $T_{\max} = 0.957$	1808 reflections with $I > 2\sigma(I)$
	$R_{\rm int} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$	132 parameters
$wR(F^2) = 0.154$	H-atom parameters constrained
$S = 1.08$	$\Delta\rho_{\max} = 0.16$ e Å^{-3}
2253 reflections	$\Delta\rho_{\min} = -0.14$ e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1^{i}	0.86	2.22	3.0644 (17)	168
C5—H5···O1	0.93	2.28	2.842 (2)	118

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, z$.

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *CHEMDRAW Ultra* (CambridgeSoft, 2001).

The authors extend their appreciation to the Deanship of Scientific Research at King Saud University for its funding of this research through the research group project RGP-VPP-239.

Supporting information for this paper is available from the IUCr electronic archives (Reference: MW2120).

References

Abdel-Megeed, M. F., Badr, B. E., Azaam, M. M. & El-Hiti, G. A. (2012). *Bioorg. Med. Chem.* **20**, 2252–2258.
Agilent (2014). *CrysAlis PRO*. Agilent Technologies, Yarnton, Oxfordshire, England.
CambridgeSoft (2001). *CHEMDRAW Ultra*. CambridgeSoft Corporation, Cambridge, Massachusetts, USA.
Candia, M. de, Fiorella, F., Lopopolo, G., Carotti, A., Romano, M. R., Lograno, M. D., Martel, S., Carrupt, P.-A., Belviso, B. D., Caliandro, R. & Altomare, C. (2013). *J. Med. Chem.* **56**, 8696–8711.
El-Hiti, G. A. (2003). *Monatsh. Chem.* **134**, 837–841.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Hodorowicz, M., Stadnicka, K., Trzewik, B. & Zaleska, B. (2007). *Acta Cryst.* E**63**, o4115.
Joule, J. A. & Mills, K. (2000). *Heterocyclic Chemistry*, 4th ed. England: Blackwell Science Publishers.
Koch, P., Schollmeyer, D. & Laufer, S. (2008). *Acta Cryst.* E**64**, o2216.
Liang, D., Gao, L.-X., Gao, Y., Xu, J. & Wang, W. (2008). *Acta Cryst.* E**64**, o201.
Mazik, M., Radunz, W. & Boese, R. (2004). *J. Org. Chem.* **69**, 7448–7462.
Mazik, M. & Sicking, W. (2004). *Tetrahedron Lett.* **45**, 3117–3121.
Seidler, T., Gryl, M., Trzewik, B. & Stadnicka, K. (2011). *Acta Cryst.* E**67**, o1507.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smith, K., Anderson, D. & Matthews, I. (1995). *Sulfur Lett.* **18**, 79–95.
Smith, K., El-Hiti, G. A. & Alshammari, M. B. (2013). *Synthesis*, **45**, 3426–3434.
Smith, K., El-Hiti, G. A., Fekri, A. & Alshammari, M. B. (2012). *Heterocycles*, **86**, 391–410.

Smith, K., El-Hiti, G. A., Pritchard, G. J. & Hamilton, A. (1999). *J. Chem. Soc. Perkin Trans. 1*, pp. 2299–2303.

Smith, K., Lindsay, C. M., Morris, I. K., Matthews, I. & Pritchard, G. J. (1994). *Sulfur Lett.* **17**, 197–216.

Thorat, S. A., Kang, D. W., Ryu, H. C., Kim, M. S., Kim, H. S., Ann, J., Ha, T., Kim, S.-E., Son, K., Choi, S., Blumberg, P. M., Frank, R., Bahrenberg, G., Schiene, K., Christoph, T. & Lee, J. (2013). *Eur. J. Med. Chem.* **64**, 589–602.

Turner, J. A. (1983). *J. Org. Chem.* **48**, 3401–3408.

o352 El-Hiti *et al.* • $C_{11}H_{16}N_2O$ *Acta Cryst.* (2014). E**70**, o351–o352

supporting information

Acta Cryst. (2014). E**70**, o351–o352 [doi:10.1107/S1600536814003729]

2,2-Dimethyl-*N*-(4-methylpyridin-2-yl)propanamide

Gamal A. El-Hiti, Keith Smith, Asim A. Balakit, Amany S. Hegazy and Benson M. Kariuki

S1. Structural commentary

Synthetic and naturally occurring pyridine derivatives have a broad range of biological activities (Thorat *et al.*, 2013) including anticancer and antimicrobial (Abdel-Megeed *et al.*, 2012) and anticoagulant (de Candia *et al.*, 2013) properties. Hence, pyridine derivatives are important compounds (Joule and Mills, 2000) and some synthetic approaches involve lithiation of 2-acylaminopyridines (Smith *et al.*, 1995; Turner, 1983). The structures of a number of 2-acylaminopyridines have been determined (Mazik & Sicking, 2004; Mazik *et al.*, 2004; Hodorowicz *et al.*, 2007; Koch *et al.*, 2008; Liang *et al.*, 2008; Seidler *et al.*, 2011). During research focused on new synthetic routes towards novel substituted pyridine derivatives (Smith *et al.*, 1994; Smith *et al.*, 1995; Smith *et al.*, 1999; El-Hiti, 2003; Smith *et al.*, 2012; Smith *et al.*, 2013) we have synthesized the title compound in high yield. In the 4-methyl-2-pivaloylaminopyridine molecule (Figure 1), the least squares plane through the 4-methypyridine group makes a dihedral angle of 16.7 (1)° with the plane through the amide link and a short intramolecular C5—H5···O1 contact is observed (Table 1). In the crystal structure (Figure 2) N—H···O hydrogen bonding between amide groups forms chains parallel to the *b* axis. Pairs of methyl-pyridine groups in molecules from adjacent chains are parallel but there is minimal *π*-*π* interaction. The ring nitrogen is not involved in strong hydrogen bonding.

S2. Synthesis and crystallization

To a cooled solution (0 °C) of 2-amino-4-methylpyridine (5.41 g, 50.0 mmol) and triethylamine (10 ml) in dichloromethane (DCM, 80 ml) pivaloyl chloride (6.63 g, 55.0 mmol) was slowly added in a drop-wise manner over 10 min. The reaction mixture was stirred at 0 °C for an extra 1 h. The mixture was poured into H_2O (100 ml) and the organic layer was separated, washed with H_2O (2 × 50 ml), dried ($MgSO_4$) and evaporated under reduced pressure to remove the solvent. The solid obtained was purified by crystallization from Et_2O–hexane (2:1) to give 4-methyl-2-pivaloylaminopyridine (9.04 g, 47.0 mmol; 94%) as colourless crystals, m.p. 103–104 °C [lit. 96–98 °C (hexane); Turner (1983)]. ^{1}H NMR (500 MHz, $CDCl_3$, δ, p.p.m.) 8.11–8.10 (br, 2 H, H-3 and H-6), 8.05 (br, exch., 1 H, NH), 6.85 (m, 1 H, H-5), 2.34 (s, 3 H, CH_3), 1.31 [s, 9 H, $C(CH_3)_3$]. ^{13}CNMR (125 MHz, $CDCl_3$, δ, p.p.m.) 177.2 (s, C=O), 151.5 (s, C-4), 149.9 (s, C-2), 147.2 (d, C-6), 120.9 (d, C-5), 114.5 (d, C-3), 39.8 [s, $\mathit{C}(CH_3)_3$], 27.5 [q, $C(\mathit{C}H_3)_3$]), 21.4 (q, CH_3). EI^+–MS (*m/z*, %): 192 (M^+, 43), 177 (5), 149 (11), 135 (25), 108 (100), 92 (15), 81 (15), 57 (25). HRMS (EI^+): Calculated for $C_{11}H_{16}N_2O$ [*M*] 192.1263; found, 192.1260.

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were positioned geometrically and refined using a riding model with $U_{iso}(H)$ = 1.2 times U_{eq} for the atom they are bonded to except for the methyl groups where 1.5 times U_{eq} was used with free rotation about the C—C bond.

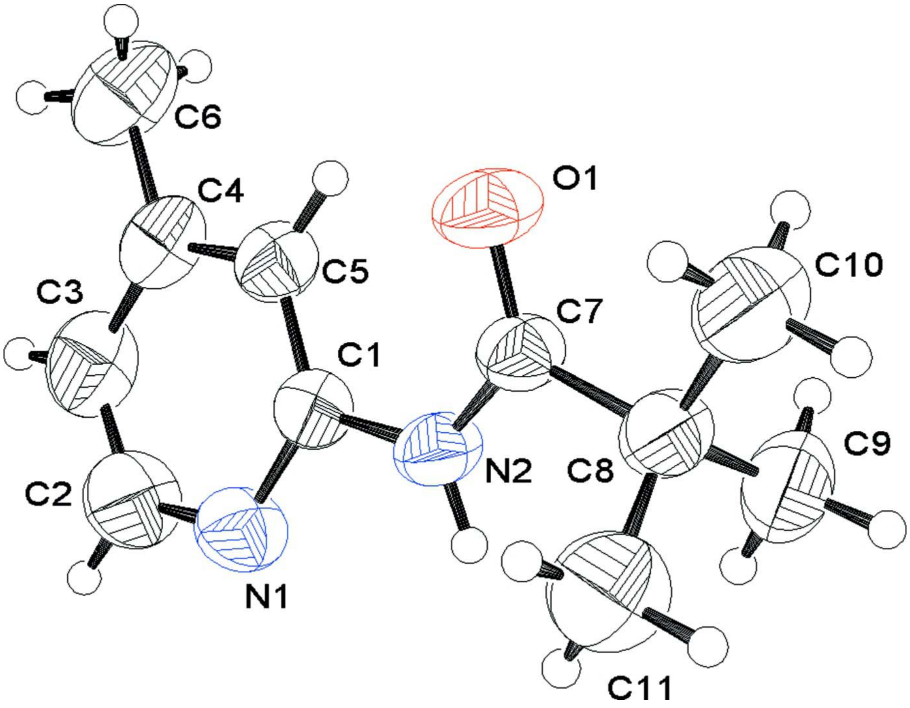


Figure 1

A molecule showing atom labels and 50% probability displacement ellipsoids for non-H atoms.

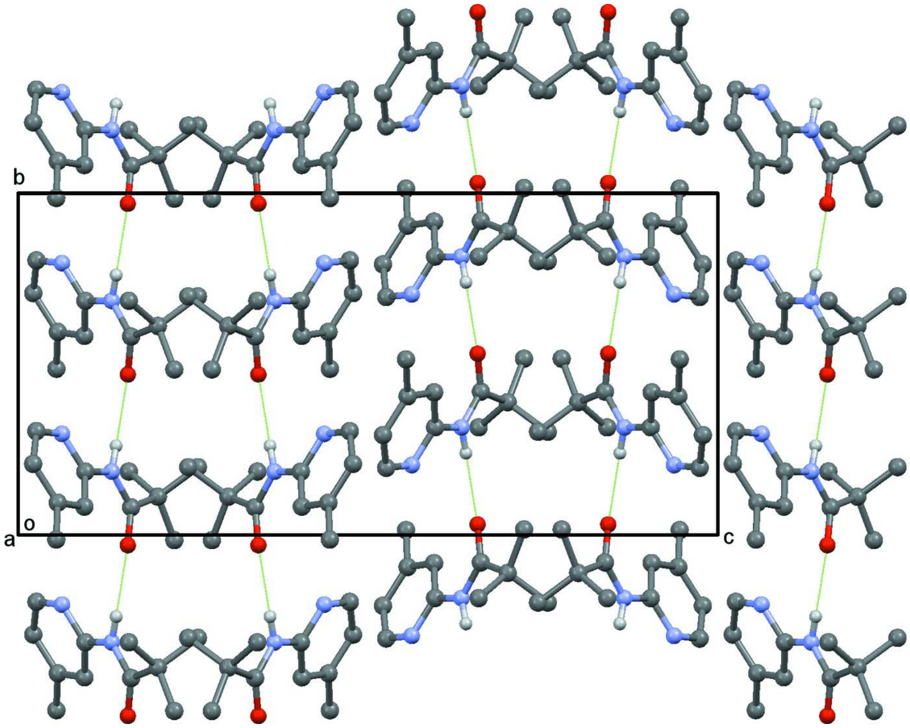


Figure 2

Crystal structure packing showing NH..O hydrogen bonds as green dotted lines with the rest of the hydrogen atoms omitted for clarity.

2,2-Dimethyl-*N*-(4-methylpyridin-2-yl)propanamide

Crystal data

$C_{11}H_{16}N_2O$
$M_r = 192.26$
Orthorhombic, *Pbca*
$a = 10.7954$ (3) Å
$b = 10.1809$ (2) Å
$c = 20.8390$ (5) Å
$V = 2290.35$ (10) Å^3
$Z = 8$
$F(000) = 832$
$D_x = 1.115$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 1808 reflections
$\theta = 4.2$–74.0°
$\mu = 0.58$ mm^{-1}
$T = 296$ K
Block, colourless
0.27 × 0.19 × 0.14 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Radiation source: sealed X-ray tube
ω scans
Absorption correction: gaussian (*CrysAlis PRO*; Agilent, 2014)
$T_{min} = 0.930$, $T_{max} = 0.957$
5219 measured reflections
2253 independent reflections
1808 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 74.0°$, $\theta_{min} = 4.2°$
$h = -7 \rightarrow 13$
$k = -12 \rightarrow 8$
$l = -25 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.154$
$S = 1.08$
2253 reflections
132 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2 + 0.4299P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL2013* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0037 (5)

Special details

Experimental. Absorption correction: CrysAlisPro (Agilent, 2014): Numerical absorption correction based on Gaussian integration over a multifaceted crystal model. Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.90730 (14)	0.81479 (14)	0.59591 (7)	0.0489 (4)
C2	1.0228 (2)	0.6963 (2)	0.52600 (11)	0.0816 (6)
H2	1.0345	0.6201	0.5021	0.098*
C3	1.1102 (2)	0.7922 (2)	0.52141 (11)	0.0792 (6)
H3	1.1786	0.7811	0.4948	0.095*
C4	1.09637 (16)	0.90571 (19)	0.55651 (9)	0.0632 (5)

C5	0.99149 (14)	0.91744 (16)	0.59451 (8)	0.0552 (4)
H5	0.9779	0.9929	0.6186	0.066*
C6	1.1912 (2)	1.0138 (3)	0.55444 (12)	0.0922 (7)
H6A	1.2409	1.0107	0.5926	0.138*
H6B	1.1499	1.0972	0.5520	0.138*
H6C	1.2431	1.0025	0.5175	0.138*
C7	0.74226 (15)	0.91931 (14)	0.66136 (8)	0.0515 (4)
C8	0.62472 (16)	0.88824 (16)	0.69967 (9)	0.0606 (5)
C9	0.6598 (2)	0.8017 (2)	0.75696 (11)	0.0851 (7)
H9A	0.7235	0.8442	0.7815	0.128*
H9B	0.6895	0.7185	0.7418	0.128*
H9C	0.5882	0.7883	0.7835	0.128*
C10	0.5677 (2)	1.0158 (2)	0.72312 (13)	0.0997 (9)
H10A	0.6251	1.0600	0.7509	0.149*
H10B	0.4929	0.9970	0.7463	0.149*
H10C	0.5490	1.0709	0.6870	0.149*
C11	0.53133 (19)	0.8146 (3)	0.65810 (13)	0.0933 (8)
H11A	0.4578	0.7976	0.6826	0.140*
H11B	0.5668	0.7329	0.6442	0.140*
H11C	0.5107	0.8670	0.6213	0.140*
N1	0.92138 (14)	0.70437 (14)	0.56251 (8)	0.0654 (4)
N2	0.79937 (12)	0.81473 (12)	0.63366 (7)	0.0554 (4)
H2A	0.7654	0.7394	0.6400	0.067*
O1	0.78283 (12)	1.03049 (11)	0.65664 (7)	0.0699 (4)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0475 (8)	0.0469 (8)	0.0523 (8)	0.0056 (6)	0.0003 (6)	0.0012 (6)
C2	0.0788 (13)	0.0735 (12)	0.0924 (14)	0.0131 (10)	0.0224 (11)	−0.0152 (11)
C3	0.0626 (11)	0.0889 (14)	0.0861 (13)	0.0180 (10)	0.0226 (10)	0.0029 (11)
C4	0.0481 (9)	0.0720 (11)	0.0694 (10)	0.0027 (8)	0.0020 (7)	0.0171 (9)
C5	0.0518 (9)	0.0531 (9)	0.0607 (9)	0.0001 (7)	0.0028 (7)	0.0032 (7)
C6	0.0587 (11)	0.1052 (17)	0.1126 (18)	−0.0166 (11)	0.0083 (11)	0.0244 (15)
C7	0.0533 (8)	0.0390 (7)	0.0622 (8)	0.0022 (6)	0.0061 (7)	−0.0009 (6)
C8	0.0592 (10)	0.0478 (8)	0.0749 (10)	0.0016 (7)	0.0190 (8)	−0.0021 (7)
C9	0.0969 (16)	0.0788 (13)	0.0798 (13)	0.0035 (12)	0.0275 (12)	0.0104 (10)
C10	0.1026 (17)	0.0605 (12)	0.136 (2)	0.0135 (11)	0.0640 (16)	−0.0025 (12)
C11	0.0548 (11)	0.1121 (19)	0.1129 (18)	−0.0056 (11)	0.0126 (12)	−0.0159 (15)
N1	0.0646 (9)	0.0546 (8)	0.0770 (9)	0.0056 (7)	0.0107 (7)	−0.0124 (7)
N2	0.0551 (8)	0.0401 (7)	0.0711 (8)	−0.0038 (5)	0.0153 (6)	−0.0040 (6)
O1	0.0673 (8)	0.0389 (6)	0.1034 (10)	−0.0010 (5)	0.0213 (7)	−0.0030 (6)

Geometric parameters (Å, º)

C1—N1	1.3310 (19)	C7—N2	1.3590 (19)
C1—C5	1.385 (2)	C7—C8	1.532 (2)
C1—N2	1.406 (2)	C8—C10	1.518 (2)

C2—N1	1.336 (3)	C8—C11	1.526 (3)
C2—C3	1.361 (3)	C8—C9	1.531 (3)
C2—H2	0.9300	C9—H9A	0.9600
C3—C4	1.376 (3)	C9—H9B	0.9600
C3—H3	0.9300	C9—H9C	0.9600
C4—C5	1.387 (2)	C10—H10A	0.9600
C4—C6	1.503 (3)	C10—H10B	0.9600
C5—H5	0.9300	C10—H10C	0.9600
C6—H6A	0.9600	C11—H11A	0.9600
C6—H6B	0.9600	C11—H11B	0.9600
C6—H6C	0.9600	C11—H11C	0.9600
C7—O1	1.2177 (18)	N2—H2A	0.8600

N1—C1—C5	123.45 (15)	C11—C8—C9	108.85 (18)
N1—C1—N2	112.76 (13)	C10—C8—C7	109.09 (14)
C5—C1—N2	123.78 (14)	C11—C8—C7	110.66 (15)
N1—C2—C3	124.35 (19)	C9—C8—C7	108.69 (15)
N1—C2—H2	117.8	C8—C9—H9A	109.5
C3—C2—H2	117.8	C8—C9—H9B	109.5
C2—C3—C4	119.33 (18)	H9A—C9—H9B	109.5
C2—C3—H3	120.3	C8—C9—H9C	109.5
C4—C3—H3	120.3	H9A—C9—H9C	109.5
C3—C4—C5	117.68 (17)	H9B—C9—H9C	109.5
C3—C4—C6	121.72 (19)	C8—C10—H10A	109.5
C5—C4—C6	120.60 (19)	C8—C10—H10B	109.5
C1—C5—C4	118.87 (16)	H10A—C10—H10B	109.5
C1—C5—H5	120.6	C8—C10—H10C	109.5
C4—C5—H5	120.6	H10A—C10—H10C	109.5
C4—C6—H6A	109.5	H10B—C10—H10C	109.5
C4—C6—H6B	109.5	C8—C11—H11A	109.5
H6A—C6—H6B	109.5	C8—C11—H11B	109.5
C4—C6—H6C	109.5	H11A—C11—H11B	109.5
H6A—C6—H6C	109.5	C8—C11—H11C	109.5
H6B—C6—H6C	109.5	H11A—C11—H11C	109.5
O1—C7—N2	122.06 (15)	H11B—C11—H11C	109.5
O1—C7—C8	122.14 (14)	C1—N1—C2	116.32 (16)
N2—C7—C8	115.80 (13)	C7—N2—C1	127.83 (13)
C10—C8—C11	109.58 (19)	C7—N2—H2A	116.1
C10—C8—C9	109.95 (18)	C1—N2—H2A	116.1

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1$^{\rm i}$	0.86	2.22	3.0644 (17)	168
C5—H5···O1	0.93	2.28	2.842 (2)	118

Symmetry code: (i) $-x+3/2, y-1/2, z$.