Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

OPEN ACCESS

ISSN 2056-9890

Crystal structure of 2,2-dimethyl-*N*-(5-methylpyridin-2-yl)propanamide

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

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

Received 16 May 2015; accepted 17 May 2015

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

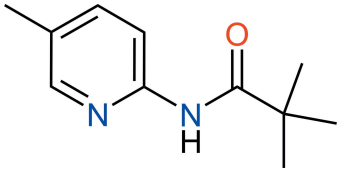
There are two molecules in the asymmetric unit of the title compound, $C_{11}H_{16}N_2O$. The pyridine rings and amide groups overlap almost perfectly (r.m.s. overlay fit = 0.053 Å), but the tertiary butyl groups have different orientations: in one molecule, one of the methyl C atoms is *syn* to the amide O atom [O—C—C—C = −0.8 (3)°] and in the other the equivalent torsion angle is 31.0 (2)°. In the crystal, the two independent molecules are linked by a pair of N—H···N hydrogen bonds in the form of an $R_2^2(8)$ loop to form a dimer. A C—H···O interaction connects the dimers into [100] chains.

Keywords: crystal structure; propanamide; hydrogen bonding.

CCDC reference: 1401551

1. Related literature

For the synthesis and spectroscopic data, see: Turner (1983). For related compounds, see: El-Hiti *et al.* (2015*a*,*b*); de Candia *et al.* (2013); Smith *et al.* (2013, 2012); Abdel-Megeed *et al.* (2012); Joule & Mills (2000). For the crystal structures of related compounds, see: El-Hiti *et al.* (2014); Seidler *et al.* (2011); Koch *et al.* (2008).



2. Experimental

2.1. Crystal data

$C_{11}H_{16}N_2O$ M_r = 192.26
Monoclinic, $P2_1/n$
a = 11.1969 (2) Å
b = 8.6439 (2) Å
c = 23.8844 (5) Å
β = 94.549 (2)°
V = 2304.37 (8) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 0.57 mm^{-1}
T = 296 K
0.47 × 0.33 × 0.11 mm

2.2. Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Absorption correction: gaussian (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.925, T_{max} = 0.975
8391 measured reflections
4503 independent reflections
3684 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.148
S = 1.04
4503 reflections
262 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 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
N2—H2*A*···N3	0.86	2.31	3.1192 (16)	156
N4—H4*A*···N1	0.86	2.25	3.0837 (16)	163
C4—H4···O2^{i}	0.93	2.43	3.3262 (19)	160

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

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2015); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Acknowledgements

The authors extend their appreciation to the Cornea Research Chair, Department of Optometry, College of Applied Medical Sciences, King Saud University, for funding this research.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7430).

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, England.
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., Smith, K., Balakit, A. A., Hegazy, A. S. & Kariuki, B. M. (2014). *Acta Cryst.* E**70**, o351–o352.
El-Hiti, A., Smith, G. & Hegazy, K. (2015*a*). *Heterocycles*, **91**, 479–504.
El-Hiti, G. A., Smith, K., Hegazy, A. S., Alanazi, S. A. & Kariuki, B. M. (2015*b*). *Acta Cryst.* E**71**, o246–o247.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
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.
Seidler, T., Gryl, M., Trzewik, B. & Stadnicka, K. (2011). *Acta Cryst.* E**67**, o1507.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Smith, K., El-Hiti, G. A., Alshammari, M. B. & Fekri, A. (2013). *Synthesis*, **45**, 3426–3434.
Smith, K. A., El-Hiti, G. A., Fekri, A. & Alshammari, B. (2012). *Heterocycles*, **86**, 391–410.
Turner, J. A. (1983). *J. Org. Chem.* **48**, 3401–3408.

supporting information

Acta Cryst. (2015). E**71**, o419–o420 [doi:10.1107/S2056989015009378]

Crystal structure of 2,2-dimethyl-*N*-(5-methylpyridin-2-yl)propanamide

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

S1. Introduction

Substituted pyridines are important compounds (Joule & Mills, 2000) and show a range of biological activities (de Candia *et al.*, 2013; Abdel-Megeed *et al.*, 2012). The pyridine ring system has been modified using various efficient and simple procedures that include the use of lithium reagents as intermediates (El-Hiti *et al.*, 2015; Smith *et al.*, 2013, Smith *et al.*, 2012; Turner, 1983). The X-ray crystal structures of related compounds have been reported (El-Hiti *et al.*, 2015; El-Hiti *et al.*, 2014; Seidler *et al.*, 2011; Koch *et al.*, 2008).

S2. Experimental

S2.1. Synthesis and crystallization

2,2-Dimethyl-*N*-(5-methylpyridin-2-yl)propanamide was obtained in 83% yield from reaction of 2-amino-5-methylpyridine with trimethylacetyl chloride in the presence of triethylamine in dichloromethane at 0 °C for 15 minutes and then at room temperature for 2 h (Turner, 1983). The crude product was purified by column chromatography (silica gel; dichloromethane) followed by crystallization from hexane to give colourless crystals of the title compound. The NMR spectral data and elemental analyses for the title compound were identical with those previously reported (Turner, 1983).

S2.2. Refinement

H atoms were positioned geometrically and refined using a riding model with U_{iso}(H) constrained to be 1.2 times U_{eq} for the atom it is bonded to except for methyl groups where it was 1.5 times with free rotation about the C—C bond.

S3. Results and discussion

The asymmetric unit (Figure 1) consists of two independent molecules of $C_{11}H_{16}N_2O$. The amide group and pyridine ring within the molecule are not co-planar as indicated by the torsion angles [N1—C1—N2—C7 = 142.20 (14), N3—C12—N4—C18 = 148.58 (15)]. The two independent molecules are linked by a pair of N—H···N hydrogen bonds (Table 1, Figure 2) to form an $R_2^2(8)$ ring.

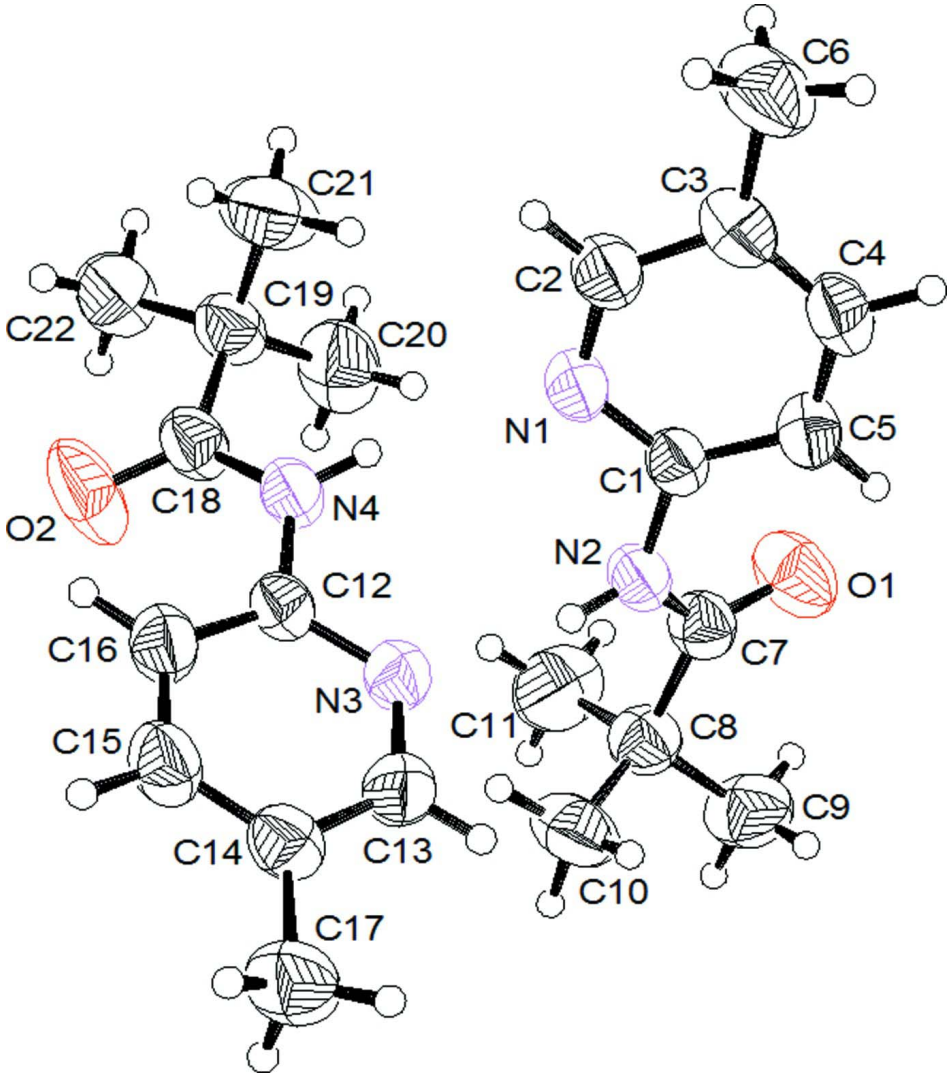


Figure 1

The asymmetric unit of $C_{11}H_{16}N_2O$ with 50% probability displacement ellipsoids for nonhydrogen atoms.

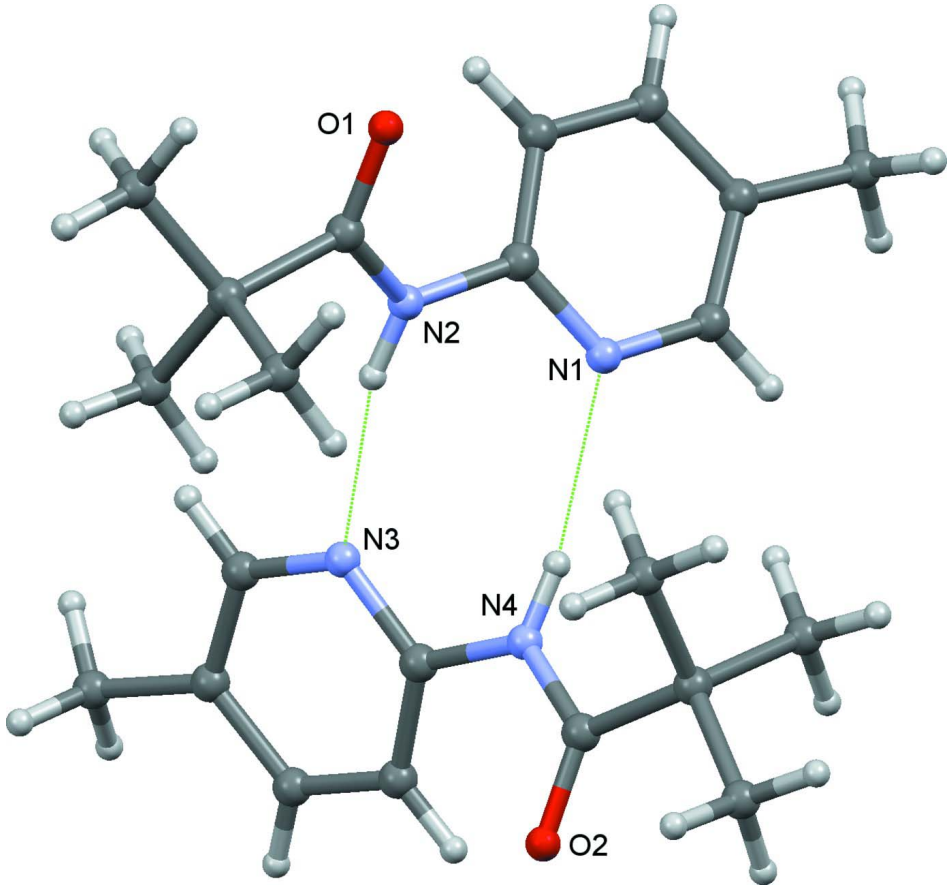


Figure 2

The asymmetric unit showing N—H···N interactions as dotted lines.

2,2-Dimethyl-*N*-(5-methylpyridin-2-yl)propanamide

Crystal data

$C_{11}H_{16}N_2O$
$M_r = 192.26$
Monoclinic, $P2_1/n$
$a = 11.1969$ (2) Å
$b = 8.6439$ (2) Å
$c = 23.8844$ (5) Å
$\beta = 94.549$ (2)°
$V = 2304.37$ (8) Å^3
$Z = 8$
$F(000) = 832$
$D_x = 1.108$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 3890 reflections
$\theta = 3.9$–$73.6°$
$\mu = 0.57$ mm^{-1}
$T = 296$ K
Plate, colourless
0.47 × 0.33 × 0.11 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Radiation source: sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
ω scans
Absorption correction: gaussian (*CrysAlis PRO*; Agilent, 2014)
$T_{min} = 0.925$, $T_{max} = 0.975$
8391 measured reflections
4503 independent reflections
3684 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 74.0°$, $\theta_{min} = 3.7°$
$h = -13 \rightarrow 13$
$k = -9 \rightarrow 10$
$l = -29 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.148$
$S = 1.04$
4503 reflections
262 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.081P)^2 + 0.208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL2013* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0075 (6)

Special details

Experimental. Absorption correction: CrysAlisPro, Agilent Technologies, Version 1.171.37.33 (release 27-03-2014 CrysAlis171 .NET) (compiled Mar 27 2014,17:12:48) 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.20122 (11)	0.54209 (16)	0.13526 (5)	0.0525 (3)
C2	0.19343 (14)	0.77138 (18)	0.08875 (7)	0.0650 (4)
H2	0.1480	0.8535	0.0736	0.097*
C3	0.31560 (14)	0.77581 (19)	0.08401 (7)	0.0670 (4)
C4	0.38057 (13)	0.6528 (2)	0.10739 (7)	0.0701 (4)
H4	0.4632	0.6499	0.1055	0.105*
C5	0.32450 (12)	0.5351 (2)	0.13327 (7)	0.0654 (4)
H5	0.3681	0.4524	0.1492	0.098*
C6	0.3730 (2)	0.9087 (3)	0.05543 (12)	0.1036 (7)
H6A	0.4566	0.9132	0.0677	0.155*
H6B	0.3349	1.0037	0.0649	0.155*
H6C	0.3639	0.8939	0.0155	0.155*
C7	0.15945 (14)	0.27433 (18)	0.16115 (6)	0.0618 (4)
C8	0.07563 (15)	0.16852 (19)	0.19135 (7)	0.0689 (4)
C9	0.1450 (2)	0.0226 (2)	0.21008 (9)	0.0942 (6)
H9A	0.1777	−0.0243	0.1782	0.141*
H9B	0.0920	−0.0490	0.2263	0.141*
H9C	0.2090	0.0496	0.2375	0.141*
C10	0.0273 (2)	0.2443 (2)	0.24272 (9)	0.0918 (6)
H10A	−0.0163	0.1692	0.2626	0.138*
H10B	−0.0250	0.3280	0.2309	0.138*
H10C	0.0929	0.2832	0.2670	0.138*
C11	−0.0263 (2)	0.1254 (3)	0.14762 (12)	0.1106 (8)
H11A	0.0066	0.0838	0.1149	0.166*
H11B	−0.0726	0.2160	0.1374	0.166*

H11C	−0.0768	0.0494	0.1631	0.166*
C12	−0.12246 (11)	0.71207 (16)	0.19991 (6)	0.0520 (3)
C13	−0.02137 (14)	0.65237 (19)	0.28397 (6)	0.0650 (4)
H13	0.0409	0.5995	0.3037	0.098*
C14	−0.09434 (14)	0.74294 (19)	0.31429 (6)	0.0634 (4)
C15	−0.18413 (15)	0.8222 (2)	0.28376 (7)	0.0710 (4)
H15	−0.2350	0.8861	0.3023	0.106*
C16	−0.19970 (14)	0.8082 (2)	0.22610 (7)	0.0660 (4)
H16	−0.2603	0.8618	0.2054	0.099*
C17	−0.0774 (2)	0.7526 (3)	0.37741 (7)	0.0909 (6)
H17A	−0.0997	0.8538	0.3894	0.136*
H17B	0.0051	0.7335	0.3895	0.136*
H17C	−0.1268	0.6765	0.3936	0.136*
C18	−0.22818 (13)	0.6925 (2)	0.10586 (6)	0.0671 (4)
C19	−0.21118 (14)	0.6626 (2)	0.04398 (6)	0.0714 (4)
C20	−0.1518 (2)	0.5048 (3)	0.03776 (9)	0.1036 (7)
H20A	−0.0736	0.5054	0.0575	0.155*
H20B	−0.1445	0.4838	−0.0013	0.155*
H20C	−0.1999	0.4260	0.0532	0.155*
C21	−0.1325 (2)	0.7892 (3)	0.02172 (9)	0.1074 (8)
H21A	−0.1659	0.8887	0.0293	0.161*
H21B	−0.1291	0.7767	−0.0181	0.161*
H21C	−0.0532	0.7819	0.0400	0.161*
C22	−0.33408 (17)	0.6641 (3)	0.01113 (8)	0.0955 (7)
H22A	−0.3810	0.5790	0.0230	0.143*
H22B	−0.3242	0.6546	−0.0283	0.143*
H22C	−0.3742	0.7596	0.0180	0.143*
N1	0.13561 (10)	0.65829 (14)	0.11338 (5)	0.0578 (3)
N2	0.13689 (10)	0.42840 (14)	0.16285 (5)	0.0577 (3)
H2A	0.0795	0.4589	0.1821	0.086*
N3	−0.03374 (10)	0.63507 (14)	0.22795 (5)	0.0589 (3)
N4	−0.12739 (10)	0.69100 (15)	0.14133 (5)	0.0585 (3)
H4A	−0.0606	0.6758	0.1267	0.088*
O1	0.23876 (14)	0.22212 (15)	0.13479 (7)	0.0942 (4)
O2	−0.32545 (10)	0.7145 (3)	0.12290 (6)	0.1234 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0471 (6)	0.0604 (7)	0.0506 (6)	−0.0005 (5)	0.0069 (5)	−0.0061 (5)
C2	0.0609 (8)	0.0606 (8)	0.0750 (9)	0.0020 (6)	0.0152 (7)	0.0019 (7)
C3	0.0619 (8)	0.0670 (9)	0.0739 (9)	−0.0103 (7)	0.0180 (7)	−0.0082 (7)
C4	0.0458 (7)	0.0872 (11)	0.0783 (10)	−0.0058 (7)	0.0111 (6)	−0.0054 (8)
C5	0.0476 (7)	0.0792 (10)	0.0695 (9)	0.0046 (7)	0.0061 (6)	0.0024 (7)
C6	0.0929 (14)	0.0845 (13)	0.139 (2)	−0.0182 (11)	0.0419 (13)	0.0105 (12)
C7	0.0633 (8)	0.0634 (8)	0.0595 (8)	0.0033 (7)	0.0093 (6)	−0.0058 (6)
C8	0.0743 (10)	0.0600 (8)	0.0730 (9)	−0.0017 (7)	0.0096 (8)	0.0039 (7)
C9	0.1260 (17)	0.0716 (11)	0.0864 (12)	0.0159 (11)	0.0172 (12)	0.0096 (9)

C10	0.1089 (15)	0.0753 (11)	0.0977 (13)	0.0020 (10)	0.0486 (12)	0.0135 (10)
C11	0.0982 (15)	0.1086 (17)	0.1210 (18)	−0.0325 (13)	−0.0168 (14)	0.0207 (14)
C12	0.0441 (6)	0.0584 (7)	0.0541 (7)	−0.0037 (5)	0.0082 (5)	−0.0028 (5)
C13	0.0649 (8)	0.0748 (9)	0.0553 (8)	0.0084 (7)	0.0041 (6)	0.0003 (7)
C14	0.0628 (8)	0.0718 (9)	0.0566 (8)	−0.0045 (7)	0.0101 (6)	−0.0074 (6)
C15	0.0643 (9)	0.0830 (10)	0.0672 (9)	0.0103 (8)	0.0145 (7)	−0.0160 (8)
C16	0.0574 (8)	0.0769 (9)	0.0643 (8)	0.0125 (7)	0.0078 (6)	−0.0056 (7)
C17	0.0947 (13)	0.1195 (16)	0.0590 (9)	0.0039 (12)	0.0092 (9)	−0.0124 (10)
C18	0.0479 (7)	0.0957 (11)	0.0581 (8)	−0.0060 (7)	0.0062 (6)	−0.0059 (7)
C19	0.0574 (8)	0.1030 (12)	0.0540 (8)	−0.0056 (8)	0.0058 (6)	−0.0037 (8)
C20	0.1021 (15)	0.1323 (18)	0.0765 (12)	0.0172 (14)	0.0078 (10)	−0.0312 (12)
C21	0.0894 (13)	0.161 (2)	0.0718 (11)	−0.0318 (14)	0.0046 (10)	0.0262 (13)
C22	0.0681 (10)	0.153 (2)	0.0636 (10)	−0.0080 (12)	−0.0058 (8)	−0.0082 (11)
N1	0.0483 (6)	0.0612 (6)	0.0649 (7)	0.0024 (5)	0.0111 (5)	−0.0014 (5)
N2	0.0517 (6)	0.0616 (7)	0.0612 (6)	0.0020 (5)	0.0141 (5)	0.0003 (5)
N3	0.0564 (6)	0.0654 (7)	0.0555 (6)	0.0082 (5)	0.0078 (5)	−0.0030 (5)
N4	0.0464 (6)	0.0772 (8)	0.0526 (6)	0.0019 (5)	0.0081 (5)	−0.0045 (5)
O1	0.1027 (10)	0.0738 (8)	0.1127 (10)	0.0100 (7)	0.0496 (8)	−0.0114 (7)
O2	0.0451 (6)	0.250 (2)	0.0754 (8)	−0.0005 (9)	0.0067 (5)	−0.0370 (11)

Geometric parameters (Å, º)

C1—N1	1.3269 (18)	C12—C16	1.384 (2)
C1—C5	1.3862 (19)	C12—N4	1.4077 (17)
C1—N2	1.4122 (17)	C13—N3	1.3427 (19)
C2—N1	1.3346 (19)	C13—C14	1.378 (2)
C2—C3	1.382 (2)	C13—H13	0.9300
C2—H2	0.9300	C14—C15	1.377 (2)
C3—C4	1.381 (2)	C14—C17	1.507 (2)
C3—C6	1.506 (2)	C15—C16	1.380 (2)
C4—C5	1.368 (2)	C15—H15	0.9300
C4—H4	0.9300	C16—H16	0.9300
C5—H5	0.9300	C17—H17A	0.9600
C6—H6A	0.9600	C17—H17B	0.9600
C6—H6B	0.9600	C17—H17C	0.9600
C6—H6C	0.9600	C18—O2	1.208 (2)
C7—O1	1.2155 (19)	C18—N4	1.3565 (19)
C7—N2	1.3567 (19)	C18—C19	1.527 (2)
C7—C8	1.531 (2)	C19—C21	1.526 (3)
C8—C10	1.527 (3)	C19—C22	1.529 (2)
C8—C9	1.529 (3)	C19—C20	1.531 (3)
C8—C11	1.531 (3)	C20—H20A	0.9600
C9—H9A	0.9600	C20—H20B	0.9600
C9—H9B	0.9600	C20—H20C	0.9600
C9—H9C	0.9600	C21—H21A	0.9600
C10—H10A	0.9600	C21—H21B	0.9600
C10—H10B	0.9600	C21—H21C	0.9600
C10—H10C	0.9600	C22—H22A	0.9600

C11—H11A	0.9600	C22—H22B	0.9600
C11—H11B	0.9600	C22—H22C	0.9600
C11—H11C	0.9600	N2—H2A	0.8600
C12—N3	1.3314 (18)	N4—H4A	0.8600
N1—C1—C5	122.76 (13)	N3—C13—H13	117.7
N1—C1—N2	115.06 (11)	C14—C13—H13	117.7
C5—C1—N2	122.13 (13)	C15—C14—C13	116.30 (14)
N1—C2—C3	125.05 (15)	C15—C14—C17	122.08 (15)
N1—C2—H2	117.5	C13—C14—C17	121.62 (16)
C3—C2—H2	117.5	C14—C15—C16	120.94 (14)
C4—C3—C2	116.01 (14)	C14—C15—H15	119.5
C4—C3—C6	122.73 (16)	C16—C15—H15	119.5
C2—C3—C6	121.25 (17)	C15—C16—C12	118.01 (14)
C5—C4—C3	120.60 (14)	C15—C16—H16	121.0
C5—C4—H4	119.7	C12—C16—H16	121.0
C3—C4—H4	119.7	C14—C17—H17A	109.5
C4—C5—C1	118.49 (15)	C14—C17—H17B	109.5
C4—C5—H5	120.8	H17A—C17—H17B	109.5
C1—C5—H5	120.8	C14—C17—H17C	109.5
C3—C6—H6A	109.5	H17A—C17—H17C	109.5
C3—C6—H6B	109.5	H17B—C17—H17C	109.5
H6A—C6—H6B	109.5	O2—C18—N4	121.25 (14)
C3—C6—H6C	109.5	O2—C18—C19	122.54 (14)
H6A—C6—H6C	109.5	N4—C18—C19	116.20 (13)
H6B—C6—H6C	109.5	C21—C19—C18	109.61 (16)
O1—C7—N2	121.70 (15)	C21—C19—C22	109.63 (17)
O1—C7—C8	121.50 (15)	C18—C19—C22	108.60 (14)
N2—C7—C8	116.72 (13)	C21—C19—C20	109.78 (18)
C10—C8—C9	108.82 (16)	C18—C19—C20	109.45 (16)
C10—C8—C11	111.11 (19)	C22—C19—C20	109.75 (18)
C9—C8—C11	109.45 (17)	C19—C20—H20A	109.5
C10—C8—C7	113.06 (14)	C19—C20—H20B	109.5
C9—C8—C7	108.31 (15)	H20A—C20—H20B	109.5
C11—C8—C7	106.00 (15)	C19—C20—H20C	109.5
C8—C9—H9A	109.5	H20A—C20—H20C	109.5
C8—C9—H9B	109.5	H20B—C20—H20C	109.5
H9A—C9—H9B	109.5	C19—C21—H21A	109.5
C8—C9—H9C	109.5	C19—C21—H21B	109.5
H9A—C9—H9C	109.5	H21A—C21—H21B	109.5
H9B—C9—H9C	109.5	C19—C21—H21C	109.5
C8—C10—H10A	109.5	H21A—C21—H21C	109.5
C8—C10—H10B	109.5	H21B—C21—H21C	109.5
H10A—C10—H10B	109.5	C19—C22—H22A	109.5
C8—C10—H10C	109.5	C19—C22—H22B	109.5
H10A—C10—H10C	109.5	H22A—C22—H22B	109.5
H10B—C10—H10C	109.5	C19—C22—H22C	109.5
C8—C11—H11A	109.5	H22A—C22—H22C	109.5

C8—C11—H11B	109.5	H22B—C22—H22C	109.5
H11A—C11—H11B	109.5	C1—N1—C2	117.08 (12)
C8—C11—H11C	109.5	C7—N2—C1	124.47 (12)
H11A—C11—H11C	109.5	C7—N2—H2A	117.8
H11B—C11—H11C	109.5	C1—N2—H2A	117.8
N3—C12—C16	122.75 (13)	C12—N3—C13	117.35 (12)
N3—C12—N4	113.83 (11)	C18—N4—C12	125.79 (12)
C16—C12—N4	123.38 (13)	C18—N4—H4A	117.1
N3—C13—C14	124.64 (14)	C12—N4—H4A	117.1
N1—C2—C3—C4	−0.6 (3)	O2—C18—C19—C21	119.0 (2)
N1—C2—C3—C6	−179.97 (18)	N4—C18—C19—C21	−61.9 (2)
C2—C3—C4—C5	0.2 (2)	O2—C18—C19—C22	−0.8 (3)
C6—C3—C4—C5	179.56 (18)	N4—C18—C19—C22	178.35 (18)
C3—C4—C5—C1	0.3 (2)	O2—C18—C19—C20	−120.6 (2)
N1—C1—C5—C4	−0.3 (2)	N4—C18—C19—C20	58.5 (2)
N2—C1—C5—C4	−177.72 (14)	C5—C1—N1—C2	0.0 (2)
O1—C7—C8—C10	151.67 (19)	N2—C1—N1—C2	177.54 (12)
N2—C7—C8—C10	−31.6 (2)	C3—C2—N1—C1	0.5 (2)
O1—C7—C8—C9	31.0 (2)	O1—C7—N2—C1	−0.2 (2)
N2—C7—C8—C9	−152.30 (15)	C8—C7—N2—C1	−176.94 (13)
O1—C7—C8—C11	−86.4 (2)	N1—C1—N2—C7	142.20 (14)
N2—C7—C8—C11	90.33 (19)	C5—C1—N2—C7	−40.2 (2)
N3—C13—C14—C15	−1.3 (3)	C16—C12—N3—C13	0.6 (2)
N3—C13—C14—C17	177.95 (17)	N4—C12—N3—C13	178.41 (13)
C13—C14—C15—C16	0.9 (3)	C14—C13—N3—C12	0.5 (2)
C17—C14—C15—C16	−178.27 (18)	O2—C18—N4—C12	0.7 (3)
C14—C15—C16—C12	0.1 (3)	C19—C18—N4—C12	−178.44 (15)
N3—C12—C16—C15	−0.9 (2)	N3—C12—N4—C18	148.58 (15)
N4—C12—C16—C15	−178.46 (15)	C16—C12—N4—C18	−33.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···N3	0.86	2.31	3.1192 (16)	156
N4—H4*A*···N1	0.86	2.25	3.0837 (16)	163
C4—H4···O2^{i}	0.93	2.43	3.3262 (19)	160

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