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Diethyl 4-(4-cyanophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate

Peng Zhang and Weiqun Zhu*

School of Chemistry and Chemical Engineering, Shandong University, Jinan 250100, People's Republic of China
Correspondence e-mail: zhuwq@sdu.edu.cn

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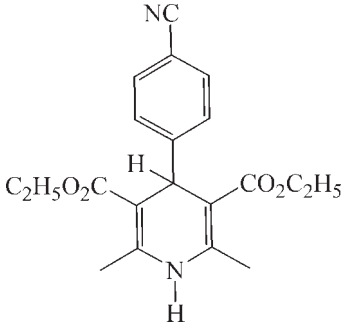
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.051; wR factor = 0.162; data-to-parameter ratio = 13.2.

In the title compound, $C_{20}H_{22}N_2O_4$, the dihedral angle between the roughly planar dihydropyridine ring (r.m.s. deviation = 0.092 Å) and the benzene ring is 87.09 (6)°. One of the ethoxy side chains is disordered over two orientations in a 0.669 (14):0.331 (14) ratio. In the crystal, molecules are linked by N—H···N hydrogen bonds, generating chains.

Related literature

For general background to dihydropyridine derivatives, see: Gaudio *et al.* (1994).



Experimental

Crystal data

$C_{20}H_{22}N_2O_4$
M_r = 354.40
Monoclinic, $P2_1/n$
a = 10.4596 (13) Å
b = 9.5117 (12) Å
c = 19.160 (2) Å
β = 91.493 (1)°
V = 1905.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.990, T_{max} = 0.993
10000 measured reflections
3298 independent reflections
2408 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.162
S = 1.02
3298 reflections
249 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N1^{i}	0.86	2.32	3.098 (3)	150

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; 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: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5444).

References

Bruker (2001). *SAINT-Plus* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Gaudio, A. C., Korolkovas, A. & Takahata, Y. (1994). *J. Pharm. Sci. A*, **83**, 1110–1115.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o1424 [https://doi.org/10.1107/S1600536810018155]

Diethyl 4-(4-cyanophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate

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S1. Comment

The synthesis of 1,4-dihydropyridine derivatives has attracted continuous research interest due to various vasodilator, anti-hypertensive, bronchodilator, heptaprotective, anti-tumor, anti-mutagenic, geroprotective and anti-diabetic agents (Gaudio *et al.*, 1994). Here, we describe the recystallization and structural characterization of the title compound.

The molecular structure is shown in Fig 1. The dihedral angle between the two rings is 87.09 (6) °. The mean devation of the dihydropyridine plane is 0.0824 Å. The intermolecular hydrogen bonding of N2—H2···N1 leads to a consolidation of the structure (Fig. 2; Table 1).

S2. Experimental

Diethyl 2,6-dimethyl-4-(4-cyanophenyl)-1,4-dihydropyridine-3,5-dicarboxylate (1 mmol 0.39 g) was dissolved in 20 ml ethanol was evaporated in one open flask at room temperature. One week later, yellow blocks of (I) were obained. Anal. $C_{20}H_{22}N_2O_4$: C, 67.72; H, 5.64; N, 7.90 %. Found: C, 67.56; H, 5.46; N, 7.61 %.

S3. Refinement

All hydrogen atoms bound to aromatic carbon atoms were refined in calculated positions using a riding model with a C—H distance of 0.93 Å and $U_{iso} = 1.2U_{eq}$(C). Hydrogen atoms attached to aromatic N atoms were refined with a N—H distance of 0.86 Å and $U_{iso} = 1.2U_{eq}$(N).

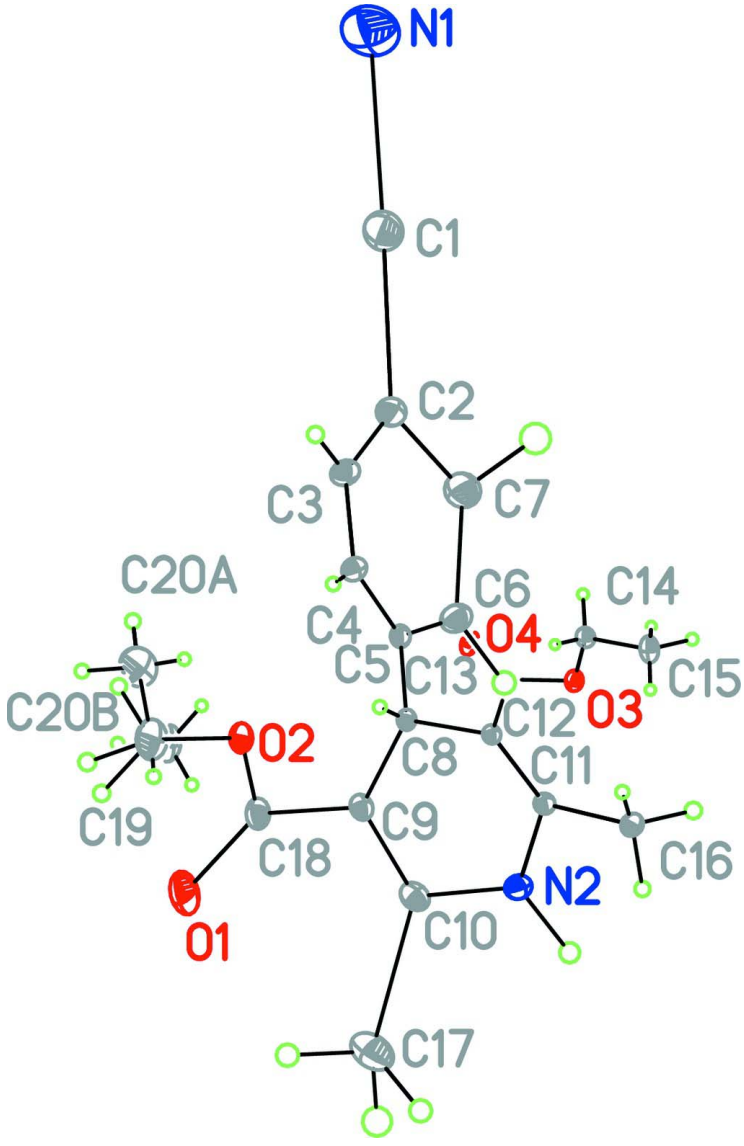

Figure 1

The molecular structure of (I) showing displacement ellipsoids drawn at the 30% probability level.

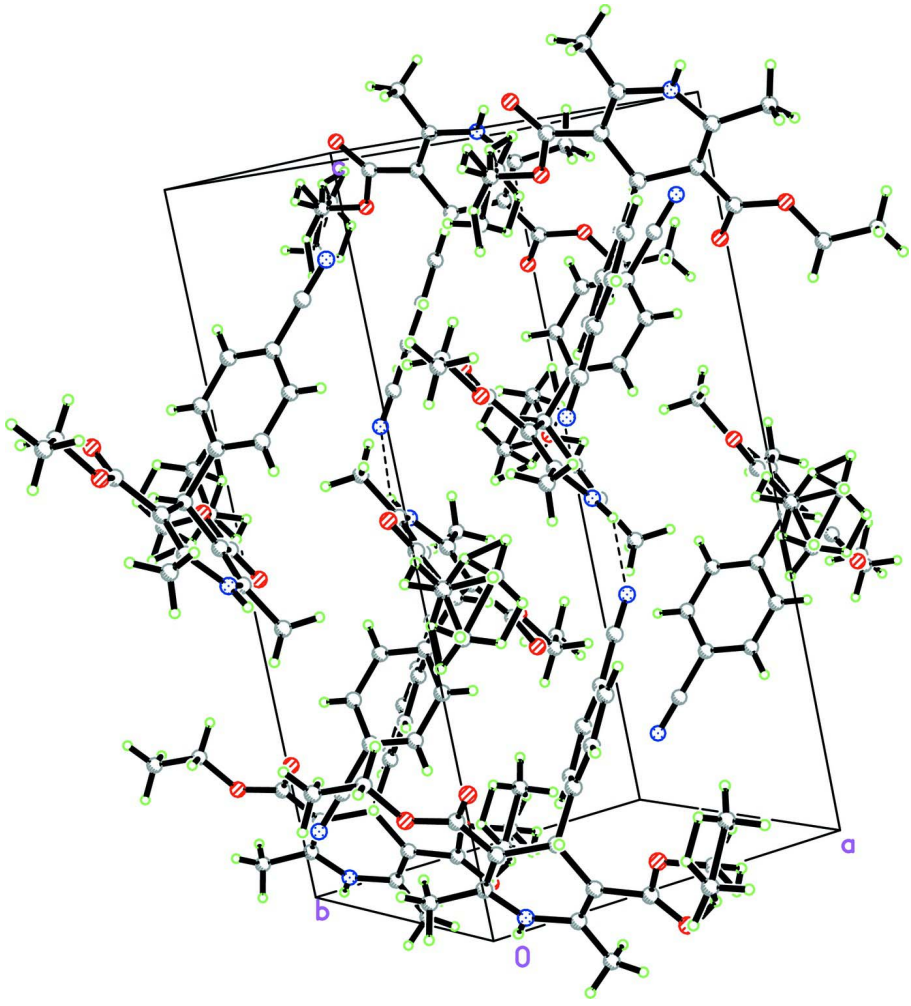

Figure 2

The crystal packing of (I), displayed with N—H···N hydrogen bonds as dashed lines.

Diethyl 4-(4-cyanophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxylate

Crystal data

$C_{20}H_{22}N_2O_4$
$M_r = 354.40$
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Hall symbol: -P 2yn
$a = 10.4596\ (13)$ Å
$b = 9.5117\ (12)$ Å
$c = 19.160\ (2)$ Å
$\beta = 91.493\ (1)°$
$V = 1905.6\ (4)$ Å^3
$Z = 4$

$F(000) = 752$
$D_x = 1.235$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3532 reflections
$\theta = 2.4–25.9°$
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Block, colorless
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator

phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.990$, $T_{max} = 0.993$

10000 measured reflections
3298 independent reflections
2408 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -12 \rightarrow 10$
$k = -9 \rightarrow 11$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.162$
$S = 1.02$
3298 reflections
249 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0857P)^2 + 0.6251P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.012 (2)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}	Occ. (<1)
C1	0.5366 (2)	0.1062 (3)	0.32734 (12)	0.0574 (6)	
C2	0.4507 (2)	0.0690 (2)	0.27004 (10)	0.0483 (5)	
C3	0.3890 (2)	−0.0596 (3)	0.26959 (12)	0.0607 (6)	
H3	0.4024	−0.1222	0.3063	0.073*	
C4	0.3078 (2)	−0.0948 (2)	0.21466 (12)	0.0573 (6)	
H4	0.2670	−0.1817	0.2145	0.069*	
C5	0.28599 (19)	−0.0030 (2)	0.15987 (10)	0.0419 (5)	
C6	0.3497 (2)	0.1239 (2)	0.16056 (12)	0.0535 (6)	
H6	0.3378	0.1854	0.1233	0.064*	
C7	0.4306 (2)	0.1614 (2)	0.21529 (12)	0.0552 (6)	
H7	0.4713	0.2483	0.2154	0.066*	
C8	0.19219 (19)	−0.0393 (2)	0.09975 (11)	0.0444 (5)	
H8	0.1533	−0.1305	0.1098	0.053*	
C9	0.2616 (2)	−0.0512 (2)	0.03111 (12)	0.0507 (6)	
C10	0.2559 (2)	0.0529 (3)	−0.01647 (11)	0.0528 (6)	
C11	0.08375 (19)	0.1693 (2)	0.04492 (10)	0.0445 (5)	
C12	0.08624 (18)	0.0706 (2)	0.09551 (10)	0.0425 (5)	
C13	−0.0033 (2)	0.0607 (2)	0.15302 (12)	0.0490 (5)	
C14	−0.1644 (3)	0.1750 (3)	0.21848 (15)	0.0738 (8)	

H14A	−0.1152	0.1750	0.2621	0.089*	
H14B	−0.2190	0.0925	0.2175	0.089*	
C15	−0.2415 (3)	0.3012 (3)	0.2130 (2)	0.0980 (11)	
H15A	−0.1871	0.3823	0.2168	0.147*	
H15B	−0.3019	0.3025	0.2498	0.147*	
H15C	−0.2864	0.3024	0.1687	0.147*	
C16	−0.0089 (2)	0.2888 (3)	0.03443 (13)	0.0586 (6)	
H16A	−0.0883	0.2659	0.0557	0.088*	
H16B	−0.0235	0.3045	−0.0146	0.088*	
H16C	0.0261	0.3724	0.0555	0.088*	
C17	0.3287 (3)	0.0650 (4)	−0.08253 (13)	0.0756 (8)	
H17A	0.4001	0.1272	−0.0752	0.113*	
H17B	0.2735	0.1017	−0.1190	0.113*	
H17C	0.3592	−0.0261	−0.0958	0.113*	
C18	0.3377 (2)	−0.1776 (3)	0.01887 (15)	0.0676 (7)	
C19	0.3950 (6)	−0.3990 (5)	0.0605 (3)	0.158 (2)	
H19A	0.3515	−0.4556	0.0251	0.190*	0.669 (14)
H19B	0.4793	−0.3765	0.0437	0.190*	0.669 (14)
H19C	0.4473	−0.3909	0.0196	0.190*	0.331 (14)
H19D	0.4514	−0.4147	0.1007	0.190*	0.331 (14)
C20A	0.4079 (13)	−0.4759 (9)	0.1207 (5)	0.185 (6)	0.669 (14)
H20A	0.4549	−0.5603	0.1116	0.278*	0.669 (14)
H20B	0.3248	−0.4999	0.1371	0.278*	0.669 (14)
H20C	0.4530	−0.4213	0.1556	0.278*	0.669 (14)
C20B	0.3114 (12)	−0.5133 (12)	0.0522 (12)	0.141 (9)	0.331 (14)
H20D	0.3600	−0.5980	0.0467	0.212*	0.331 (14)
H20E	0.2573	−0.4988	0.0116	0.212*	0.331 (14)
H20F	0.2598	−0.5213	0.0927	0.212*	0.331 (14)
N1	0.6066 (2)	0.1329 (3)	0.37179 (12)	0.0765 (7)	
N2	0.17490 (18)	0.1647 (2)	−0.00618 (9)	0.0534 (5)	
H2	0.1813	0.2363	−0.0333	0.064*	
O1	0.4059 (2)	−0.2026 (3)	−0.02939 (13)	0.1062 (8)	
O2	0.3246 (2)	−0.2705 (2)	0.06982 (13)	0.0967 (7)	
O3	−0.07949 (16)	0.17231 (17)	0.16008 (9)	0.0641 (5)	
O4	−0.00600 (19)	−0.0369 (2)	0.19268 (11)	0.0824 (6)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0631 (14)	0.0625 (15)	0.0467 (13)	0.0055 (12)	−0.0004 (11)	−0.0063 (11)
C2	0.0482 (12)	0.0555 (13)	0.0413 (11)	0.0057 (10)	−0.0013 (9)	−0.0061 (9)
C3	0.0722 (15)	0.0591 (15)	0.0503 (13)	−0.0007 (12)	−0.0094 (12)	0.0147 (11)
C4	0.0642 (14)	0.0456 (13)	0.0617 (14)	−0.0084 (11)	−0.0080 (11)	0.0095 (10)
C5	0.0410 (10)	0.0396 (11)	0.0452 (11)	0.0051 (9)	0.0025 (8)	−0.0017 (8)
C6	0.0648 (14)	0.0462 (13)	0.0489 (12)	−0.0052 (11)	−0.0103 (11)	0.0075 (10)
C7	0.0603 (13)	0.0466 (12)	0.0582 (13)	−0.0064 (11)	−0.0068 (11)	−0.0006 (10)
C8	0.0446 (11)	0.0371 (11)	0.0513 (12)	−0.0006 (9)	−0.0025 (9)	−0.0032 (9)
C9	0.0453 (12)	0.0539 (13)	0.0525 (12)	0.0024 (10)	−0.0052 (10)	−0.0144 (10)

C10	0.0461 (12)	0.0658 (15)	0.0464 (12)	−0.0015 (11)	−0.0022 (9)	−0.0127 (11)
C11	0.0433 (11)	0.0461 (12)	0.0436 (11)	0.0011 (9)	−0.0056 (9)	−0.0029 (9)
C12	0.0398 (11)	0.0418 (11)	0.0456 (11)	−0.0011 (9)	−0.0029 (9)	−0.0042 (9)
C13	0.0465 (12)	0.0448 (12)	0.0557 (12)	−0.0004 (10)	0.0026 (10)	0.0015 (10)
C14	0.0735 (17)	0.0753 (18)	0.0740 (17)	0.0031 (14)	0.0293 (14)	−0.0027 (14)
C15	0.088 (2)	0.074 (2)	0.135 (3)	0.0022 (16)	0.058 (2)	−0.0069 (19)
C16	0.0624 (14)	0.0569 (14)	0.0562 (13)	0.0106 (11)	−0.0044 (11)	0.0070 (11)
C17	0.0687 (16)	0.106 (2)	0.0526 (14)	0.0028 (15)	0.0107 (12)	−0.0091 (14)
C18	0.0628 (15)	0.0681 (17)	0.0715 (17)	0.0159 (13)	−0.0080 (13)	−0.0218 (14)
C19	0.203 (5)	0.090 (3)	0.183 (5)	0.084 (4)	0.027 (4)	−0.008 (3)
C20A	0.279 (14)	0.093 (5)	0.186 (9)	0.083 (7)	0.039 (9)	0.046 (6)
C20B	0.128 (12)	0.070 (9)	0.23 (2)	0.017 (7)	0.035 (12)	0.030 (10)
N1	0.0915 (16)	0.0847 (16)	0.0522 (12)	−0.0014 (13)	−0.0187 (12)	−0.0122 (11)
N2	0.0585 (11)	0.0569 (11)	0.0450 (10)	0.0036 (9)	0.0028 (8)	0.0062 (8)
O1	0.1042 (16)	0.1076 (18)	0.1084 (17)	0.0390 (14)	0.0303 (14)	−0.0284 (14)
O2	0.1248 (18)	0.0670 (13)	0.0989 (16)	0.0474 (13)	0.0125 (13)	−0.0023 (12)
O3	0.0684 (10)	0.0557 (10)	0.0694 (11)	0.0101 (8)	0.0264 (9)	0.0053 (8)
O4	0.0811 (13)	0.0715 (12)	0.0963 (14)	0.0160 (10)	0.0341 (11)	0.0318 (11)

Geometric parameters (Å, º)

C1—N1	1.137 (3)	C14—H14A	0.9700
C1—C2	1.444 (3)	C14—H14B	0.9700
C2—C7	1.380 (3)	C15—H15A	0.9600
C2—C3	1.383 (3)	C15—H15B	0.9600
C3—C4	1.377 (3)	C15—H15C	0.9600
C3—H3	0.9300	C16—H16A	0.9600
C4—C5	1.380 (3)	C16—H16B	0.9600
C4—H4	0.9300	C16—H16C	0.9600
C5—C6	1.378 (3)	C17—H17A	0.9600
C5—C8	1.533 (3)	C17—H17B	0.9600
C6—C7	1.377 (3)	C17—H17C	0.9600
C6—H6	0.9300	C18—O1	1.206 (3)
C7—H7	0.9300	C18—O2	1.326 (4)
C8—C9	1.523 (3)	C19—C20A	1.370 (8)
C8—C12	1.524 (3)	C19—C20B	1.401 (9)
C8—H8	0.9800	C19—O2	1.440 (4)
C9—C10	1.346 (3)	C19—H19A	0.9700
C9—C18	1.464 (3)	C19—H19B	0.9700
C10—N2	1.377 (3)	C19—H19C	0.9700
C10—C17	1.499 (3)	C19—H19D	0.9700
C11—C12	1.349 (3)	C20A—H20A	0.9600
C11—N2	1.385 (3)	C20A—H20B	0.9600
C11—C16	1.504 (3)	C20A—H20C	0.9600
C12—C13	1.467 (3)	C20B—H20D	0.9600
C13—O4	1.201 (3)	C20B—H20E	0.9600
C13—O3	1.336 (3)	C20B—H20F	0.9600
C14—O3	1.447 (3)	N2—H2	0.8600

C14—C15	1.448 (4)		
N1—C1—C2	178.1 (3)	H15B—C15—H15C	109.5
C7—C2—C3	119.8 (2)	C11—C16—H16A	109.5
C7—C2—C1	120.1 (2)	C11—C16—H16B	109.5
C3—C2—C1	120.1 (2)	H16A—C16—H16B	109.5
C4—C3—C2	119.9 (2)	C11—C16—H16C	109.5
C4—C3—H3	120.0	H16A—C16—H16C	109.5
C2—C3—H3	120.0	H16B—C16—H16C	109.5
C3—C4—C5	120.9 (2)	C10—C17—H17A	109.5
C3—C4—H4	119.5	C10—C17—H17B	109.5
C5—C4—H4	119.5	H17A—C17—H17B	109.5
C6—C5—C4	118.5 (2)	C10—C17—H17C	109.5
C6—C5—C8	120.27 (18)	H17A—C17—H17C	109.5
C4—C5—C8	121.25 (19)	H17B—C17—H17C	109.5
C7—C6—C5	121.4 (2)	O1—C18—O2	120.5 (3)
C7—C6—H6	119.3	O1—C18—C9	128.2 (3)
C5—C6—H6	119.3	O2—C18—C9	111.2 (2)
C6—C7—C2	119.5 (2)	C20A—C19—C20B	74.2 (9)
C6—C7—H7	120.3	C20A—C19—O2	112.8 (5)
C2—C7—H7	120.3	C20B—C19—O2	110.7 (7)
C9—C8—C12	111.57 (17)	C20A—C19—H19A	109.0
C9—C8—C5	110.82 (16)	O2—C19—H19A	109.0
C12—C8—C5	109.63 (16)	C20A—C19—H19B	109.0
C9—C8—H8	108.2	C20B—C19—H19B	134.7
C12—C8—H8	108.2	O2—C19—H19B	109.0
C5—C8—H8	108.2	H19A—C19—H19B	107.8
C10—C9—C18	120.7 (2)	C20A—C19—H19C	132.7
C10—C9—C8	121.06 (19)	C20B—C19—H19C	109.5
C18—C9—C8	118.2 (2)	O2—C19—H19C	109.5
C9—C10—N2	119.2 (2)	H19A—C19—H19C	75.2
C9—C10—C17	127.9 (2)	C20B—C19—H19D	109.5
N2—C10—C17	112.9 (2)	O2—C19—H19D	109.5
C12—C11—N2	119.03 (18)	H19A—C19—H19D	137.2
C12—C11—C16	128.49 (19)	H19B—C19—H19D	76.0
N2—C11—C16	112.47 (18)	H19C—C19—H19D	108.1
C11—C12—C13	125.70 (19)	C19—C20A—H20A	109.5
C11—C12—C8	121.06 (18)	C19—C20A—H20B	109.5
C13—C12—C8	113.16 (18)	H20A—C20A—H20B	109.5
O4—C13—O3	121.7 (2)	C19—C20A—H20C	109.5
O4—C13—C12	123.4 (2)	H20A—C20A—H20C	109.5
O3—C13—C12	114.78 (19)	H20B—C20A—H20C	109.5
O3—C14—C15	108.1 (2)	C19—C20B—H20D	109.5
O3—C14—H14A	110.1	C19—C20B—H20E	109.5
C15—C14—H14A	110.1	H20D—C20B—H20E	109.5
O3—C14—H14B	110.1	C19—C20B—H20F	109.5
C15—C14—H14B	110.1	H20D—C20B—H20F	109.5
H14A—C14—H14B	108.4	H20E—C20B—H20F	109.5

C14—C15—H15A	109.5	C10—N2—C11	124.26 (19)
C14—C15—H15B	109.5	C10—N2—H2	117.9
H15A—C15—H15B	109.5	C11—N2—H2	117.9
C14—C15—H15C	109.5	C18—O2—C19	114.2 (3)
H15A—C15—H15C	109.5	C13—O3—C14	118.16 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N1^{i}	0.86	2.32	3.098 (3)	150

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