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ISSN 2414-3146

Methyl 2-phenylquinoline-4-carboxylate

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Received 20 September 2016
Accepted 22 September 2016

Edited by E. R. T. Tiekink, Sunway University, Malaysia

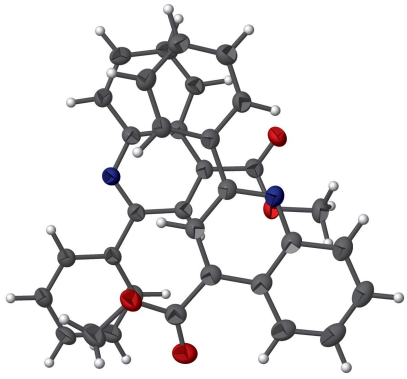
Keywords: crystal structure; quinoline; π–π stacking; cincophene.

CCDC reference: 1505747

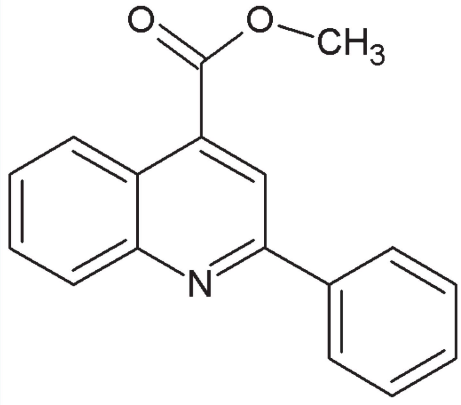
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $C_{17}H_{13}NO_2$, contains two independent molecules which differ primarily in the rotational orientation of the pendant phenyl group, being conrotatory, with respect to the plane of the quinoline moiety. In the crystal, the molecules form stacks parallel to the *b* axis through π–π stacking interactions of the heterocyclic rings [shortest inter-centroid distance = 3.5775 (8) Å].

3D view



Chemical scheme



Structure description

Quinoline-4-carboxylic acids have a variety of medicinal effects and are applied as active components in industrial antioxidants (Wang *et al.*, 2009). Cincophene, named 2-phenylquinoline-4-carboxylic acid, has been proven to be a powerful antimicrobial agent (Wadher *et al.*, 2009). Cinchophen has been used as an antirheumatic agent for more than 60 years (Abd El-Aal & El-Emary, 2014). Moreover, cinchophen is shown to exert remarkable biological activity (Kaila *et al.*, 2007; Deady *et al.*, 2000) and has been proven to be a powerful analgesic, antimicrobial and antifungal agent (Metwally *et al.*, 2010). As part of our studies in this area, the title compound has been synthesized to be used a precursor for further cincophene derivatives.

The asymmetric unit of the title compound contains two independent molecules, which differ primarily in the rotational orientation of the pendant phenyl group with respect to the plane of the quinoline moiety, Fig. 1. The dihedral angle between the N1/C1/C6–C9 and C12–C17 planes is 17.00 (6)° while that between the N2/C18/C23–C26 and C29–C34 planes is 16.63 (7)° in the opposite direction. Each asymmetric unit interacts with the one above it and one below it along the *b*-axis direction through π–π stacking of the

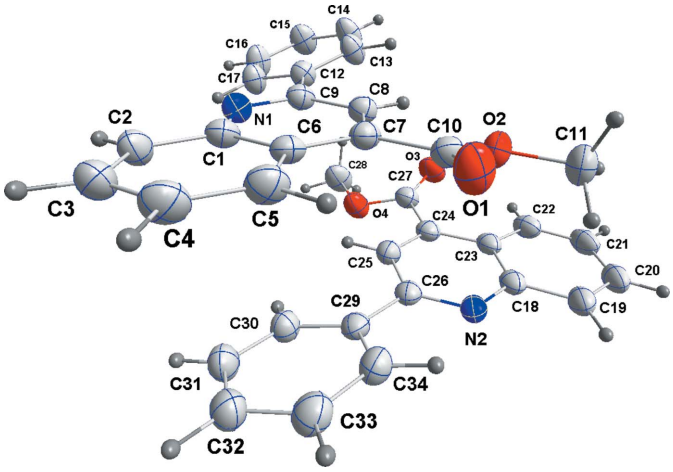


Figure 1
The asymmetric unit with labeling scheme and 50% probability ellipsoids.

heterocyclic rings, Fig. 2. In these, the distance between the centers of gravity of the heterocycles is 3.5775 (8) Å and the dihedral angle between the the rings is 4.59 (7)°.

Synthesis and crystallization

A mixture of 2-phenylquinoline-4-carboxylic acid (0.03 mol), methanol (20 ml) and concentrated sulfuric acid (3 ml) was refluxed for 6 h, then and cooled to room temperature. Sodium carbonate solution was added to neutralize the mixture which was then left to stand for 1 h at room temperature. Pale-yellow crystals were precipitated and washed with distilled water then recrystallized from ethanol solution to afford the title compound in 50% yield; m.p. 332 K.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

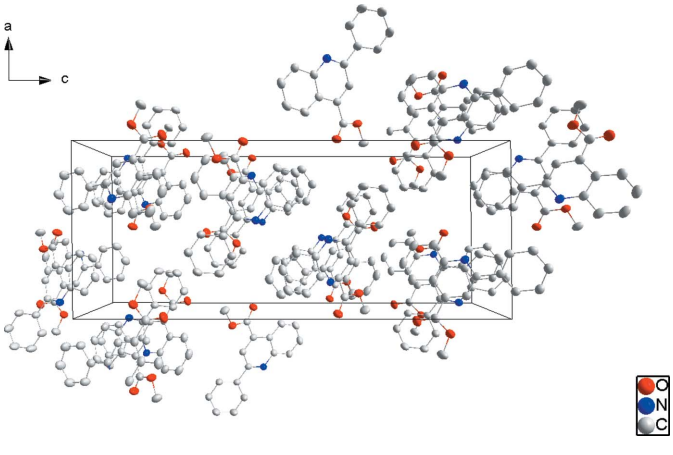


Figure 2
Packing viewed along the *b* axis.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{13}NO_2$
M_r	263.28
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	150
a, b, c (Å)	11.5383 (7), 7.9873 (5), 28.3149 (19)
β (°)	90.525 (3)
V (Å^3)	2609.4 (3)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.71
Crystal size (mm)	0.23 × 0.18 × 0.02
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.90, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19441, 5118, 4152
R_{int}	0.043
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.114, 1.04
No. of reflections	5118
No. of parameters	466
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.18

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Acknowledgements

The support of NSF–MRI Grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

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full crystallographic data

IUCrData (2016). **1**, x161500 [https://doi.org/10.1107/S2414314616015005]

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Crystal data

$C_{17}H_{13}NO_2$
M_r = 263.28
Monoclinic, $P2_1/n$
a = 11.5383 (7) Å
b = 7.9873 (5) Å
c = 28.3149 (19) Å
β = 90.525 (3)°
V = 2609.4 (3) Å^3
Z = 8
$F(000)$ = 1104
D_x = 1.340 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9981 reflections
θ = 4.1–72.4°
μ = 0.71 mm^{-1}
T = 150 K
Plate, pale-yellow
0.23 × 0.18 × 0.02 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.90, T_{max} = 0.99
19441 measured reflections
5118 independent reflections
4152 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 72.4°, θ_{min} = 3.1°
h = −12→14
k = −9→9
l = −32→34

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.114
S = 1.04
5118 reflections
466 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.6267P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00105 (15)

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.01888 (10)	0.89113 (17)	0.60650 (4)	0.0478 (3)
O2	0.12589 (9)	0.75224 (14)	0.65997 (4)	0.0379 (3)
H2	0.4531 (18)	1.185 (2)	0.5026 (7)	0.051 (5)*
N1	0.44689 (11)	1.04777 (16)	0.58249 (4)	0.0332 (3)
C1	0.35355 (13)	1.06996 (18)	0.55306 (5)	0.0314 (3)
C2	0.37379 (14)	1.1544 (2)	0.50991 (5)	0.0360 (3)
C3	0.28477 (15)	1.1895 (2)	0.47961 (6)	0.0403 (4)
H3	0.3011 (16)	1.253 (2)	0.4509 (7)	0.047 (5)*
C4	0.17146 (15)	1.1382 (2)	0.49094 (6)	0.0414 (4)
H4	0.1074 (17)	1.166 (2)	0.4676 (7)	0.050 (5)*
C5	0.14937 (14)	1.0531 (2)	0.53144 (6)	0.0373 (4)
H5	0.0658 (17)	1.015 (2)	0.5386 (7)	0.049 (5)*
C6	0.23973 (13)	1.01572 (17)	0.56423 (5)	0.0299 (3)
C7	0.22671 (12)	0.92727 (18)	0.60774 (5)	0.0300 (3)
C8	0.32219 (13)	0.90581 (18)	0.63663 (5)	0.0309 (3)
H8	0.3147 (12)	0.8488 (18)	0.6653 (5)	0.016 (3)*
C9	0.43156 (12)	0.97042 (18)	0.62335 (5)	0.0302 (3)
C10	0.11265 (13)	0.85839 (19)	0.62339 (5)	0.0333 (3)
C11	0.02004 (16)	0.6797 (2)	0.67742 (7)	0.0460 (4)
H11A	−0.0240 (18)	0.625 (3)	0.6503 (8)	0.057 (6)*
H11B	0.0484 (19)	0.587 (3)	0.7019 (8)	0.061 (6)*
H11C	−0.0297 (18)	0.768 (3)	0.6918 (7)	0.061 (6)*
C12	0.53541 (12)	0.95654 (19)	0.65488 (5)	0.0321 (3)
C13	0.52674 (14)	0.9197 (2)	0.70269 (6)	0.0398 (4)
H13	0.4511 (17)	0.901 (2)	0.7169 (7)	0.046 (5)*
C14	0.62524 (15)	0.9116 (3)	0.73115 (6)	0.0471 (4)
H14	0.6190 (17)	0.885 (3)	0.7639 (7)	0.055 (6)*
C15	0.73415 (14)	0.9400 (3)	0.71248 (6)	0.0461 (4)
H15	0.8035 (18)	0.937 (3)	0.7324 (7)	0.057 (6)*
C16	0.74342 (14)	0.9749 (3)	0.66476 (6)	0.0457 (4)
H16	0.8198 (18)	0.993 (2)	0.6509 (7)	0.051 (5)*
C17	0.64574 (14)	0.9836 (2)	0.63621 (6)	0.0386 (4)
H17	0.6507 (16)	1.007 (2)	0.6023 (7)	0.046 (5)*
O3	0.51904 (11)	0.42341 (18)	0.71200 (4)	0.0516 (3)
O4	0.58364 (10)	0.47874 (18)	0.63946 (4)	0.0487 (3)

N2	0.16967 (11)	0.41399 (15)	0.59871 (4)	0.0322 (3)
C18	0.18457 (13)	0.33694 (18)	0.64132 (5)	0.0327 (3)
C19	0.08718 (15)	0.2508 (2)	0.65977 (6)	0.0394 (4)
H19	0.0144 (17)	0.250 (2)	0.6412 (7)	0.047 (5)*
C20	0.09464 (17)	0.1690 (2)	0.70210 (6)	0.0444 (4)
H20	0.0267 (18)	0.109 (2)	0.7142 (7)	0.052 (5)*
C21	0.19865 (18)	0.1703 (2)	0.72802 (6)	0.0464 (4)
H21	0.2068 (18)	0.111 (3)	0.7576 (8)	0.059 (6)*
C22	0.29470 (16)	0.2526 (2)	0.71136 (6)	0.0415 (4)
H22	0.3667 (16)	0.248 (2)	0.7290 (6)	0.042 (5)*
C23	0.29029 (14)	0.33797 (18)	0.66756 (5)	0.0334 (3)
C24	0.38529 (14)	0.42421 (19)	0.64630 (5)	0.0334 (3)
C25	0.36901 (14)	0.49811 (19)	0.60313 (5)	0.0324 (3)
H25	0.4317 (15)	0.558 (2)	0.5895 (6)	0.035 (4)*
C26	0.25979 (13)	0.49146 (17)	0.57976 (5)	0.0302 (3)
C27	0.50072 (14)	0.4400 (2)	0.67028 (5)	0.0373 (4)
C28	0.69816 (17)	0.5010 (4)	0.65908 (8)	0.0582 (5)
H28A	0.7213 (19)	0.395 (3)	0.6728 (8)	0.063 (7)*
H28B	0.6998 (19)	0.594 (3)	0.6840 (8)	0.062 (6)*
H28C	0.746 (2)	0.526 (3)	0.6318 (9)	0.069 (7)*
C29	0.24158 (13)	0.57273 (18)	0.53317 (5)	0.0306 (3)
C30	0.33512 (14)	0.6189 (2)	0.50488 (5)	0.0363 (3)
H30	0.4153 (16)	0.594 (2)	0.5146 (6)	0.039 (5)*
C31	0.31678 (15)	0.6928 (2)	0.46116 (6)	0.0422 (4)
H31	0.3842 (17)	0.723 (3)	0.4430 (7)	0.053 (5)*
C32	0.20487 (15)	0.7204 (2)	0.44489 (6)	0.0426 (4)
H32	0.1895 (16)	0.773 (2)	0.4136 (7)	0.047 (5)*
C33	0.11146 (15)	0.6745 (2)	0.47229 (6)	0.0420 (4)
H33	0.0322 (18)	0.699 (2)	0.4615 (7)	0.054 (6)*
C34	0.12983 (14)	0.6026 (2)	0.51620 (6)	0.0365 (3)
H34	0.0642 (17)	0.572 (2)	0.5368 (7)	0.049 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0299 (6)	0.0702 (8)	0.0432 (6)	−0.0049 (5)	−0.0045 (5)	0.0056 (6)
O2	0.0313 (6)	0.0396 (6)	0.0430 (6)	−0.0057 (5)	0.0037 (4)	0.0047 (5)
N1	0.0301 (6)	0.0377 (7)	0.0319 (6)	0.0007 (5)	−0.0005 (5)	0.0007 (5)
C1	0.0325 (8)	0.0317 (7)	0.0300 (7)	−0.0002 (6)	−0.0019 (6)	−0.0043 (6)
C2	0.0362 (8)	0.0412 (8)	0.0305 (8)	−0.0042 (7)	0.0015 (6)	0.0008 (6)
C3	0.0499 (10)	0.0406 (9)	0.0302 (8)	−0.0049 (7)	−0.0052 (7)	0.0017 (7)
C4	0.0419 (9)	0.0449 (9)	0.0371 (8)	−0.0039 (7)	−0.0119 (7)	−0.0017 (7)
C5	0.0347 (8)	0.0408 (8)	0.0364 (8)	−0.0036 (7)	−0.0074 (6)	−0.0023 (7)
C6	0.0322 (8)	0.0274 (7)	0.0301 (7)	−0.0008 (6)	−0.0012 (6)	−0.0067 (6)
C7	0.0286 (7)	0.0295 (7)	0.0319 (7)	−0.0014 (6)	0.0016 (6)	−0.0059 (6)
C8	0.0302 (7)	0.0320 (7)	0.0304 (7)	0.0011 (6)	0.0005 (6)	−0.0021 (6)
C9	0.0272 (7)	0.0318 (7)	0.0316 (7)	0.0025 (6)	0.0003 (6)	−0.0033 (6)
C10	0.0306 (8)	0.0361 (8)	0.0332 (8)	−0.0028 (6)	−0.0007 (6)	−0.0066 (6)

C11	0.0368 (9)	0.0495 (10)	0.0518 (10)	−0.0098 (8)	0.0103 (8)	0.0014 (9)
C12	0.0264 (7)	0.0358 (8)	0.0341 (8)	0.0024 (6)	−0.0002 (6)	−0.0004 (6)
C13	0.0264 (8)	0.0590 (10)	0.0340 (8)	0.0013 (7)	0.0018 (6)	0.0007 (7)
C14	0.0339 (9)	0.0746 (13)	0.0328 (8)	0.0065 (8)	−0.0010 (6)	0.0023 (8)
C15	0.0291 (8)	0.0679 (12)	0.0413 (9)	0.0033 (8)	−0.0075 (7)	0.0028 (8)
C16	0.0264 (8)	0.0645 (11)	0.0463 (9)	0.0012 (8)	−0.0003 (7)	0.0099 (8)
C17	0.0300 (8)	0.0493 (9)	0.0365 (8)	0.0018 (7)	0.0013 (6)	0.0066 (7)
O3	0.0511 (7)	0.0717 (9)	0.0320 (6)	−0.0030 (6)	−0.0067 (5)	−0.0028 (6)
O4	0.0350 (6)	0.0750 (9)	0.0360 (6)	−0.0056 (6)	−0.0040 (5)	−0.0025 (6)
N2	0.0334 (7)	0.0307 (6)	0.0326 (6)	−0.0019 (5)	0.0044 (5)	−0.0014 (5)
C18	0.0394 (8)	0.0279 (7)	0.0310 (7)	0.0006 (6)	0.0055 (6)	−0.0038 (6)
C19	0.0428 (9)	0.0369 (8)	0.0388 (8)	−0.0040 (7)	0.0099 (7)	−0.0022 (7)
C20	0.0555 (11)	0.0381 (9)	0.0399 (9)	−0.0059 (8)	0.0161 (8)	−0.0014 (7)
C21	0.0691 (12)	0.0383 (9)	0.0319 (8)	−0.0009 (8)	0.0101 (8)	0.0024 (7)
C22	0.0550 (11)	0.0377 (8)	0.0319 (8)	0.0011 (8)	0.0005 (7)	−0.0013 (7)
C23	0.0429 (9)	0.0283 (7)	0.0289 (7)	0.0005 (6)	0.0043 (6)	−0.0042 (6)
C24	0.0382 (8)	0.0316 (7)	0.0304 (7)	0.0013 (6)	0.0015 (6)	−0.0059 (6)
C25	0.0325 (8)	0.0318 (7)	0.0331 (8)	−0.0011 (6)	0.0022 (6)	−0.0022 (6)
C26	0.0322 (7)	0.0267 (7)	0.0315 (7)	−0.0006 (6)	0.0029 (6)	−0.0038 (6)
C27	0.0391 (9)	0.0398 (8)	0.0328 (8)	0.0010 (7)	−0.0002 (6)	−0.0057 (6)
C28	0.0368 (10)	0.0908 (17)	0.0468 (11)	−0.0066 (10)	−0.0081 (8)	−0.0059 (11)
C29	0.0321 (7)	0.0271 (7)	0.0326 (7)	−0.0025 (6)	0.0005 (6)	−0.0024 (6)
C30	0.0314 (8)	0.0416 (8)	0.0360 (8)	−0.0020 (7)	0.0008 (6)	0.0022 (7)
C31	0.0408 (9)	0.0488 (10)	0.0371 (8)	−0.0065 (7)	0.0040 (7)	0.0059 (7)
C32	0.0448 (9)	0.0450 (9)	0.0378 (8)	−0.0060 (7)	−0.0037 (7)	0.0092 (7)
C33	0.0373 (9)	0.0425 (9)	0.0460 (9)	−0.0033 (7)	−0.0055 (7)	0.0071 (7)
C34	0.0320 (8)	0.0363 (8)	0.0413 (8)	−0.0039 (6)	0.0001 (6)	0.0043 (7)

Geometric parameters (Å, º)

O1—C10	1.2076 (19)	O3—C27	1.206 (2)
O2—C10	1.3461 (19)	O4—C27	1.337 (2)
O2—C11	1.443 (2)	O4—C28	1.440 (2)
N1—C9	1.3249 (19)	N2—C26	1.3274 (19)
N1—C1	1.3672 (19)	N2—C18	1.3639 (19)
C1—C2	1.417 (2)	C18—C19	1.421 (2)
C1—C6	1.421 (2)	C18—C23	1.422 (2)
C2—C3	1.361 (2)	C19—C20	1.367 (2)
C2—H2	0.97 (2)	C19—H19	0.986 (19)
C3—C4	1.410 (2)	C20—C21	1.401 (3)
C3—H3	0.98 (2)	C20—H20	0.98 (2)
C4—C5	1.359 (2)	C21—C22	1.375 (3)
C4—H4	1.013 (19)	C21—H21	0.97 (2)
C5—C6	1.421 (2)	C22—C23	1.416 (2)
C5—H5	1.03 (2)	C22—H22	0.966 (19)
C6—C7	1.429 (2)	C23—C24	1.432 (2)
C7—C8	1.377 (2)	C24—C25	1.369 (2)
C7—C10	1.497 (2)	C24—C27	1.495 (2)

C8—C9	1.417 (2)	C25—C26	1.419 (2)
C8—H8	0.936 (14)	C25—H25	0.953 (18)
C9—C12	1.4918 (19)	C26—C29	1.484 (2)
C11—H11A	1.01 (2)	C28—H28A	0.97 (2)
C11—H11B	1.06 (2)	C28—H28B	1.03 (2)
C11—H11C	1.00 (2)	C28—H28C	0.97 (2)
C12—C13	1.390 (2)	C29—C34	1.393 (2)
C12—C17	1.400 (2)	C29—C30	1.399 (2)
C13—C14	1.389 (2)	C30—C31	1.386 (2)
C13—H13	0.98 (2)	C30—H30	0.984 (18)
C14—C15	1.386 (3)	C31—C32	1.385 (2)
C14—H14	0.95 (2)	C31—H31	0.97 (2)
C15—C16	1.385 (3)	C32—C33	1.384 (2)
C15—H15	0.98 (2)	C32—H32	1.00 (2)
C16—C17	1.383 (2)	C33—C34	1.384 (2)
C16—H16	0.98 (2)	C33—H33	0.98 (2)
C17—H17	0.98 (2)	C34—H34	0.99 (2)

C10—O2—C11	115.21 (13)	C27—O4—C28	115.87 (14)
C9—N1—C1	118.81 (13)	C26—N2—C18	118.36 (13)
N1—C1—C2	116.88 (13)	N2—C18—C19	116.74 (14)
N1—C1—C6	123.31 (13)	N2—C18—C23	124.05 (14)
C2—C1—C6	119.80 (14)	C19—C18—C23	119.21 (14)
C3—C2—C1	120.80 (15)	C20—C19—C18	120.67 (17)
C3—C2—H2	121.3 (12)	C20—C19—H19	120.8 (11)
C1—C2—H2	117.9 (12)	C18—C19—H19	118.6 (11)
C2—C3—C4	119.52 (15)	C19—C20—C21	120.16 (16)
C2—C3—H3	118.7 (11)	C19—C20—H20	119.6 (12)
C4—C3—H3	121.7 (11)	C21—C20—H20	120.2 (12)
C5—C4—C3	121.25 (15)	C22—C21—C20	120.87 (16)
C5—C4—H4	121.4 (11)	C22—C21—H21	117.3 (13)
C3—C4—H4	117.4 (11)	C20—C21—H21	121.8 (13)
C4—C5—C6	120.93 (15)	C21—C22—C23	120.51 (17)
C4—C5—H5	119.7 (11)	C21—C22—H22	119.7 (11)
C6—C5—H5	119.4 (11)	C23—C22—H22	119.7 (11)
C1—C6—C5	117.67 (14)	C22—C23—C18	118.59 (15)
C1—C6—C7	116.56 (13)	C22—C23—C24	125.34 (15)
C5—C6—C7	125.78 (14)	C18—C23—C24	116.06 (14)
C8—C7—C6	118.94 (13)	C25—C24—C23	118.92 (14)
C8—C7—C10	118.63 (13)	C25—C24—C27	118.93 (14)
C6—C7—C10	122.43 (13)	C23—C24—C27	122.13 (14)
C7—C8—C9	120.43 (14)	C24—C25—C26	120.95 (14)
C7—C8—H8	119.8 (9)	C24—C25—H25	118.7 (11)
C9—C8—H8	119.8 (8)	C26—C25—H25	120.4 (10)
N1—C9—C8	121.86 (13)	N2—C26—C25	121.64 (14)
N1—C9—C12	116.38 (13)	N2—C26—C29	117.28 (13)
C8—C9—C12	121.76 (13)	C25—C26—C29	121.07 (13)
O1—C10—O2	122.42 (14)	O3—C27—O4	123.01 (15)

O1—C10—C7	126.16 (15)	O3—C27—C24	125.70 (15)
O2—C10—C7	111.42 (12)	O4—C27—C24	111.28 (13)
O2—C11—H11A	109.4 (12)	O4—C28—H28A	107.2 (14)
O2—C11—H11B	104.2 (12)	O4—C28—H28B	111.5 (12)
H11A—C11—H11B	110.2 (16)	H28A—C28—H28B	110.8 (18)
O2—C11—H11C	110.4 (12)	O4—C28—H28C	104.1 (14)
H11A—C11—H11C	109.0 (17)	H28A—C28—H28C	110.0 (19)
H11B—C11—H11C	113.5 (17)	H28B—C28—H28C	112.9 (19)
C13—C12—C17	118.32 (14)	C34—C29—C30	118.29 (14)
C13—C12—C9	122.28 (13)	C34—C29—C26	120.33 (13)
C17—C12—C9	119.40 (14)	C30—C29—C26	121.38 (14)
C14—C13—C12	120.61 (15)	C31—C30—C29	120.74 (15)
C14—C13—H13	118.9 (11)	C31—C30—H30	118.3 (10)
C12—C13—H13	120.5 (11)	C29—C30—H30	120.9 (10)
C15—C14—C13	120.73 (16)	C32—C31—C30	119.95 (15)
C15—C14—H14	118.9 (12)	C32—C31—H31	122.3 (12)
C13—C14—H14	120.4 (12)	C30—C31—H31	117.8 (12)
C16—C15—C14	118.91 (15)	C33—C32—C31	120.00 (16)
C16—C15—H15	119.9 (12)	C33—C32—H32	118.6 (11)
C14—C15—H15	121.2 (12)	C31—C32—H32	121.4 (11)
C17—C16—C15	120.71 (16)	C32—C33—C34	120.02 (16)
C17—C16—H16	119.3 (12)	C32—C33—H33	120.0 (12)
C15—C16—H16	120.0 (12)	C34—C33—H33	119.9 (12)
C16—C17—C12	120.71 (15)	C33—C34—C29	120.99 (15)
C16—C17—H17	121.9 (11)	C33—C34—H34	121.3 (11)
C12—C17—H17	117.4 (11)	C29—C34—H34	117.7 (11)

C9—N1—C1—C2	179.55 (13)	C26—N2—C18—C19	−178.05 (13)
C9—N1—C1—C6	0.7 (2)	C26—N2—C18—C23	1.8 (2)
N1—C1—C2—C3	−176.61 (15)	N2—C18—C19—C20	179.50 (15)
C6—C1—C2—C3	2.3 (2)	C23—C18—C19—C20	−0.3 (2)
C1—C2—C3—C4	−1.3 (3)	C18—C19—C20—C21	0.5 (3)
C2—C3—C4—C5	−0.5 (3)	C19—C20—C21—C22	−0.4 (3)
C3—C4—C5—C6	1.2 (3)	C20—C21—C22—C23	0.1 (3)
N1—C1—C6—C5	177.27 (14)	C21—C22—C23—C18	0.1 (2)
C2—C1—C6—C5	−1.5 (2)	C21—C22—C23—C24	−178.48 (15)
N1—C1—C6—C7	−2.9 (2)	N2—C18—C23—C22	−179.77 (14)
C2—C1—C6—C7	178.26 (13)	C19—C18—C23—C22	0.0 (2)
C4—C5—C6—C1	−0.2 (2)	N2—C18—C23—C24	−1.1 (2)
C4—C5—C6—C7	−179.93 (15)	C19—C18—C23—C24	178.72 (13)
C1—C6—C7—C8	2.5 (2)	C22—C23—C24—C25	178.47 (15)
C5—C6—C7—C8	−177.74 (14)	C18—C23—C24—C25	−0.1 (2)
C1—C6—C7—C10	−178.17 (13)	C22—C23—C24—C27	−3.2 (2)
C5—C6—C7—C10	1.6 (2)	C18—C23—C24—C27	178.24 (13)
C6—C7—C8—C9	0.0 (2)	C23—C24—C25—C26	0.6 (2)
C10—C7—C8—C9	−179.41 (13)	C27—C24—C25—C26	−177.81 (13)
C1—N1—C9—C8	2.0 (2)	C18—N2—C26—C25	−1.2 (2)
C1—N1—C9—C12	−177.40 (13)	C18—N2—C26—C29	179.38 (12)

C7—C8—C9—N1	−2.3 (2)	C24—C25—C26—N2	0.1 (2)
C7—C8—C9—C12	177.00 (13)	C24—C25—C26—C29	179.45 (13)
C11—O2—C10—O1	0.3 (2)	C28—O4—C27—O3	−0.3 (3)
C11—O2—C10—C7	179.97 (13)	C28—O4—C27—C24	178.53 (16)
C8—C7—C10—O1	166.36 (15)	C25—C24—C27—O3	158.55 (17)
C6—C7—C10—O1	−13.0 (2)	C23—C24—C27—O3	−19.8 (3)
C8—C7—C10—O2	−13.33 (19)	C25—C24—C27—O4	−20.3 (2)
C6—C7—C10—O2	167.33 (13)	C23—C24—C27—O4	161.37 (14)
N1—C9—C12—C13	162.76 (15)	N2—C26—C29—C34	15.8 (2)
C8—C9—C12—C13	−16.6 (2)	C25—C26—C29—C34	−163.66 (14)
N1—C9—C12—C17	−16.3 (2)	N2—C26—C29—C30	−163.24 (14)
C8—C9—C12—C17	164.28 (15)	C25—C26—C29—C30	17.3 (2)
C17—C12—C13—C14	0.6 (3)	C34—C29—C30—C31	0.2 (2)
C9—C12—C13—C14	−178.52 (16)	C26—C29—C30—C31	179.19 (15)
C12—C13—C14—C15	0.0 (3)	C29—C30—C31—C32	−0.6 (3)
C13—C14—C15—C16	−0.7 (3)	C30—C31—C32—C33	0.1 (3)
C14—C15—C16—C17	0.9 (3)	C31—C32—C33—C34	0.7 (3)
C15—C16—C17—C12	−0.3 (3)	C32—C33—C34—C29	−1.1 (3)
C13—C12—C17—C16	−0.4 (3)	C30—C29—C34—C33	0.7 (2)
C9—C12—C17—C16	178.69 (16)	C26—C29—C34—C33	−178.36 (15)