Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Acetyl-5-phenyl-1-*p*-tolyl-1*H*-pyrazole-4-carbonitrile

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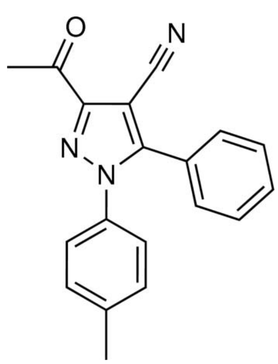
Received 16 March 2012; accepted 19 March 2012

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.118; data-to-parameter ratio = 12.8.

In the title pyrazole derivative, $C_{19}H_{15}N_3O$, the central pyrazole ring makes dihedral angles of 42.71 (9) and 61.34 (9)°, respectively, with the phenyl and *p*-tolyl rings. The dihedral angle between the phenyl and *p*-tolyl rings is 58.22 (9)°. The 3-acetyl-1*H*-pyrazole-4-carbonitrile unit is essentially planar, with an r.m.s. deviation of 0.0295 (1) Å for the ten non-H atoms.

Related literature

For bond-length data, see: Allen *et al.* (1987). For background to and the bioactivity of pyrazole derivatives, see: Abdel-Aziz *et al.* (2009, 2010); Abdel-Wahab *et al.* (2009); Dawood *et al.* (2003). For a related structure, see: Abdel-Aziz *et al.* (2012).



Experimental

Crystal data

$C_{19}H_{15}N_3O$
M_r = 301.34
Monoclinic, $P2_1/c$
a = 10.2433 (2) Å
b = 10.6467 (2) Å
c = 15.7547 (3) Å
β = 109.684 (1)°
V = 1617.76 (5) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.63 mm^{-1}
T = 296 K
0.57 × 0.28 × 0.22 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.718, T_{max} = 0.876
10344 measured reflections
2720 independent reflections
2427 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.118
S = 1.05
2720 reflections
213 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Deanship of Scientific Research and the Research Center, College of Pharmacy, King Saud University, and Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160. HKF also thanks the King Saud University, Riyadh, Saudi Arabia, for the award of a visiting Professorship (December 23rd 2011 to January 14th 2012).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5093).

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supporting information

Acta Cryst. (2012). E**68**, o1167   [https://doi.org/10.1107/S1600536812011762]

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S1. Comment

During the course of our medicinal chemistry research on pyrazole derivatives (Abdel-Aziz *et al.*, 2009, 2010; Abdel-Wahab *et al.*, 2009), we previously reported the crystal structure of 3-acetyl-1,5-diphenyl-1*H*-pyrazole-4-carbonitrile (I) (Abdel-Aziz *et al.*, 2012). The title compound (II), was synthesized by retaining the core part but changing the phenyl group which was attached to N atom at position 1 of the pyrazole ring in compound (I) to the *p*-tolyl in order to investigate the influence of the substituents to their biological properties. Herein, the crystal structure of (II) was reported.

The molecule of (II), $C_{19}H_{15}N_3O$, has the same butterfly-like structure as in (I) (Abdel-Aziz *et al.*, 2012). However there are differences in the dihedral angles between the equivalent moieties and the crystal packing of (I) and (II). In (II), the pyrazole ring forms dihedral angles of 42.71 (9) and 61.34 (9)°, respectively, with the C5–C10 and C11-C16 benzene rings [the corresponding values in (I) are 59.31 (8) and 57.24 (8)°] and the dihedral angle between these two benzene rings is 58.22 (9)° [the corresponding value in (I) is 64.03 (8)°]. The cabonitrile and acetyl substituents in (II) lie essentially on the same plane with the pyrazole ring with the *r.m.s.* 0.0295 (1) Å for the ten non H atoms (C1–C4/C17/C18/N1–N3/O1) and the dihedral angle between the C-C=O planes of the acetyl unit and pyrazole ring is 4.8 (2)° [whereas in (I) the acetyl moiety is slightly deviated from the pyrazole ring with the dihedral angle between the C-C=O planes of the acetyl and pyrazole moieties being 7.95 (18)°]. The bond distances in (II) are within normal ranges (Allen *et al.*, 1987) and are comparable to the closely related structure (Abdel-Aziz *et al.*, 2012). The crystal packing of (II) is stabilized by van der Waals interactions. Even there is no hydrogen bonds, the crystal packing of (II) was shown in Fig. 2 for comparison with that of (I).

S2. Experimental

The title compound was prepared according to the reported method (Dawood *et al.*, 2003). Single crystals of the title compound suitable for X-ray structure determination were recrystallized from ethanol by the slow evaporation of the solvent at room temperature after several days.

S3. Refinement

All H atoms were placed in calculated positions with C—H = 0.93 Å for aromatic and 0.96 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

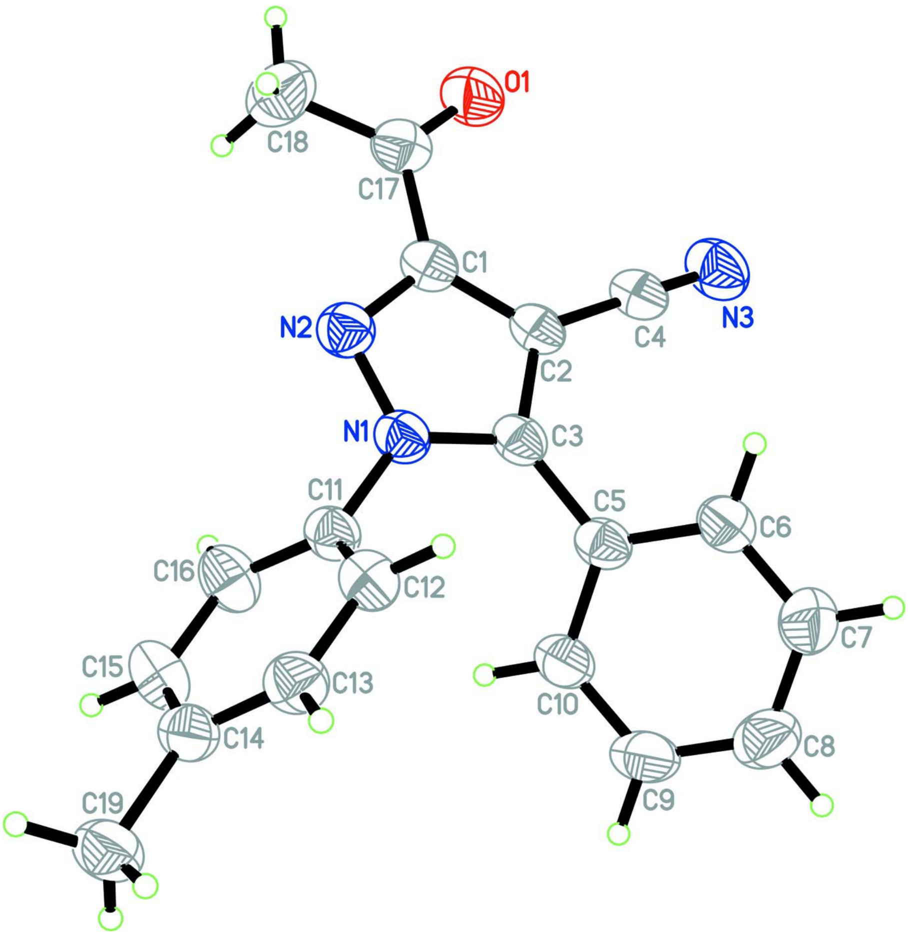

Figure 1

The structure of the title compound, showing 40% probability displacement ellipsoids and the atom-numbering scheme.

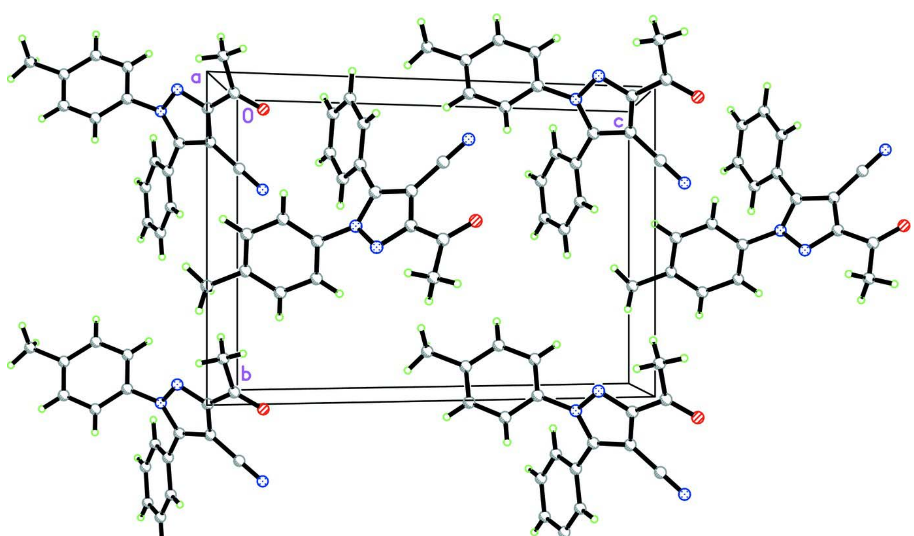

Figure 2

A packing diagram of the title compound viewed along the *a* axis.

3-Acetyl-5-phenyl-1-*p*-tolyl-1*H*-pyrazole-4-carbonitrile

Crystal data

$C_{19}H_{15}N_3O$
$M_r = 301.34$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.2433$ (2) Å
$b = 10.6467$ (2) Å
$c = 15.7547$ (3) Å
$\beta = 109.684$ (1)°
$V = 1617.76$ (5) Å^3
$Z = 4$
$F(000) = 632$
$D_x = 1.237$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 272 reflections
$\theta = 4.6$–$65.0°$
$\mu = 0.63$ mm^{-1}
$T = 296$ K
Block, colorless
0.57 × 0.28 × 0.22 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.718$, $T_{max} = 0.876$
10344 measured reflections
2720 independent reflections
2427 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 65.0°$, $\theta_{min} = 4.6°$
$h = -12 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.118$
$S = 1.05$
2720 reflections
213 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
$w = 1/[\sigma^2(F_o^2) + (0.050P)^2 + 0.2871P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0113 (9)

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	−0.28761 (14)	0.55281 (14)	0.42141 (9)	0.0837 (4)
N1	0.05513 (13)	0.54275 (12)	0.68539 (8)	0.0536 (3)
N2	−0.05757 (13)	0.47588 (13)	0.63730 (8)	0.0585 (3)
N3	−0.07458 (17)	0.81153 (17)	0.43476 (10)	0.0795 (5)
C1	−0.11442 (15)	0.54122 (15)	0.56191 (10)	0.0544 (4)
C2	−0.03698 (14)	0.65058 (14)	0.56181 (9)	0.0513 (4)
C3	0.07256 (14)	0.64969 (14)	0.64318 (9)	0.0499 (4)
C4	−0.05890 (15)	0.74023 (16)	0.49130 (10)	0.0578 (4)
C5	0.18570 (15)	0.74010 (15)	0.67940 (9)	0.0521 (4)
C6	0.15724 (18)	0.86766 (16)	0.67039 (11)	0.0637 (4)
H6A	0.0669	0.8947	0.6412	0.076*
C7	0.2610 (2)	0.9544 (2)	0.70412 (14)	0.0833 (6)
H7A	0.2405	1.0398	0.6983	0.100*
C8	0.3956 (2)	0.9150 (2)	0.74673 (14)	0.0876 (6)
H8A	0.4659	0.9736	0.7699	0.105*
C9	0.42553 (18)	0.7887 (2)	0.75483 (13)	0.0781 (6)
H9A	0.5165	0.7624	0.7828	0.094*
C10	0.32237 (16)	0.70133 (18)	0.72207 (11)	0.0644 (4)
H10A	0.3435	0.6161	0.7283	0.077*
C11	0.13953 (15)	0.49602 (15)	0.77206 (9)	0.0530 (4)
C12	0.15432 (17)	0.56511 (16)	0.84811 (10)	0.0587 (4)
H12A	0.1096	0.6420	0.8442	0.070*
C13	0.23678 (17)	0.51883 (17)	0.93088 (10)	0.0625 (4)
H13A	0.2470	0.5654	0.9827	0.075*
C14	0.30436 (16)	0.40481 (17)	0.93820 (10)	0.0605 (4)
C15	0.2845 (2)	0.33649 (18)	0.86054 (12)	0.0723 (5)
H15A	0.3274	0.2587	0.8644	0.087*
C16	0.2024 (2)	0.38067 (17)	0.77706 (11)	0.0690 (5)
H16A	0.1900	0.3334	0.7253	0.083*
C17	−0.24206 (18)	0.49704 (17)	0.49213 (11)	0.0641 (4)
C18	−0.3103 (2)	0.3819 (2)	0.51218 (15)	0.0947 (7)
H18A	−0.3925	0.3639	0.4621	0.142*
H18B	−0.3346	0.3959	0.5652	0.142*
H18C	−0.2475	0.3122	0.5222	0.142*
C19	0.39693 (19)	0.3572 (2)	1.02859 (12)	0.0778 (5)
H19C	0.3602	0.2800	1.0425	0.117*
H19A	0.4011	0.4185	1.0741	0.117*
H19B	0.4884	0.3429	1.0266	0.117*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0771 (8)	0.0812 (9)	0.0701 (8)	0.0013 (7)	−0.0049 (6)	0.0061 (7)
N1	0.0510 (7)	0.0597 (7)	0.0486 (6)	0.0035 (5)	0.0147 (5)	0.0065 (5)
N2	0.0554 (7)	0.0637 (8)	0.0545 (7)	0.0002 (6)	0.0162 (6)	0.0032 (6)

N3	0.0781 (10)	0.0908 (11)	0.0680 (9)	0.0115 (8)	0.0223 (7)	0.0254 (9)
C1	0.0505 (8)	0.0616 (9)	0.0511 (8)	0.0074 (7)	0.0171 (6)	0.0015 (7)
C2	0.0465 (7)	0.0596 (8)	0.0488 (7)	0.0116 (6)	0.0171 (6)	0.0055 (6)
C3	0.0453 (7)	0.0566 (8)	0.0503 (7)	0.0093 (6)	0.0194 (6)	0.0063 (6)
C4	0.0488 (8)	0.0695 (10)	0.0540 (8)	0.0105 (7)	0.0161 (6)	0.0076 (8)
C5	0.0471 (7)	0.0632 (9)	0.0467 (7)	0.0054 (6)	0.0169 (6)	0.0085 (6)
C6	0.0585 (9)	0.0638 (10)	0.0639 (9)	0.0070 (7)	0.0144 (7)	0.0091 (7)
C7	0.0894 (14)	0.0662 (11)	0.0825 (12)	−0.0095 (10)	0.0134 (10)	0.0130 (9)
C8	0.0761 (12)	0.0946 (15)	0.0774 (12)	−0.0279 (11)	0.0067 (9)	0.0208 (11)
C9	0.0497 (9)	0.1040 (15)	0.0710 (10)	−0.0060 (9)	0.0075 (7)	0.0297 (10)
C10	0.0502 (8)	0.0743 (10)	0.0661 (9)	0.0068 (7)	0.0163 (7)	0.0184 (8)
C11	0.0500 (8)	0.0608 (9)	0.0486 (7)	0.0026 (6)	0.0172 (6)	0.0099 (6)
C12	0.0587 (9)	0.0603 (9)	0.0573 (8)	0.0057 (7)	0.0198 (7)	0.0049 (7)
C13	0.0638 (9)	0.0709 (10)	0.0504 (8)	−0.0040 (8)	0.0162 (7)	0.0027 (7)
C14	0.0503 (8)	0.0732 (10)	0.0569 (8)	−0.0028 (7)	0.0168 (7)	0.0164 (8)
C15	0.0816 (12)	0.0679 (10)	0.0680 (10)	0.0210 (9)	0.0259 (9)	0.0178 (8)
C16	0.0836 (12)	0.0681 (10)	0.0553 (9)	0.0156 (9)	0.0234 (8)	0.0060 (8)
C17	0.0581 (9)	0.0690 (10)	0.0606 (9)	0.0049 (8)	0.0139 (7)	−0.0033 (8)
C18	0.0866 (14)	0.1014 (16)	0.0848 (13)	−0.0296 (12)	0.0139 (11)	0.0028 (12)
C19	0.0634 (10)	0.0968 (14)	0.0650 (10)	0.0000 (9)	0.0110 (8)	0.0238 (10)

Geometric parameters (Å, º)

O1—C17	1.209 (2)	C9—H9A	0.9300
N1—N2	1.3507 (18)	C10—H10A	0.9300
N1—C3	1.3603 (19)	C11—C12	1.370 (2)
N1—C11	1.4367 (18)	C11—C16	1.377 (2)
N2—C1	1.330 (2)	C12—C13	1.384 (2)
N3—C4	1.140 (2)	C12—H12A	0.9300
C1—C2	1.409 (2)	C13—C14	1.383 (3)
C1—C17	1.473 (2)	C13—H13A	0.9300
C2—C3	1.390 (2)	C14—C15	1.378 (3)
C2—C4	1.424 (2)	C14—C19	1.508 (2)
C3—C5	1.466 (2)	C15—C16	1.383 (2)
C5—C6	1.386 (2)	C15—H15A	0.9300
C5—C10	1.396 (2)	C16—H16A	0.9300
C6—C7	1.373 (3)	C17—C18	1.496 (3)
C6—H6A	0.9300	C18—H18A	0.9600
C7—C8	1.380 (3)	C18—H18B	0.9600
C7—H7A	0.9300	C18—H18C	0.9600
C8—C9	1.375 (3)	C19—H19C	0.9600
C8—H8A	0.9300	C19—H19A	0.9600
C9—C10	1.372 (3)	C19—H19B	0.9600
N2—N1—C3	113.27 (12)	C12—C11—N1	119.94 (14)
N2—N1—C11	118.53 (12)	C16—C11—N1	118.91 (14)
C3—N1—C11	128.20 (13)	C11—C12—C13	119.05 (15)
C1—N2—N1	105.05 (13)	C11—C12—H12A	120.5

N2—C1—C2	110.85 (13)	C13—C12—H12A	120.5
N2—C1—C17	120.73 (15)	C14—C13—C12	121.38 (16)
C2—C1—C17	128.41 (14)	C14—C13—H13A	119.3
C3—C2—C1	105.85 (13)	C12—C13—H13A	119.3
C3—C2—C4	126.16 (14)	C15—C14—C13	117.98 (14)
C1—C2—C4	127.87 (13)	C15—C14—C19	121.26 (17)
N1—C3—C2	104.97 (13)	C13—C14—C19	120.77 (17)
N1—C3—C5	125.17 (13)	C14—C15—C16	121.69 (16)
C2—C3—C5	129.86 (13)	C14—C15—H15A	119.2
N3—C4—C2	178.98 (17)	C16—C15—H15A	119.2
C6—C5—C10	118.77 (16)	C11—C16—C15	118.74 (16)
C6—C5—C3	119.47 (13)	C11—C16—H16A	120.6
C10—C5—C3	121.76 (15)	C15—C16—H16A	120.6
C7—C6—C5	120.73 (16)	O1—C17—C1	120.06 (17)
C7—C6—H6A	119.6	O1—C17—C18	122.28 (17)
C5—C6—H6A	119.6	C1—C17—C18	117.66 (16)
C6—C7—C8	119.97 (19)	C17—C18—H18A	109.5
C6—C7—H7A	120.0	C17—C18—H18B	109.5
C8—C7—H7A	120.0	H18A—C18—H18B	109.5
C9—C8—C7	119.85 (19)	C17—C18—H18C	109.5
C9—C8—H8A	120.1	H18A—C18—H18C	109.5
C7—C8—H8A	120.1	H18B—C18—H18C	109.5
C10—C9—C8	120.58 (17)	C14—C19—H19C	109.5
C10—C9—H9A	119.7	C14—C19—H19A	109.5
C8—C9—H9A	119.7	H19C—C19—H19A	109.5
C9—C10—C5	120.08 (17)	C14—C19—H19B	109.5
C9—C10—H10A	120.0	H19C—C19—H19B	109.5
C5—C10—H10A	120.0	H19A—C19—H19B	109.5
C12—C11—C16	121.13 (14)		

C3—N1—N2—C1	0.11 (16)	C6—C7—C8—C9	0.4 (3)
C11—N1—N2—C1	179.67 (13)	C7—C8—C9—C10	−1.0 (3)
N1—N2—C1—C2	0.18 (16)	C8—C9—C10—C5	0.5 (3)
N1—N2—C1—C17	−179.55 (13)	C6—C5—C10—C9	0.5 (2)
N2—C1—C2—C3	−0.39 (16)	C3—C5—C10—C9	179.78 (15)
C17—C1—C2—C3	179.31 (15)	N2—N1—C11—C12	−117.95 (16)
N2—C1—C2—C4	175.73 (14)	C3—N1—C11—C12	61.5 (2)
C17—C1—C2—C4	−4.6 (2)	N2—N1—C11—C16	60.91 (19)
N2—N1—C3—C2	−0.35 (16)	C3—N1—C11—C16	−119.60 (18)
C11—N1—C3—C2	−179.86 (13)	C16—C11—C12—C13	1.6 (2)
N2—N1—C3—C5	−179.96 (13)	N1—C11—C12—C13	−179.59 (14)
C11—N1—C3—C5	0.5 (2)	C11—C12—C13—C14	0.1 (3)
C1—C2—C3—N1	0.43 (15)	C12—C13—C14—C15	−1.6 (2)
C4—C2—C3—N1	−175.78 (13)	C12—C13—C14—C19	178.34 (16)
C1—C2—C3—C5	−179.98 (14)	C13—C14—C15—C16	1.5 (3)
C4—C2—C3—C5	3.8 (2)	C19—C14—C15—C16	−178.44 (18)
N1—C3—C5—C6	−138.02 (15)	C12—C11—C16—C15	−1.7 (3)
C2—C3—C5—C6	42.5 (2)	N1—C11—C16—C15	179.48 (16)

N1—C3—C5—C10	42.7 (2)	C14—C15—C16—C11	0.1 (3)
C2—C3—C5—C10	−136.79 (16)	N2—C1—C17—O1	−175.12 (16)
C10—C5—C6—C7	−1.1 (3)	C2—C1—C17—O1	5.2 (3)
C3—C5—C6—C7	179.61 (16)	N2—C1—C17—C18	4.4 (2)
C5—C6—C7—C8	0.7 (3)	C2—C1—C17—C18	−175.32 (18)