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Crystal structure of 1-benzyl-4-(2,4-dichlorophenyl)-2-imino-1,2,5,6,7,8,9,10-octahydrocycloocta[*b*]pyridine-3-carbonitrile

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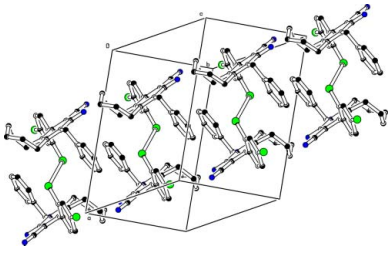
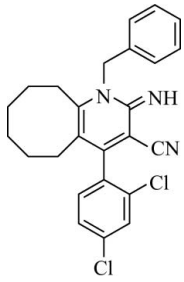
In the title compound, $C_{25}H_{23}Cl_2N_3$, the cyclooctene ring adopts a twist chair–chair conformation. The dihedral angles between the central pyridine ring (r.m.s. deviation = 0.013 Å) and the pendant chlorobenzene and benzyl rings are 78.07 (11) and 87.47 (12)°, respectively. No directional interactions could be identified in the crystal and the packing is governed by van der Waals interactions.

1. Chemical context

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. They also have numerous applications in medicinal chemistry (Passannanti *et al.*, 1998). The naturally occurring B6-vitamins pyridoxine, pyrodoxal, pyridoxamine and codecarbaxylase contain a pyridine nucleus (Shankaraiah *et al.*, 2010). The study of the properties and the formation of imines is of great interest due to the role they play in several important chemical and biological processes (Larkin, 1990). Imines and their complexes have a variety of applications in biological, clinical and analytical fields (Singh *et al.*, 1975; Patel *et al.*, 1999). Many pyridine-2-one and 3-cyano-2-imino pyridine derivatives exhibit antiproliferative activity (McNamara & Cook, 1987; Abadi *et al.*,1998). As part of our ongoing studies of substituted pyridine systems (Vishnupriya *et al.*, 2014*a*,*b*), we now describe herein the synthesis and crystal structure of the title compound, (I).





2. Structural commentary

The molecular structure of (I) is shown in Fig. 1. The cyclooctane ring adopts a twisted chair–chair conformation. Steric hindrance rotates the phenyl (C13–C18) and aromatic

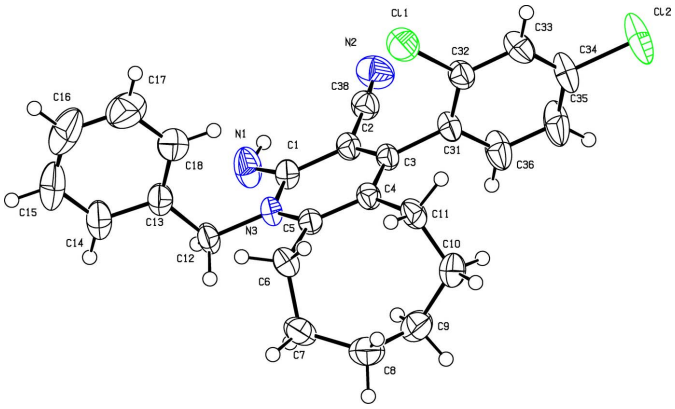


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

(C31–C36) rings out of the plane of the central pyridine ring by 87.47 (12) and 78.07 (11)°, respectively. The imino group is nearly coplanar with the pyridine ring as indicated by the torsion angle N1—C1—N3—C5 = −179.8 (2)°. The C—C and C—N bond lengths [C1—C2 = 1.453 (3), C4—C3 = 1.416 (3), C5—N3 = 1.376 (2) and C1—N3 = 1.398 (3) Å] are shorter than the standard C—C and C—N bond lengths (1.54 and 1.47 Å, respectively), while the C═C bond lengths [C4═C5 = 1.374 (3) and C2═C3 = 1.367 (3) Å] are longer than the standard C═C bond (1.34 Å). This shows that there is a homo-conjugation effect on the pyridine ring. The C38—C2 (Csp^2—Csp) single bond [1.432 (3) Å] tends towards an aromatic bond length rather than a σ bond length (1.50 Å), presumably due to conjugation.

3. Supramolecular features

No short directional contacts are observed in the crystal structure of (I) and the packing is governed by van der Waals interactions.

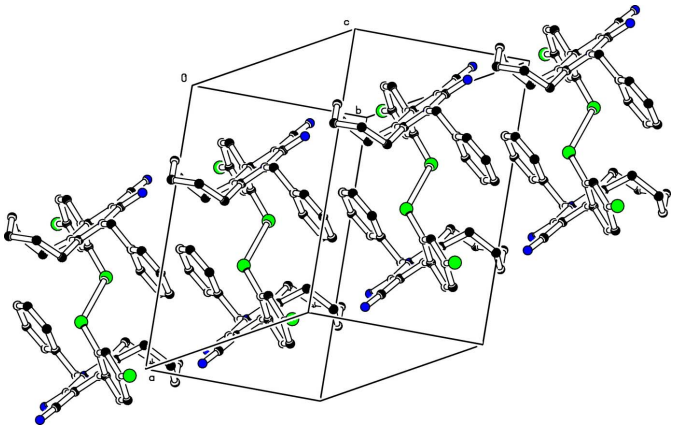


Figure 2
Partial packing diagram of the title compound. For clarity, H atoms are not shown.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{25}H_{23}Cl_2N_3$
M_r	436.36
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, b, c (Å)	13.0297 (6), 8.5901 (3), 19.7449 (8)
β (°)	98.337 (1)
V (Å^3)	2186.62 (15)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.31
Crystal size (mm)	0.21 × 0.19 × 0.18
Data collection	
Diffractometer	Bruker Kappa APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.967, 0.974
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	24005, 4762, 3607
R_{int}	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.639
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.151, 1.05
No. of reflections	4762
No. of parameters	275
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.65, −0.55

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SHELXS97* and *SHELXL2014*/6 (Sheldrick, 2008) and *PLATON* (Spek, 2009).

4. Database survey

Similar structures reported in the literature are 2-methoxy-4-(2-methoxyphenyl)-5,6,7,8,9,10-hexahydrocycloocta[*b*]pyridine-3-carbonitrile (Vishnupriya *et al.*, 2014*a*), 4-(2-fluorophenyl)-2-methoxy-5,6,7,8,9,10-hexahydrocyclo*o*cta[*b*]pyridine-3-carbonitrile (Vishnupriya *et al.*, 2014*b*) and 1-benzyl-4-(4-chlorophenyl)-2-imino-1,2,5,6,7,8,9,10-octahydrocycloocta-[*b*]pyridine-3-carbonitrile (Nagalakshmi *et al.*, 2014). In the structure of (I) reported here, the *d*-planar conformation of the pyridine ring is similar to those found in related structures (Vishnupriya *et al.*, 2014*a*,*b*). There are no significant intramolecular interactions or intermolecular C—H···N interactions, as in the case of the related structures (Vishnupriya *et al.*, 2014*a*,*b*). In the title compound, the bond lengths in the central pyridine ring span the range 1.367 (3)–1.453 (3) Å, which compares well with the range (1.369–1.447 Å) observed in a similar structure (Nagalakshmi *et al.*, 2014), but these bonds are systematically longer in the title compound, due to the substitution by Cl atoms in the aromatic ring.

5. Synthesis and crystallization

A mixture of cyclooctanone (1 mmol), 2,4 dicholoro-benzaldehyde (1 mmol) and malononitrile (1 mmol) was taken in ethanol (10 ml) to which pTSA (*p*-toluenesulfonic acid) (0.5 mmol) was added. The reaction mixture was heated

under reflux for 2–3 h. After completion of the reaction (TLC), the reaction mixture was poured into crushed ice and extracted with ethyl acetate. The excess solvent was removed under vacuum and the residue was subjected to column chromatography using a petroleum ether/ethyl acetate mixture (97:3 *v*/*v*) as eluent to afford pure product. The product was recrystallized from ethyl acetate solution, affording colourless blocks. Melting point: 407 K, yield: 65%.

6. Refinement

C-bound H atoms were placed in calculated positions and allowed to ride on their carrier atoms, with C—H = 0.93 (aromatic CH) or 0.97 Å (methylene CH_2). Imine atom H1 was found in a difference map and refined freely, with the N—H distance restrained to 0.84 (2) Å. Isotropic displacement parameters for H atoms were calculated as U_{iso}(H) = 1.2U_{eq}(C) for CH and CH_2 groups, while the U_{iso} factor for H1 was refined. Crystal data, data collection and structure refinement details are summarized in Table 1.

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Crystal structure of 1-benzyl-4-(2,4-dichlorophenyl)-2-imino-1,2,5,6,7,8,9,10-octahydrocycloocta[*b*]pyridine-3-carbonitrile

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Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014*/6 (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014*/6 (Sheldrick, 2008).

1-Benzyl-4-(2,4-dichlorophenyl)-2-imino-1,2,5,6,7,8,9,10-octahydrocycloocta[*b*]pyridine-3-carbonitrile

Crystal data

$C_{25}H_{23}Cl_2N_3$
$M_r = 436.36$
Monoclinic, $P2_1/n$
$a = 13.0297$ (6) Å
$b = 8.5901$ (3) Å
$c = 19.7449$ (8) Å
$\beta = 98.337$ (1)°
$V = 2186.62$ (15) Å^3
$Z = 4$
$F(000) = 912$
$D_x = 1.326$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2000 reflections
$\theta = 2$–31°
$\mu = 0.31$ mm^{-1}
$T = 293$ K
Block, colourless
0.21 × 0.19 × 0.18 mm

Data collection

Bruker Kappa APEXII diffractometer
Radiation source: fine-focus sealed tube
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.967$, $T_{max} = 0.974$
24005 measured reflections
4762 independent reflections
3607 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.1°$
$h = -16\rightarrow16$
$k = -10\rightarrow10$
$l = -25\rightarrow25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.151$
$S = 1.05$
4762 reflections
275 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0654P)^2 + 1.5156P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.65$ e Å^{-3}
$\Delta\rho_{min} = -0.55$ e Å^{-3}

Special details

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	0.20063 (16)	0.4628 (2)	0.12039 (10)	0.0359 (4)
C2	0.18748 (15)	0.4805 (2)	0.04645 (10)	0.0330 (4)
C3	0.24407 (15)	0.3969 (2)	0.00591 (9)	0.0312 (4)
C4	0.31948 (15)	0.2887 (2)	0.03557 (9)	0.0321 (4)
C5	0.33572 (15)	0.2735 (2)	0.10560 (9)	0.0307 (4)
C6	0.41621 (16)	0.1647 (3)	0.14117 (10)	0.0378 (5)
H6A	0.4428	0.2077	0.1857	0.045*
H6B	0.4735	0.1591	0.1149	0.045*
C7	0.3769 (2)	−0.0005 (3)	0.15108 (12)	0.0494 (6)
H7A	0.4257	−0.0518	0.1857	0.059*
H7B	0.3113	0.0069	0.1687	0.059*
C8	0.3611 (2)	−0.1035 (3)	0.08746 (14)	0.0609 (7)
H8A	0.4264	−0.1091	0.0694	0.073*
H8B	0.3449	−0.2078	0.1015	0.073*
C9	0.2775 (2)	−0.0542 (3)	0.02966 (14)	0.0605 (7)
H9A	0.2413	−0.1468	0.0110	0.073*
H9B	0.2276	0.0100	0.0489	0.073*
C10	0.3141 (2)	0.0348 (3)	−0.02886 (12)	0.0520 (6)
H10A	0.2540	0.0604	−0.0620	0.062*
H10B	0.3579	−0.0335	−0.0514	0.062*
C11	0.37366 (17)	0.1839 (3)	−0.00902 (10)	0.0399 (5)
H11A	0.4417	0.1575	0.0151	0.048*
H11B	0.3835	0.2398	−0.0503	0.048*
C12	0.29562 (17)	0.3405 (3)	0.22118 (9)	0.0398 (5)
H12A	0.3144	0.2335	0.2327	0.048*
H12B	0.2314	0.3628	0.2387	0.048*
C13	0.37932 (18)	0.4462 (3)	0.25617 (11)	0.0414 (5)
C14	0.3998 (2)	0.4435 (3)	0.32736 (12)	0.0560 (7)
H14	0.3629	0.3757	0.3516	0.067*
C15	0.4736 (3)	0.5392 (4)	0.36243 (16)	0.0791 (10)
H15	0.4858	0.5369	0.4100	0.095*
C16	0.5294 (3)	0.6381 (4)	0.3273 (2)	0.0870 (11)
H16	0.5793	0.7030	0.3510	0.104*
C17	0.5117 (3)	0.6416 (4)	0.25697 (19)	0.0786 (9)
H17	0.5498	0.7083	0.2331	0.094*
C18	0.4366 (2)	0.5451 (3)	0.22166 (14)	0.0576 (7)
H18	0.4249	0.5475	0.1741	0.069*
C31	0.22607 (16)	0.4217 (3)	−0.06963 (9)	0.0358 (4)
C32	0.29212 (17)	0.5111 (3)	−0.10249 (11)	0.0397 (5)

C33	0.27671 (19)	0.5318 (3)	−0.17275 (11)	0.0485 (6)
H33	0.3221	0.5922	−0.1939	0.058*
C34	0.19288 (19)	0.4609 (4)	−0.21027 (11)	0.0534 (6)
C35	0.1247 (2)	0.3726 (4)	−0.18008 (12)	0.0650 (8)
H35	0.0677	0.3265	−0.2064	0.078*
C36	0.14182 (18)	0.3531 (4)	−0.10991 (11)	0.0547 (7)
H36	0.0960	0.2927	−0.0892	0.066*
C38	0.11331 (17)	0.5948 (3)	0.01833 (11)	0.0395 (5)
N1	0.15090 (18)	0.5350 (3)	0.16254 (10)	0.0566 (6)
N2	0.05483 (17)	0.6890 (3)	−0.00069 (12)	0.0586 (6)
N3	0.27777 (13)	0.3564 (2)	0.14615 (8)	0.0333 (4)
Cl1	0.39949 (6)	0.59550 (9)	−0.05516 (3)	0.0674 (2)
Cl2	0.17481 (6)	0.48449 (13)	−0.29858 (3)	0.0841 (3)
H1	0.109 (2)	0.598 (3)	0.1392 (14)	0.097 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0379 (11)	0.0406 (11)	0.0286 (9)	0.0020 (9)	0.0026 (8)	−0.0034 (8)
C2	0.0313 (10)	0.0384 (11)	0.0284 (9)	−0.0006 (8)	0.0010 (7)	0.0008 (8)
C3	0.0303 (9)	0.0384 (11)	0.0241 (9)	−0.0070 (8)	0.0012 (7)	0.0010 (8)
C4	0.0318 (10)	0.0381 (11)	0.0270 (9)	−0.0027 (8)	0.0064 (7)	0.0003 (8)
C5	0.0307 (9)	0.0338 (10)	0.0275 (9)	−0.0021 (8)	0.0036 (7)	−0.0001 (8)
C6	0.0373 (11)	0.0450 (12)	0.0305 (10)	0.0037 (9)	0.0026 (8)	0.0048 (9)
C7	0.0635 (15)	0.0448 (13)	0.0420 (12)	0.0054 (11)	0.0147 (11)	0.0117 (10)
C8	0.089 (2)	0.0399 (13)	0.0577 (15)	−0.0023 (13)	0.0241 (14)	0.0028 (12)
C9	0.0794 (19)	0.0506 (15)	0.0532 (15)	−0.0222 (14)	0.0155 (14)	−0.0092 (12)
C10	0.0692 (16)	0.0524 (14)	0.0359 (11)	−0.0017 (12)	0.0128 (11)	−0.0112 (10)
C11	0.0470 (12)	0.0480 (12)	0.0274 (9)	0.0036 (10)	0.0144 (8)	0.0022 (9)
C12	0.0493 (12)	0.0488 (13)	0.0213 (9)	0.0015 (10)	0.0049 (8)	−0.0001 (8)
C13	0.0469 (12)	0.0432 (12)	0.0324 (10)	0.0105 (10)	−0.0001 (9)	−0.0052 (9)
C14	0.0644 (16)	0.0678 (17)	0.0335 (11)	0.0078 (13)	−0.0010 (11)	−0.0101 (11)
C15	0.084 (2)	0.102 (3)	0.0453 (15)	−0.001 (2)	−0.0122 (15)	−0.0267 (16)
C16	0.081 (2)	0.086 (2)	0.085 (2)	−0.0124 (19)	−0.0171 (19)	−0.037 (2)
C17	0.079 (2)	0.0652 (19)	0.089 (2)	−0.0210 (17)	0.0023 (18)	−0.0037 (17)
C18	0.0681 (17)	0.0525 (15)	0.0493 (14)	−0.0066 (13)	−0.0015 (12)	0.0008 (12)
C31	0.0341 (10)	0.0476 (12)	0.0249 (9)	−0.0008 (9)	0.0018 (8)	0.0027 (8)
C32	0.0392 (11)	0.0461 (12)	0.0322 (10)	−0.0047 (9)	0.0001 (8)	0.0042 (9)
C33	0.0483 (13)	0.0616 (15)	0.0370 (11)	−0.0005 (11)	0.0106 (10)	0.0126 (11)
C34	0.0502 (14)	0.0861 (19)	0.0228 (10)	0.0064 (13)	0.0016 (9)	0.0038 (11)
C35	0.0443 (13)	0.116 (3)	0.0318 (12)	−0.0175 (15)	−0.0042 (10)	−0.0060 (14)
C36	0.0392 (12)	0.092 (2)	0.0319 (11)	−0.0169 (13)	0.0006 (9)	0.0004 (12)
C38	0.0366 (11)	0.0438 (12)	0.0369 (11)	−0.0021 (10)	0.0010 (9)	0.0026 (9)
N1	0.0649 (14)	0.0694 (15)	0.0354 (10)	0.0265 (12)	0.0070 (9)	−0.0081 (10)
N2	0.0502 (12)	0.0571 (13)	0.0659 (14)	0.0074 (11)	−0.0001 (10)	0.0140 (11)
N3	0.0371 (9)	0.0409 (9)	0.0213 (7)	0.0015 (7)	0.0023 (6)	−0.0011 (7)
Cl1	0.0692 (4)	0.0768 (5)	0.0511 (4)	−0.0388 (4)	−0.0089 (3)	0.0102 (3)
Cl2	0.0784 (5)	0.1477 (8)	0.0242 (3)	0.0001 (5)	0.0007 (3)	0.0090 (4)

Geometric parameters (Å, º)

C1—N1	1.286 (3)	C12—N3	1.472 (2)
C1—N3	1.398 (3)	C12—C13	1.507 (3)
C1—C2	1.453 (3)	C12—H12A	0.9700
C2—C3	1.367 (3)	C12—H12B	0.9700
C2—C38	1.432 (3)	C13—C18	1.375 (4)
C3—C4	1.416 (3)	C13—C14	1.392 (3)
C3—C31	1.491 (2)	C14—C15	1.374 (4)
C4—C5	1.374 (3)	C14—H14	0.9300
C4—C11	1.505 (3)	C15—C16	1.371 (5)
C5—N3	1.376 (2)	C15—H15	0.9300
C5—C6	1.501 (3)	C16—C17	1.374 (5)
C6—C7	1.531 (3)	C16—H16	0.9300
C6—H6A	0.9700	C17—C18	1.390 (4)
C6—H6B	0.9700	C17—H17	0.9300
C7—C8	1.526 (4)	C18—H18	0.9300
C7—H7A	0.9700	C31—C32	1.383 (3)
C7—H7B	0.9700	C31—C36	1.390 (3)
C8—C9	1.519 (4)	C32—C33	1.384 (3)
C8—H8A	0.9700	C32—Cl1	1.725 (2)
C8—H8B	0.9700	C33—C34	1.370 (4)
C9—C10	1.519 (3)	C33—H33	0.9300
C9—H9A	0.9700	C34—C35	1.369 (4)
C9—H9B	0.9700	C34—Cl2	1.737 (2)
C10—C11	1.519 (3)	C35—C36	1.381 (3)
C10—H10A	0.9700	C35—H35	0.9300
C10—H10B	0.9700	C36—H36	0.9300
C11—H11A	0.9700	C38—N2	1.138 (3)
C11—H11B	0.9700	N1—H1	0.8599 (10)
N1—C1—N3	118.88 (19)	C10—C11—H11B	109.0
N1—C1—C2	127.1 (2)	H11A—C11—H11B	107.8
N3—C1—C2	114.05 (17)	N3—C12—C13	113.81 (18)
C3—C2—C38	121.57 (18)	N3—C12—H12A	108.8
C3—C2—C1	122.56 (18)	C13—C12—H12A	108.8
C38—C2—C1	115.84 (18)	N3—C12—H12B	108.8
C2—C3—C4	120.20 (17)	C13—C12—H12B	108.8
C2—C3—C31	119.39 (18)	H12A—C12—H12B	107.7
C4—C3—C31	120.41 (17)	C18—C13—C14	118.2 (2)
C5—C4—C3	118.32 (17)	C18—C13—C12	123.6 (2)
C5—C4—C11	121.01 (18)	C14—C13—C12	118.2 (2)
C3—C4—C11	120.47 (17)	C15—C14—C13	121.1 (3)
C4—C5—N3	121.26 (18)	C15—C14—H14	119.5
C4—C5—C6	121.66 (17)	C13—C14—H14	119.5
N3—C5—C6	117.07 (16)	C16—C15—C14	120.0 (3)
C5—C6—C7	114.38 (18)	C16—C15—H15	120.0
C5—C6—H6A	108.7	C14—C15—H15	120.0

C7—C6—H6A	108.7	C15—C16—C17	120.1 (3)
C5—C6—H6B	108.7	C15—C16—H16	120.0
C7—C6—H6B	108.7	C17—C16—H16	120.0
H6A—C6—H6B	107.6	C16—C17—C18	119.8 (3)
C8—C7—C6	116.11 (19)	C16—C17—H17	120.1
C8—C7—H7A	108.3	C18—C17—H17	120.1
C6—C7—H7A	108.3	C13—C18—C17	120.9 (3)
C8—C7—H7B	108.3	C13—C18—H18	119.6
C6—C7—H7B	108.3	C17—C18—H18	119.6
H7A—C7—H7B	107.4	C32—C31—C36	117.42 (19)
C9—C8—C7	116.9 (2)	C32—C31—C3	122.01 (18)
C9—C8—H8A	108.1	C36—C31—C3	120.56 (18)
C7—C8—H8A	108.1	C31—C32—C33	122.1 (2)
C9—C8—H8B	108.1	C31—C32—Cl1	119.34 (16)
C7—C8—H8B	108.1	C33—C32—Cl1	118.51 (17)
H8A—C8—H8B	107.3	C34—C33—C32	118.3 (2)
C10—C9—C8	116.2 (2)	C34—C33—H33	120.9
C10—C9—H9A	108.2	C32—C33—H33	120.9
C8—C9—H9A	108.2	C35—C34—C33	121.8 (2)
C10—C9—H9B	108.2	C35—C34—Cl2	119.97 (19)
C8—C9—H9B	108.2	C33—C34—Cl2	118.19 (19)
H9A—C9—H9B	107.4	C34—C35—C36	118.9 (2)
C9—C10—C11	115.67 (19)	C34—C35—H35	120.6
C9—C10—H10A	108.4	C36—C35—H35	120.6
C11—C10—H10A	108.4	C35—C36—C31	121.5 (2)
C9—C10—H10B	108.4	C35—C36—H36	119.3
C11—C10—H10B	108.4	C31—C36—H36	119.3
H10A—C10—H10B	107.4	N2—C38—C2	176.4 (2)
C4—C11—C10	112.94 (18)	C1—N1—H1	108 (2)
C4—C11—H11A	109.0	C5—N3—C1	123.54 (16)
C10—C11—H11A	109.0	C5—N3—C12	121.18 (17)
C4—C11—H11B	109.0	C1—N3—C12	115.27 (16)

N1—C1—C2—C3	−179.6 (2)	C15—C16—C17—C18	0.4 (6)
N3—C1—C2—C3	1.6 (3)	C14—C13—C18—C17	−1.1 (4)
N1—C1—C2—C38	2.2 (3)	C12—C13—C18—C17	179.0 (3)
N3—C1—C2—C38	−176.61 (18)	C16—C17—C18—C13	0.2 (5)
C38—C2—C3—C4	177.73 (19)	C2—C3—C31—C32	101.6 (2)
C1—C2—C3—C4	−0.4 (3)	C4—C3—C31—C32	−78.0 (3)
C38—C2—C3—C31	−1.9 (3)	C2—C3—C31—C36	−79.2 (3)
C1—C2—C3—C31	−179.99 (19)	C4—C3—C31—C36	101.2 (3)
C2—C3—C4—C5	−1.7 (3)	C36—C31—C32—C33	−0.5 (4)
C31—C3—C4—C5	177.90 (18)	C3—C31—C32—C33	178.7 (2)
C2—C3—C4—C11	173.25 (18)	C36—C31—C32—Cl1	−178.61 (19)
C31—C3—C4—C11	−7.1 (3)	C3—C31—C32—Cl1	0.6 (3)
C3—C4—C5—N3	2.5 (3)	C31—C32—C33—C34	0.1 (4)
C11—C4—C5—N3	−172.43 (18)	Cl1—C32—C33—C34	178.2 (2)
C3—C4—C5—C6	−178.29 (18)	C32—C33—C34—C35	0.6 (4)

C11—C4—C5—C6	6.8 (3)	C32—C33—C34—Cl2	−179.00 (19)
C4—C5—C6—C7	−89.6 (2)	C33—C34—C35—C36	−0.8 (5)
N3—C5—C6—C7	89.7 (2)	Cl2—C34—C35—C36	178.7 (2)
C5—C6—C7—C8	75.3 (3)	C34—C35—C36—C31	0.4 (5)
C6—C7—C8—C9	−65.1 (3)	C32—C31—C36—C35	0.3 (4)
C7—C8—C9—C10	98.2 (3)	C3—C31—C36—C35	−179.0 (3)
C8—C9—C10—C11	−57.6 (3)	C4—C5—N3—C1	−1.2 (3)
C5—C4—C11—C10	88.3 (2)	C6—C5—N3—C1	179.53 (18)
C3—C4—C11—C10	−86.5 (2)	C4—C5—N3—C12	−179.71 (19)
C9—C10—C11—C4	−49.5 (3)	C6—C5—N3—C12	1.1 (3)
N3—C12—C13—C18	−2.0 (3)	N1—C1—N3—C5	−179.8 (2)
N3—C12—C13—C14	178.0 (2)	C2—C1—N3—C5	−0.8 (3)
C18—C13—C14—C15	1.4 (4)	N1—C1—N3—C12	−1.2 (3)
C12—C13—C14—C15	−178.7 (3)	C2—C1—N3—C12	177.74 (17)
C13—C14—C15—C16	−0.8 (5)	C13—C12—N3—C5	86.2 (2)
C14—C15—C16—C17	−0.1 (6)	C13—C12—N3—C1	−92.4 (2)