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Crystal structure of 4-(naphthalen-2-yl)-2-oxo-6-phenyl-1,2-dihydropyridine-3-carbonitrile

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The synthesis and crystal structure of the title compound, $C_{22}H_{14}N_2O$, are described. The title compound was synthesized by a three-component one-pot reaction in DMSO involving chalcone, cyanoacetamide and elemental sulfur as catalyst. The compound was characterized by spectroscopic methods and single-crystal X-ray diffraction. The structure consists of inversion-related dimers produced by N—H···O hydrogen bonding, which further interact through π–π contacts.

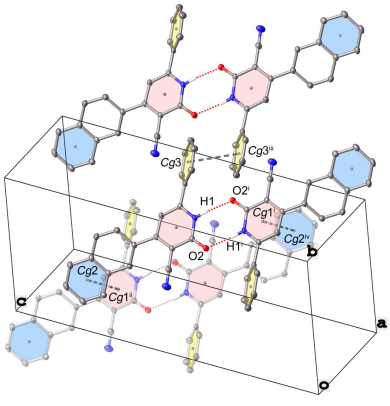
1. Chemical context

Pyridine skeletons play a pivotal role in drug discovery with more than 7000 existing drugs containing this moiety (De *et al.*, 2022). Recent investigations of 3-cyanopyrid-2-one derivatives have shown that the unsaturated and cyanide moieties significantly increase their biological activities compared to the original pyridine skeleton. The practical value of these compounds and the broad spectrum of biological activities (ranging from antitumor, anti-tuberculosis, anti-inflammatory, antimicrobial activities to anti-SARS-CoV-2) have made the 3-cyanopyrid-2-ones become the subject of intensive research in pyridine chemistry (Saleh *et al.*, 2021). Beside their promising biological activity, the 3-cyanopyrid-2-ones are also used in materials chemistry involving production of OLED devices, dyes, pigments, and other important applications.

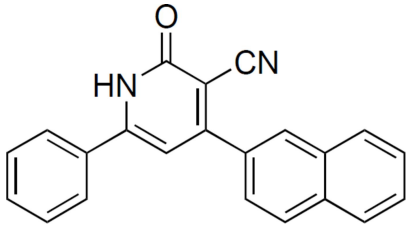
Based on a substituted pyridone scaffold, Cheney *et al.* (2007) identified a novel series of Pim-1 kinase inhibitors that could compete and interfere with Pim-1 ATP utilization (Cheney *et al.*, 2007).

By performing a high throughput screening and an NMR-based fragment screen, 3-cyanopyridones have been discovered and structurally optimized by hit-to-lead processes to become a novel inhibitor of *M. tuberculosis* thymidylate kinase (Mtb TMK) showing cellular activity against *M. tuberculosis* (Naik *et al.*, 2015).

Recently, based on the cyclization reaction between 2-nitro-1,3-dicarbonylic compounds and cyanoacetamide, 2-pyridone rings have been synthesized. These compounds are able to inhibit the aggregation of α-synuclein in human cultured cells and prevent the degeneration of dopaminergic neurons in the search for novel molecules for the treatment of Parkinson's disease (Mahía *et al.*, 2021). The syntheses of 3-cyanopyrid-2-ones are well documented and highlighted in the review of Litvinov (2006). These compounds can be synthesized by modification of a substituent in a preformed pyridine substrate or by formation of a C—N bond by a cyclization reaction. During our study on the use of elemental sulfur (Nguyen, 2017*a*,*b*, 2020) as a versatile sulfurating and oxidizing agent for



the syntheses of heterocyclic compounds such as thiophene, furan, benzothiazine, we noticed that the product of the Michael addition of cyanoacetamide on chalcone can undergo the formation of a C—N bond and aromatization to form the desired 3-cyanopyrid-2-ones in good yield.



2. Structural commentary

The title compound crystallizes in the solvent-free form in the centrosymmetric monoclinic space group $P2_1/n$ with one molecule in the asymmetric unit. The molecular structure is shown in Fig. 1. The δ-lactam moiety is almost planar with a maximum deviation from planarity for the N atom of the cyanide group (N31) of 0.047 (2) Å. The phenyl group and the lactam moiety form a dihedral angle of 50.4 (4)° while the naphthyl group is rotated by 35.6 (5)° with respect to the central lactam ring.

3. Supramolecular features

In the crystal, molecules form inversion-related dimers *via* N—H···O hydrogen bonds (Table 1 and Fig. 2). Neighboring dimers interact through π–π stacking, namely between the lactam N1–C6 ring and the phenylene C42–C49 ring [centroid-to-centroid distance $Cg1^{ii}\cdots Cg2$ of 3.991 (1) Å and a slippage of 1.968 (3) Å] and between parallel phenyl C60–C65 rings [centroid-to-centroid distance $Cg3\cdots Cg3^{iii}$ of 3.679 (5) Å and a slippage of 1.487 (3) Å].

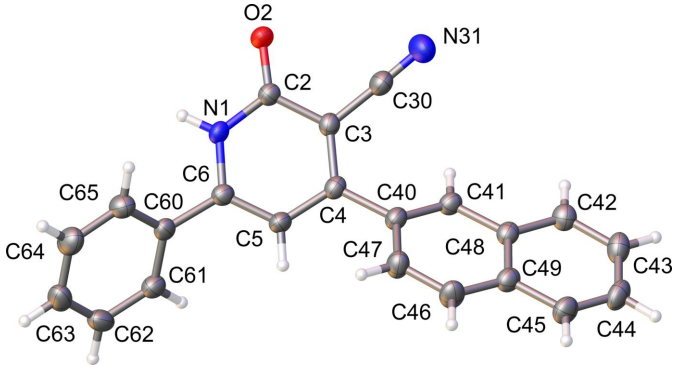


Figure 1
The molecular structure of the title compound, showing the atom labeling. Displacement ellipsoids are drawn at the 50% probability level. Generated with *OLEX2* (Dolomanov *et al.*, 2009).

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.90 (2)	1.91 (2)	2.8096 (18)	172.8 (19)

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

4. Database survey

A search in the Cambridge Structural Database (CSD, Version 5.43, update of November 2022; Groom *et al.*, 2016) for the 4,6-disubstituted 2-oxo-1,2-dihydropyridine-3-carbonitrile subunit reveals eleven hits involving four diaryl derivatives: JINTAC (Rong *et al.*, 2006, 2007), PELZIQ, PELZOW and PEQGOL (Chopra *et al.*, 2006). The rest consists of three compounds containing 4-phenyl-6-alkyl substituents [DOJBUB, DOJCEM (Rai *et al.*, 2014) and VEXYOP (Rai *et al.*, 2018)], two compounds possessing 4-alkyl-6-phenyl substituents (DUBXIH; Mishnev *et al.*, 1986 and RUGVUM; Rai *et al.*, 2015, 2018; Chen *et al.*, 2011) and two dialkyl derivatives (ERISIH; Rybakov *et al.*, 2004; Elassar, 2011; Chen *et al.*, 2011) and GIZBIB (Basheer & Rappoport, 2008). Across the series of metrics for all structures mentioned, all values regarding the pyridone moiety are in accordance with those reported herein.

5. Synthesis and crystallization

A mixture of chalcone (0.2583 g, 1.0 equiv), 2-cyanoacetamide (0.0883 g, 1.05 equiv) and DABCO (0.0224 g, 0.2 equiv) was

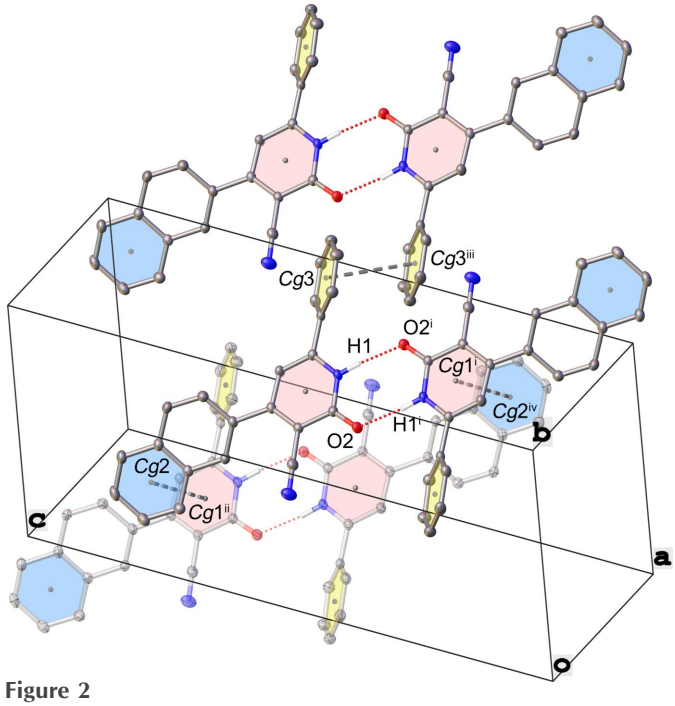


Figure 2
Packing diagram for the title compound, showing N—H···O hydrogen bonds and π–π stacking interactions. *Cg*1, *Cg*2 and *Cg*3 are the centroids of the lactam N1–C6 ring, the phenylene C42–C49 ring and the phenyl C60–C65 ring, respectively. Generated in *OLEX2* (Dolomanov *et al.*, 2009). Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x-1, y, z$; (iii) $-x+2, -y+2, -z+1$; (iv) $-x+1, -y+1, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{14}N_2O$
M_r	322.35
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	298
a, b, c (Å)	7.0845 (4), 10.4369 (6), 21.6298 (11)
β (°)	91.878 (2)
V (Å^3)	1598.45 (15)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.34 × 0.26 × 0.15
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.655, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18613, 3250, 2531
R_{int}	0.056
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.123, 1.06
No. of reflections	3250
No. of parameters	230
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.21

Computer programs: *APEX3* and *SAINT* (Bruker, 2019), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), and *OLEX2* (Dolomanov *et al.*, 2009).

dissolved in DMSO (0.2 mL). The reaction mixture was heated in a sealed tube at 353 K for 2 h. Then elemental sulfur (0.0064 g, 0.2 equiv) was added to the mixture and the temperature was raised to 393 K for 24 h. After cooling to room temperature, methanol was added to the reaction mixture to precipitate the crude product, which was then filtered and thoroughly washed with methanol and dichloromethane. Single crystals suitable for X-ray analysis were obtained by recrystallization of the compound in DMSO/DMF mixture.

^{1}H NMR (500 MHz, DMSO-d_6) δ 12.78 (*s*, 1H), 8.35 (*s*, 1H), 8.15–8.00 (*m*, 3H), 7.94 (*d*, J = 7.1 Hz, 2H), 7.84 (*dd*, J = 8.6, 2.0 Hz, 1H), 7.71–7.47 (*m*, 5H), 6.97 (*s*, 1H).

^{13}C NMR (126 MHz, DMSO-d_6) δ 164.2, 162.6, 160.3, 152.0, 133.9, 132.9, 131.7, 130.1, 129.4, 129.2, 129.1, 128.8, 128.8, 128.3, 128.2, 128.1, 127.5, 127.4, 127.1, 125.8, 125.0, 117.1, 107.1.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Positional parameters for the H atom attached to the N atom were refined. All H atoms bonded to C atoms were placed at calculated positions, with C—H = 0.93 Å, and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) for Csp^2—H.

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Crystal structure of 4-(naphthalen-2-yl)-2-oxo-6-phenyl-1,2-dihydropyridine-3-carbonitrile

Thai Thanh Thu Bui, Dinh Hung Mac, Pham Quang Trung and Chien Thang Pham

Computing details

Data collection: *APEX3* (Bruker, 2019); cell refinement: *SAINT* V8.40A (Bruker, 2019); data reduction: *SAINT* V8.40A (Bruker, 2019); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: Olex2 1.5 (Dolomanov *et al.*, 2009); software used to prepare material for publication: Olex2 1.5 (Dolomanov *et al.*, 2009).

4-(Naphthalen-2-yl)-2-oxo-6-phenyl-1,2-dihydropyridine-3-carbonitrile

Crystal data

$C_{22}H_{14}N_2O$
$M_r = 322.35$
Monoclinic, $P2_1/n$
$a = 7.0845$ (4) Å
$b = 10.4369$ (6) Å
$c = 21.6298$ (11) Å
$\beta = 91.878$ (2)°
$V = 1598.45$ (15) Å^3
$Z = 4$
$F(000) = 672$
$D_x = 1.339$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7569 reflections
$\theta = 2.7$–26.3°
$\mu = 0.08$ mm^{-1}
$T = 298$ K
Block, yellow
0.34 × 0.26 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min} = 0.655$, $T_{max} = 0.745$
18613 measured reflections
3250 independent reflections
2531 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.7°$
$h = -8 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.123$
$S = 1.06$
3250 reflections
230 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.7178P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.82728 (17)	0.39070 (12)	0.51570 (6)	0.0445 (3)
N1	0.8282 (2)	0.59995 (14)	0.54490 (6)	0.0335 (3)
C6	0.7537 (2)	0.70213 (16)	0.57481 (8)	0.0329 (4)
C2	0.7497 (2)	0.47978 (16)	0.54354 (8)	0.0331 (4)
C48	0.0535 (2)	0.45419 (16)	0.68796 (8)	0.0334 (4)
C60	0.8580 (2)	0.82447 (16)	0.57434 (8)	0.0341 (4)
C4	0.5045 (2)	0.56580 (17)	0.61101 (8)	0.0327 (4)
C3	0.5797 (2)	0.46509 (16)	0.57786 (8)	0.0322 (4)
C41	0.1982 (2)	0.46820 (17)	0.64419 (8)	0.0342 (4)
H41	0.191623	0.420700	0.607804	0.041*
C40	0.3471 (2)	0.55046 (16)	0.65470 (8)	0.0340 (4)
C5	0.5920 (2)	0.68616 (17)	0.60673 (8)	0.0366 (4)
H5	0.538843	0.756446	0.626038	0.044*
C61	0.7640 (3)	0.93869 (17)	0.56255 (8)	0.0409 (4)
H61	0.635429	0.937948	0.552319	0.049*
C49	0.0619 (2)	0.52910 (18)	0.74242 (8)	0.0381 (4)
N31	0.4577 (3)	0.23249 (18)	0.58144 (10)	0.0640 (5)
C42	−0.0972 (2)	0.36732 (18)	0.67854 (9)	0.0411 (4)
H42	−0.101608	0.315499	0.643546	0.049*
C65	1.0507 (3)	0.82723 (19)	0.58974 (9)	0.0443 (5)
H65	1.116142	0.751180	0.597029	0.053*
C47	0.3542 (3)	0.62309 (19)	0.71018 (9)	0.0437 (5)
H47	0.455112	0.678437	0.717990	0.052*
C45	−0.0852 (3)	0.51704 (19)	0.78517 (9)	0.0459 (5)
H45	−0.081273	0.565435	0.821287	0.055*
C62	0.8604 (3)	1.05386 (18)	0.56591 (10)	0.0499 (5)
H62	0.797354	1.130175	0.557074	0.060*
C30	0.5102 (2)	0.33702 (19)	0.58021 (9)	0.0422 (5)
C43	−0.2373 (3)	0.3587 (2)	0.72060 (9)	0.0478 (5)
H43	−0.336678	0.301691	0.713812	0.057*
C46	0.2157 (3)	0.61315 (19)	0.75229 (9)	0.0467 (5)
H46	0.222902	0.662601	0.788055	0.056*
C44	−0.2314 (3)	0.4353 (2)	0.77365 (9)	0.0490 (5)
H44	−0.328752	0.430277	0.801378	0.059*
C63	1.0496 (3)	1.0555 (2)	0.58234 (10)	0.0541 (6)
H63	1.113658	1.133087	0.585465	0.065*
C64	1.1440 (3)	0.9430 (2)	0.59413 (11)	0.0545 (5)
H64	1.271945	0.944829	0.605168	0.065*
H1	0.937 (3)	0.610 (2)	0.5249 (10)	0.053 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0441 (7)	0.0351 (7)	0.0557 (8)	−0.0034 (6)	0.0221 (6)	−0.0099 (6)
N1	0.0315 (7)	0.0344 (8)	0.0352 (8)	−0.0007 (6)	0.0113 (6)	−0.0002 (6)
C6	0.0349 (9)	0.0320 (9)	0.0320 (9)	0.0030 (7)	0.0059 (7)	0.0001 (7)
C2	0.0339 (8)	0.0335 (9)	0.0321 (9)	0.0007 (7)	0.0062 (7)	−0.0006 (7)
C48	0.0313 (8)	0.0351 (9)	0.0341 (9)	0.0028 (7)	0.0032 (7)	0.0049 (7)
C60	0.0378 (9)	0.0335 (9)	0.0316 (9)	−0.0009 (7)	0.0124 (7)	−0.0017 (7)
C4	0.0289 (8)	0.0372 (9)	0.0322 (9)	0.0016 (7)	0.0049 (7)	0.0018 (7)
C3	0.0306 (8)	0.0337 (9)	0.0326 (9)	−0.0018 (7)	0.0045 (7)	0.0019 (7)
C41	0.0349 (9)	0.0365 (9)	0.0315 (9)	0.0020 (7)	0.0064 (7)	−0.0016 (7)
C40	0.0317 (8)	0.0353 (9)	0.0355 (9)	0.0019 (7)	0.0076 (7)	0.0014 (7)
C5	0.0366 (9)	0.0322 (9)	0.0417 (10)	0.0027 (7)	0.0130 (7)	−0.0022 (8)
C61	0.0408 (10)	0.0387 (10)	0.0441 (10)	0.0038 (8)	0.0126 (8)	0.0003 (8)
C49	0.0398 (9)	0.0406 (10)	0.0345 (9)	0.0030 (8)	0.0085 (7)	0.0028 (8)
N31	0.0523 (11)	0.0456 (11)	0.0951 (15)	−0.0076 (9)	0.0158 (10)	−0.0058 (10)
C42	0.0389 (9)	0.0437 (11)	0.0406 (10)	−0.0026 (8)	0.0018 (8)	0.0007 (8)
C65	0.0406 (10)	0.0400 (11)	0.0526 (12)	0.0025 (8)	0.0060 (8)	−0.0039 (9)
C47	0.0408 (10)	0.0473 (11)	0.0436 (11)	−0.0099 (9)	0.0091 (8)	−0.0063 (9)
C45	0.0489 (11)	0.0520 (12)	0.0377 (10)	0.0004 (9)	0.0142 (8)	0.0008 (9)
C62	0.0655 (13)	0.0310 (10)	0.0546 (12)	0.0045 (9)	0.0220 (10)	−0.0019 (9)
C30	0.0347 (9)	0.0432 (11)	0.0494 (11)	−0.0003 (8)	0.0102 (8)	−0.0032 (9)
C43	0.0388 (10)	0.0557 (12)	0.0494 (11)	−0.0077 (9)	0.0057 (8)	0.0098 (10)
C46	0.0526 (11)	0.0510 (12)	0.0373 (10)	−0.0099 (9)	0.0130 (8)	−0.0124 (9)
C44	0.0411 (10)	0.0631 (13)	0.0438 (11)	0.0025 (10)	0.0166 (8)	0.0126 (10)
C63	0.0601 (13)	0.0418 (12)	0.0618 (13)	−0.0143 (10)	0.0225 (10)	−0.0150 (10)
C64	0.0412 (11)	0.0548 (13)	0.0680 (14)	−0.0081 (10)	0.0082 (10)	−0.0142 (11)

Geometric parameters (Å, º)

O2—C2	1.245 (2)	C61—C62	1.383 (3)
N1—C6	1.363 (2)	C49—C45	1.421 (2)
N1—C2	1.372 (2)	C49—C46	1.410 (3)
N1—H1	0.90 (2)	N31—C30	1.153 (3)
C6—C60	1.475 (2)	C42—H42	0.9300
C6—C5	1.367 (2)	C42—C43	1.371 (3)
C2—C3	1.444 (2)	C65—H65	0.9300
C48—C41	1.425 (2)	C65—C64	1.379 (3)
C48—C49	1.413 (3)	C47—H47	0.9300
C48—C42	1.411 (2)	C47—C46	1.364 (2)
C60—C61	1.385 (2)	C45—H45	0.9300
C60—C65	1.395 (3)	C45—C44	1.359 (3)
C4—C3	1.388 (2)	C62—H62	0.9300
C4—C40	1.494 (2)	C62—C63	1.375 (3)
C4—C5	1.405 (2)	C43—H43	0.9300
C3—C30	1.426 (3)	C43—C44	1.398 (3)
C41—H41	0.9300	C46—H46	0.9300

C41—C40	1.373 (2)	C44—H44	0.9300
C40—C47	1.419 (3)	C63—H63	0.9300
C5—H5	0.9300	C63—C64	1.370 (3)
C61—H61	0.9300	C64—H64	0.9300

C6—N1—C2	124.23 (14)	C46—C49—C48	118.80 (15)
C6—N1—H1	119.1 (13)	C46—C49—C45	122.42 (17)
C2—N1—H1	116.7 (13)	C48—C42—H42	119.8
N1—C6—C60	118.17 (14)	C43—C42—C48	120.48 (18)
N1—C6—C5	119.25 (16)	C43—C42—H42	119.8
C5—C6—C60	122.49 (15)	C60—C65—H65	120.1
O2—C2—N1	120.53 (15)	C64—C65—C60	119.83 (18)
O2—C2—C3	123.93 (16)	C64—C65—H65	120.1
N1—C2—C3	115.50 (15)	C40—C47—H47	119.4
C49—C48—C41	119.03 (16)	C46—C47—C40	121.16 (17)
C42—C48—C41	121.83 (16)	C46—C47—H47	119.4
C42—C48—C49	119.14 (15)	C49—C45—H45	119.8
C61—C60—C6	120.54 (16)	C44—C45—C49	120.49 (18)
C61—C60—C65	119.16 (17)	C44—C45—H45	119.8
C65—C60—C6	120.18 (16)	C61—C62—H62	120.0
C3—C4—C40	123.54 (15)	C63—C62—C61	119.97 (19)
C3—C4—C5	117.71 (14)	C63—C62—H62	120.0
C5—C4—C40	118.54 (15)	N31—C30—C3	178.4 (2)
C4—C3—C2	121.73 (15)	C42—C43—H43	119.8
C4—C3—C30	123.54 (15)	C42—C43—C44	120.30 (18)
C30—C3—C2	114.38 (15)	C44—C43—H43	119.8
C48—C41—H41	119.4	C49—C46—H46	119.5
C40—C41—C48	121.29 (16)	C47—C46—C49	121.03 (17)
C40—C41—H41	119.4	C47—C46—H46	119.5
C41—C40—C4	123.20 (15)	C45—C44—C43	120.75 (17)
C41—C40—C47	118.67 (15)	C45—C44—H44	119.6
C47—C40—C4	118.11 (15)	C43—C44—H44	119.6
C6—C5—C4	121.42 (16)	C62—C63—H63	119.9
C6—C5—H5	119.3	C64—C63—C62	120.21 (19)
C4—C5—H5	119.3	C64—C63—H63	119.9
C60—C61—H61	119.9	C65—C64—H64	119.7
C62—C61—C60	120.29 (18)	C63—C64—C65	120.52 (19)
C62—C61—H61	119.9	C63—C64—H64	119.7
C48—C49—C45	118.78 (17)		

O2—C2—C3—C4	176.33 (17)	C41—C48—C49—C46	−1.4 (3)
O2—C2—C3—C30	3.0 (3)	C41—C48—C42—C43	−178.05 (17)
N1—C6—C60—C61	135.08 (17)	C41—C40—C47—C46	−0.7 (3)
N1—C6—C60—C65	−49.1 (2)	C40—C4—C3—C2	−170.35 (16)
N1—C6—C5—C4	2.1 (3)	C40—C4—C3—C30	2.4 (3)
N1—C2—C3—C4	−1.4 (2)	C40—C4—C5—C6	170.28 (16)
N1—C2—C3—C30	−174.79 (15)	C40—C47—C46—C49	0.8 (3)
C6—N1—C2—O2	−179.26 (16)	C5—C6—C60—C61	−48.6 (2)

C6—N1—C2—C3	−1.4 (2)	C5—C6—C60—C65	127.28 (19)
C6—C60—C61—C62	176.01 (17)	C5—C4—C3—C2	4.3 (2)
C6—C60—C65—C64	−174.71 (18)	C5—C4—C3—C30	177.09 (17)
C2—N1—C6—C60	177.59 (15)	C5—C4—C40—C41	148.18 (17)
C2—N1—C6—C5	1.1 (3)	C5—C4—C40—C47	−32.9 (2)
C48—C41—C40—C4	178.35 (16)	C61—C60—C65—C64	1.2 (3)
C48—C41—C40—C47	−0.5 (3)	C61—C62—C63—C64	1.4 (3)
C48—C49—C45—C44	−0.3 (3)	C49—C48—C41—C40	1.6 (3)
C48—C49—C46—C47	0.3 (3)	C49—C48—C42—C43	2.2 (3)
C48—C42—C43—C44	−0.6 (3)	C49—C45—C44—C43	2.0 (3)
C60—C6—C5—C4	−174.25 (16)	C42—C48—C41—C40	−178.13 (16)
C60—C61—C62—C63	−1.4 (3)	C42—C48—C49—C45	−1.8 (3)
C60—C65—C64—C63	−1.2 (3)	C42—C48—C49—C46	178.30 (17)
C4—C40—C47—C46	−179.62 (18)	C42—C43—C44—C45	−1.6 (3)
C3—C4—C40—C41	−37.2 (3)	C65—C60—C61—C62	0.1 (3)
C3—C4—C40—C47	141.73 (18)	C45—C49—C46—C47	−179.63 (19)
C3—C4—C5—C6	−4.7 (3)	C62—C63—C64—C65	−0.1 (3)
C41—C48—C49—C45	178.47 (16)	C46—C49—C45—C44	179.63 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.90 (2)	1.91 (2)	2.8096 (18)	172.8 (19)

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+1.