IUCrData

ISSN 2414-3146

Triclinic polymorph of 5-(*N*-phthalimido)pentan-2-one

Faith M. Akwi, Salma Hassan Babikir, Eric C. Hosten and Paul Watts*

Nelson Mandela University, University Way, Port Elizabeth 6031, South Africa. *Correspondence e-mail: paul.watts@mandela.ac.za

Received 13 November 2026
Accepted 17 November 2026

Edited by M. Bolte, Goethe-Universität Frankfurt, Germany

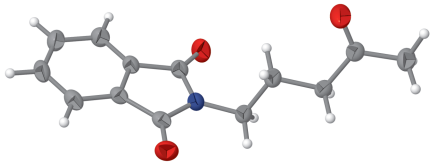
Keywords: crystal structure; triclinic; polymorph; energy framework.

CCDC reference: 2604136

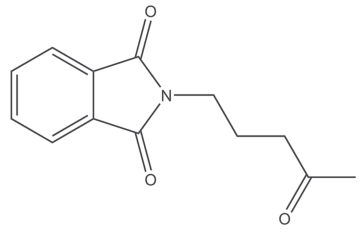
Structural data: full structural data are available from iucrdata.iucr.org

The structure of the title compound [systematic name: 2-(4-oxopentyl)-1*H*-isoindole-1,3(2*H*)-dione], $C_{13}H_{13}NO_3$, was determined by X-ray crystallography and crystallizes in the space group $P\overline{1}$ (No. 2), compared to *C*2/*c* for the known polymorph. Energy framework calculations show that non-classical C—H···O-type interactions, together with π–π ring and C═O···π ring interactions, set up a three-dimensional intermolecular framework.

3D view



Chemical scheme



Structure description

N-Alkylated phthalimido derivatives are important compounds in organic synthesis that are used as protected sources of primary amines *via* Gabriel synthesis (Gibson & Bradshaw, 1968). They have also been reported to possess biological activity, such as anti-inflammatory, anti-Alzheimer, anti-epileptic, anticonvulsant, *etc* (Fernandes *et al.*, 2023; Kushwaha & Kaushik, 2016). Herein, the synthesis and crystal structure of 5-(*N*-phthalimido)pentan-2-one, **1**, a key intermediate towards primaquine diphosphate, an important API used in the treatment of malaria as a primary prophylaxis, radical cure and presumptive antirelapse therapy (Ashley *et al.*, 2014), is discussed.

The title compound crystallizes in the triclinic space group $P\overline{1}$ with one molecule in the asymmetric unit (Fig. 1). All bond lengths, bond angles, torsions and dihedrals correspond with similar molecules whose crystal structures have been determined and whose metrical parameters have been deposited with the Cambridge Structural Database (CSD; Groom *et al.*, 2016). A search in the CSD yielded one hit for 5-(*N*-phthalimido)-pentan-2-one, which crystallizes in the monoclinic space group *C*2/*c* (Rheingold & Golen, 2020). The dihedral angles through the phthalimide and alkane groups least-squares planes is 74.71 (5)° here, which differs from the angle of 80.30° in the published monoclinic structure.

CrystalExplorer (Version 21.5) was utilized to calculate the energy framework (Mackenzie *et al.*, 2017) and shows extensive three-dimensional intermolecular inter-

OPEN ACCESS
Published under a CC BY 4.0 licence

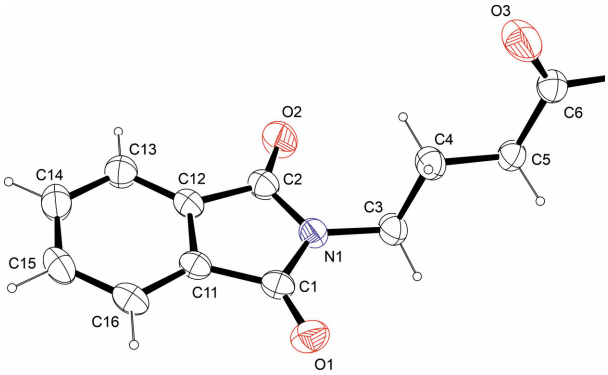


Figure 1
The molecular structure of compound **1**, with displacement ellipsoids drawn at the 50% probability level.

actions (Fig. 2). The non-classical C—H···O-type interactions appear to dominate (Table 1). The C5—H5*A*···O1 interaction is centrosymmetric, linking two molecules with an $R_2^2(14)$ descriptor, and has an interaction energy of −26.4 kJ mol^{-1}. The C15—H15···O2 and C7—H7*B*···O1 interactions link molecules in the *a* axis in chains with *C*(7) and *C*(9) descriptors, respectively, and a total interaction energy of −26.8 kJ mol^{-1}. On one side of the phthalimide ring system there is a π–π ring interaction with a centroid-to-centroid (N1/C1/C2/C11–C16) distance of 3.6848 (8) Å and −35.3 kJ mol^{-1} interaction energy. On the other side of the phthalimide group there are two centrosymmetric C6═O3···π C1═O3 carbonyl interactions of 2.984 (2) Å between O3 and C1, 0.24 Å less than the sum of the van der Waal radii and an interaction energy of −37.9 kJ mol^{-1}. These interactions alternate to form a chain linking molecules down vector [011].

The hydrogen-bond interactions, together with the π-interactions, result in a three-dimensional energy framework (Fig. 2). *TopCryst* (Shevchenko *et al.*, 2022) determines the underlying net as gpu-x (Fig. 3).

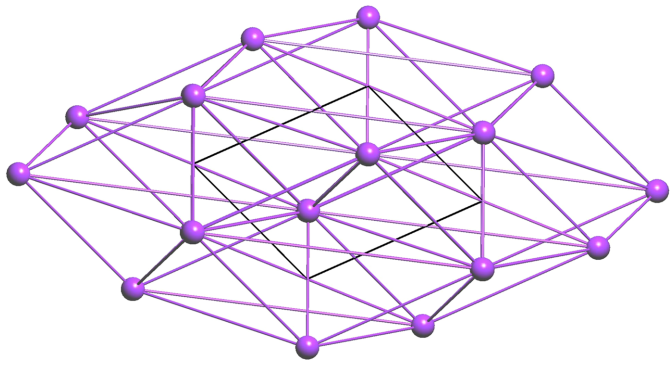


Figure 3
Underlying net viewed down the *b* axis. The spheres represent the centroids of the title compounds and the violet lines the van der Waal interactions. Black lines show the unit cell.

Synthesis and crystallization

The title compound **1** (Fig. 1) was synthesized *via* the phase-transfer-catalyzed *N*-alkylation of potassium phthalimide in 49% isolated yield (Akwi *et al.*, 2025).

Spectroscopic data for **1**: ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.86 (*s*, 4H; Ar—H), 3.58 (*t*, *J* = 8 Hz, 2H; CH_2), 2.53 (*t*, *J* = 8 Hz, 2H; CH_2), 2.09 (*s*, 3H; CH_3), 1.79 (*q*, *J* = 8 Hz, 2H; CH_2). ^{13}C NMR (100 MHz, DMSO-d_6): δ 208.50, 168.55, 134.79, 131.94, 123.39, 40.17, 37.23, 30.10, 22.47. IR: ν = 3459, 3403, 2957, 2928, 2856, 1770, 1704, 1614, 1466, 1453, 1396, 1363, 1333, 1270, 1231, 1187, 1164, 1126, 1086, 1047, 955, 873, 792, 718, 710, 619, 596, 549, 529, 464, 434 cm^{-1}.

Crystals of **1** were obtained at room temperature from a sample purified on a silica-gel chromatography column using a 9:1 ratio of hexane and ethyl acetate, respectively, as eluent> The N1—C3 bond length [1.459 (2) Å] confirms the formation of the product **1** and is consistent with a single-bond length.

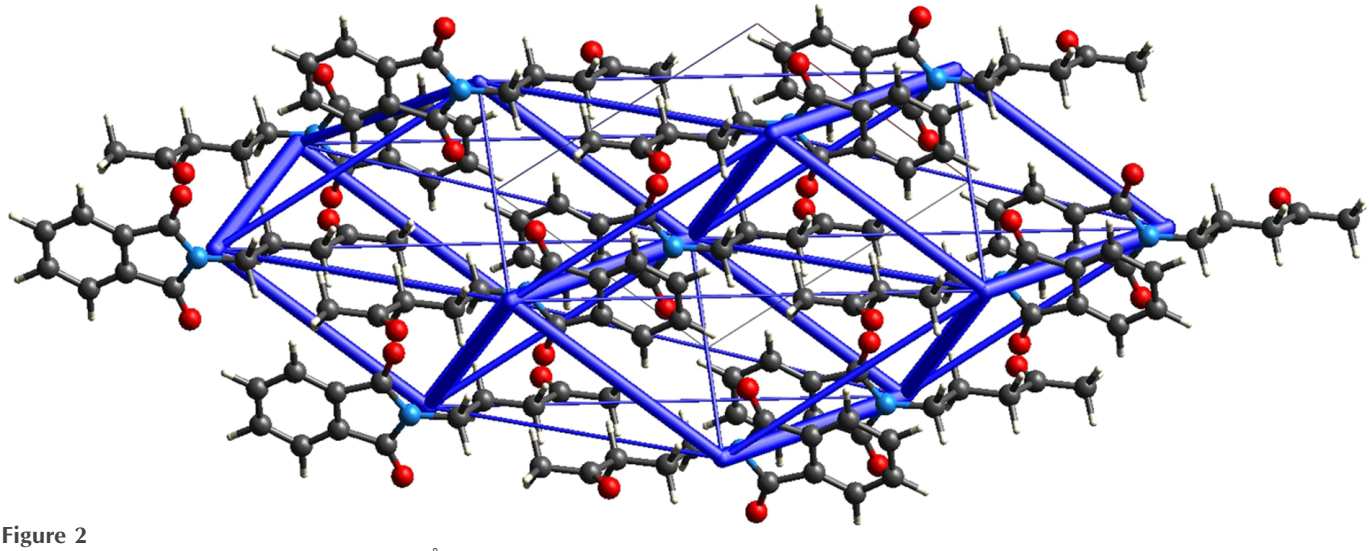


Figure 2
Energy framework drawn for all molecules 3.8 Å around compound **1** and viewed down the *b* axis. Blue tubes represent the total calculated energy drawn at a cylinder scale of 80 and a cut-off at 5 kJ mol^{-1}.

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···O1^{i}	0.99	2.44	3.384 (2)	158
C7—H7B···O1^{ii}	0.98	2.53	3.449 (2)	156
C15—H15···O2^{iii}	0.95	2.44	3.251 (2)	143

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $x+1, y, z$; (iii) $x-1, y, z$.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were located in a difference map and refined as riding on their parent atom, with aromatic C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C), methylene C—H = 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C) or methyl C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C). The methyl group was allowed to rotate to best fit the electron-density map.

Acknowledgements

We thank the National Research Fund and Nelson Mandela University for financial support.

Funding information

Funding for this research was provided by: National Research Fund (grant No. 85103).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{13}NO_3$
M_r	231.24
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	200
a, b, c (Å)	8.2691 (3), 8.4687 (2), 9.3437 (3)
α, β, γ (°)	95.9247 (11), 107.8907 (11), 107.8754 (10)
V (Å^3)	578.36 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.37 × 0.33 × 0.20
Data collection	
Diffractometer	Bruker D8 QUEST
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.693, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	47140, 2862, 2257
R_{int}	0.056
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.118, 1.13
No. of reflections	2862
No. of parameters	156
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.18

Computer programs: *APEX5* (Bruker, 2019), *SAINT* (Bruker, 2019), *SHELXT2019* (Sheldrick, 2015*a*), *SHELXL2025* (Sheldrick, 2015*b*) and *ShelXle* (Hübschle *et al.*, 2011), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2020) and *Mercury* (Macrae *et al.*, 2020).

References

Akwi, F. M., Swartbooi, S. & Watts, P. (2025). *Chem. A Eur. J.* **31**, e02183.
Ashley, E. A., Recht, J. & White, N. J. (2014). *Malar. J.* **13**, 418.
Bruker (2019). *APEX5*, *SAINT*, *SADABS* and *TWINABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Fernandes, G. F. S., Lopes, J. R., Dos Santos, J. L. & Scarim, C. B. (2023). *Drug Dev. Res.* **84**, 1346–1375.
Gibson, M. S. & Bradshaw, R. W. (1968). *Angew. Chem. Int. Ed. Engl.* **7**, 919–930.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Hübschle, C. B., Sheldrick, G. M. & Dittrich, B. (2011). *J. Appl. Cryst.* **44**, 1281–1284.
Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
Kushwaha, N. (2016). *J. Appl. Pharm. Sci.* **6**, 159–171.
Mackenzie, C. F., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *IUCrJ* **4**, 575–587.
Macrae, C. F., Sovago, I., Cottrell, S. J., Galek, P. T. A., McCabe, P., Pidcock, E., Platings, M., Shields, G. P., Stevens, J. S., Towler, M. & Wood, P. A. (2020). *J. Appl. Cryst.* **53**, 226–235.
Rheingold, A. L. & Golen, J. A. (2020). *CSD Communication* (CCDC 922964; CSD refcode ZUWTOD). CCDC, Cambridge, England.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Shevchenko, A. P., Shabalin, A. A., Karpukhin, I. Y. & Blatov, V. A. (2022). *Sci. Technol. Adv. Mater. Meth.* **2**, 250–265.
Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.

full crystallographic data

IUCrData (2026). **11**, x261208 [https://doi.org/10.1107/S2414314626012083]

Triclinic polymorph of 5-(*N*-phthalimido)pentan-2-one

Faith M. Akwi, Salma Hassan Babikir, Eric C. Hosten and Paul Watts

2-(4-Oxopentyl)-1*H*-isoindole-1,3(2*H*)-dione

Crystal data

$C_{13}H_{13}NO_3$
M_r = 231.24
Triclinic, $P\overline{1}$
a = 8.2691 (3) Å
b = 8.4687 (2) Å
c = 9.3437 (3) Å
α = 95.9247 (11)°
β = 107.8907 (11)°
γ = 107.8754 (10)°
V = 578.36 (3) Å^3
Z = 2
$F(000)$ = 244
D_x = 1.328 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9951 reflections
θ = 2.4–28.3°
μ = 0.10 mm^{-1}
T = 200 K
Block, brown
0.37 × 0.33 × 0.20 mm

Data collection

Bruker D8 QUEST diffractometer
Radiation source: sealed x-ray tube
Graphite monochromator
Detector resolution: 7.3910 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.693, T_{max} = 0.746
47140 measured reflections
2862 independent reflections
2257 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 28.3°, θ_{min} = 2.4°
h = −11→11
k = −11→11
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.118
S = 1.13
2862 reflections
156 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2 + 0.3292P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: SHELXL2025 (Sheldrick, 2015*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.068 (6)

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. All non-hydrogen atoms were refined anisotropically. Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C). The H atoms of the methyl group were allowed to rotate with a fixed angle around the C—C bond to best fit the experimental electron density (HFIX 137 in the SHELXL program (Sheldrick, 2015b)), with U_{iso}(H) set to 1.5U_{eq}(C).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.33366 (18)	0.92114 (16)	0.65108 (15)	0.0431 (3)
O2	0.77359 (17)	0.77066 (19)	0.97267 (16)	0.0492 (4)
O3	0.7372 (2)	0.49359 (16)	0.4168 (2)	0.0607 (5)
N1	0.57972 (18)	0.85546 (16)	0.78875 (16)	0.0312 (3)
C1	0.4078 (2)	0.86654 (19)	0.75650 (19)	0.0309 (3)
C2	0.6293 (2)	0.7890 (2)	0.91837 (19)	0.0330 (4)
C3	0.6938 (2)	0.9008 (2)	0.6963 (2)	0.0359 (4)
H3A	0.820614	0.967434	0.765326	0.043*
H3B	0.650510	0.973163	0.628514	0.043*
C4	0.6898 (2)	0.7437 (2)	0.5981 (2)	0.0362 (4)
H4A	0.723131	0.666816	0.665158	0.043*
H4B	0.564695	0.682156	0.523295	0.043*
C5	0.8199 (2)	0.78890 (19)	0.51145 (18)	0.0313 (3)
H5A	0.781560	0.860219	0.440167	0.038*
H5B	0.943225	0.857472	0.586275	0.038*
C6	0.8293 (2)	0.6367 (2)	0.42067 (19)	0.0330 (4)
C7	0.9579 (3)	0.6708 (2)	0.3350 (2)	0.0426 (4)
H7A	0.945904	0.563217	0.274776	0.064*
H7B	1.083010	0.725947	0.408522	0.064*
H7C	0.929202	0.745517	0.265540	0.064*
C11	0.3420 (2)	0.80079 (18)	0.87790 (18)	0.0287 (3)
C12	0.4736 (2)	0.75079 (19)	0.97349 (18)	0.0297 (3)
C13	0.4493 (2)	0.6798 (2)	1.0948 (2)	0.0373 (4)
H13	0.539334	0.644504	1.158850	0.045*
C14	0.2880 (2)	0.6617 (2)	1.1202 (2)	0.0399 (4)
H14	0.267184	0.612860	1.202981	0.048*
C15	0.1575 (2)	0.7137 (2)	1.0269 (2)	0.0383 (4)
H15	0.048980	0.701090	1.047213	0.046*
C16	0.1824 (2)	0.7843 (2)	0.9037 (2)	0.0354 (4)
H16	0.092661	0.819922	0.839577	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0478 (7)	0.0441 (7)	0.0426 (7)	0.0236 (6)	0.0136 (6)	0.0166 (6)
O2	0.0324 (7)	0.0695 (9)	0.0590 (9)	0.0288 (6)	0.0201 (6)	0.0249 (7)

O3	0.0701 (10)	0.0309 (7)	0.0867 (11)	0.0049 (6)	0.0531 (9)	−0.0001 (7)
N1	0.0313 (7)	0.0307 (7)	0.0354 (7)	0.0122 (5)	0.0158 (6)	0.0072 (6)
C1	0.0320 (8)	0.0260 (7)	0.0329 (8)	0.0116 (6)	0.0097 (7)	0.0015 (6)
C2	0.0294 (8)	0.0329 (8)	0.0375 (9)	0.0121 (7)	0.0128 (7)	0.0059 (7)
C3	0.0396 (9)	0.0293 (8)	0.0437 (10)	0.0101 (7)	0.0240 (8)	0.0079 (7)
C4	0.0383 (9)	0.0286 (8)	0.0440 (10)	0.0078 (7)	0.0230 (8)	0.0049 (7)
C5	0.0349 (8)	0.0285 (8)	0.0321 (8)	0.0094 (6)	0.0154 (7)	0.0082 (6)
C6	0.0332 (8)	0.0326 (8)	0.0320 (8)	0.0093 (7)	0.0128 (7)	0.0059 (7)
C7	0.0456 (10)	0.0431 (10)	0.0409 (10)	0.0120 (8)	0.0231 (8)	0.0050 (8)
C11	0.0274 (8)	0.0251 (7)	0.0306 (8)	0.0096 (6)	0.0082 (6)	0.0001 (6)
C12	0.0263 (7)	0.0297 (8)	0.0330 (8)	0.0109 (6)	0.0107 (6)	0.0034 (6)
C13	0.0354 (9)	0.0424 (9)	0.0378 (9)	0.0173 (7)	0.0134 (7)	0.0119 (7)
C14	0.0416 (10)	0.0418 (10)	0.0396 (10)	0.0125 (8)	0.0216 (8)	0.0089 (8)
C15	0.0302 (8)	0.0381 (9)	0.0462 (10)	0.0092 (7)	0.0195 (8)	−0.0005 (7)
C16	0.0277 (8)	0.0373 (9)	0.0402 (9)	0.0151 (7)	0.0097 (7)	0.0015 (7)

Geometric parameters (Å, º)

O1—C1	1.207 (2)	C5—H5B	0.9900
O2—C2	1.210 (2)	C6—C7	1.496 (2)
O3—C6	1.207 (2)	C7—H7A	0.9800
N1—C2	1.389 (2)	C7—H7B	0.9800
N1—C1	1.395 (2)	C7—H7C	0.9800
N1—C3	1.459 (2)	C11—C16	1.382 (2)
C1—C11	1.489 (2)	C11—C12	1.387 (2)
C2—C12	1.487 (2)	C12—C13	1.376 (2)
C3—C4	1.522 (2)	C13—C14	1.391 (2)
C3—H3A	0.9900	C13—H13	0.9500
C3—H3B	0.9900	C14—C15	1.383 (3)
C4—C5	1.519 (2)	C14—H14	0.9500
C4—H4A	0.9900	C15—C16	1.391 (3)
C4—H4B	0.9900	C15—H15	0.9500
C5—C6	1.504 (2)	C16—H16	0.9500
C5—H5A	0.9900		

C2—N1—C1	111.82 (13)	O3—C6—C7	121.41 (15)
C2—N1—C3	122.93 (14)	O3—C6—C5	121.68 (15)
C1—N1—C3	125.21 (14)	C7—C6—C5	116.91 (14)
O1—C1—N1	125.12 (16)	C6—C7—H7A	109.5
O1—C1—C11	128.98 (15)	C6—C7—H7B	109.5
N1—C1—C11	105.89 (13)	H7A—C7—H7B	109.5
O2—C2—N1	124.74 (15)	C6—C7—H7C	109.5
O2—C2—C12	128.90 (16)	H7A—C7—H7C	109.5
N1—C2—C12	106.36 (13)	H7B—C7—H7C	109.5
N1—C3—C4	111.51 (13)	C16—C11—C12	120.79 (15)
N1—C3—H3A	109.3	C16—C11—C1	131.06 (15)
C4—C3—H3A	109.3	C12—C11—C1	108.14 (13)
N1—C3—H3B	109.3	C13—C12—C11	121.71 (15)

C4—C3—H3B	109.3	C13—C12—C2	130.54 (15)
H3A—C3—H3B	108.0	C11—C12—C2	107.75 (14)
C5—C4—C3	111.88 (13)	C12—C13—C14	117.60 (16)
C5—C4—H4A	109.2	C12—C13—H13	121.2
C3—C4—H4A	109.2	C14—C13—H13	121.2
C5—C4—H4B	109.2	C15—C14—C13	120.98 (16)
C3—C4—H4B	109.2	C15—C14—H14	119.5
H4A—C4—H4B	107.9	C13—C14—H14	119.5
C6—C5—C4	113.70 (13)	C14—C15—C16	121.14 (15)
C6—C5—H5A	108.8	C14—C15—H15	119.4
C4—C5—H5A	108.8	C16—C15—H15	119.4
C6—C5—H5B	108.8	C11—C16—C15	117.76 (15)
C4—C5—H5B	108.8	C11—C16—H16	121.1
H5A—C5—H5B	107.7	C15—C16—H16	121.1
C2—N1—C1—O1	179.33 (15)	N1—C1—C11—C12	−1.28 (16)
C3—N1—C1—O1	−2.9 (2)	C16—C11—C12—C13	1.5 (2)
C2—N1—C1—C11	0.07 (17)	C1—C11—C12—C13	−178.19 (15)
C3—N1—C1—C11	177.81 (13)	C16—C11—C12—C2	−178.44 (14)
C1—N1—C2—O2	−177.99 (16)	C1—C11—C12—C2	1.91 (17)
C3—N1—C2—O2	4.2 (3)	O2—C2—C12—C13	−2.8 (3)
C1—N1—C2—C12	1.06 (17)	N1—C2—C12—C13	178.25 (17)
C3—N1—C2—C12	−176.73 (13)	O2—C2—C12—C11	177.15 (17)
C2—N1—C3—C4	74.3 (2)	N1—C2—C12—C11	−1.86 (17)
C1—N1—C3—C4	−103.22 (18)	C11—C12—C13—C14	−0.9 (2)
N1—C3—C4—C5	−175.34 (14)	C2—C12—C13—C14	179.01 (16)
C3—C4—C5—C6	176.35 (15)	C12—C13—C14—C15	−0.2 (3)
C4—C5—C6—O3	1.5 (2)	C13—C14—C15—C16	0.6 (3)
C4—C5—C6—C7	−178.61 (15)	C12—C11—C16—C15	−1.0 (2)
O1—C1—C11—C16	−0.1 (3)	C1—C11—C16—C15	178.60 (15)
N1—C1—C11—C16	179.12 (16)	C14—C15—C16—C11	−0.1 (2)
O1—C1—C11—C12	179.51 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*B*···O1	0.99	2.60	2.938 (2)	100
C5—H5*A*···O1^{i}	0.99	2.44	3.384 (2)	158
C7—H7*B*···O1^{ii}	0.98	2.53	3.449 (2)	156
C15—H15···O2^{iii}	0.95	2.44	3.251 (2)	143

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1; (ii) *x*+1, *y*, *z*; (iii) *x*−1, *y*, *z*.