Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Benzyl-2-[(2*RS*,3*SR*,6*SR*)-3-methyl-4-oxo-2,6-diphenylpiperidin-1-ylcarbonyl]-2-phenylethenyl phenylacetate

Kanagasabapathy Thanikasalam,[a] Ramasubbu Jeyaraman,[a] Krishnaswamy Panchanatheswaran,[a] John N. Low[b] and Christopher Glidewell[c]*

[a]School of Chemistry, Bharathidasan University, Tiruchirappalli, Tamil Nadu 620 024, India, [b]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and [c]School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland

Correspondence e-mail: cg@st-andrews.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.062
wR factor = 0.153
Data-to-parameter ratio = 17.5

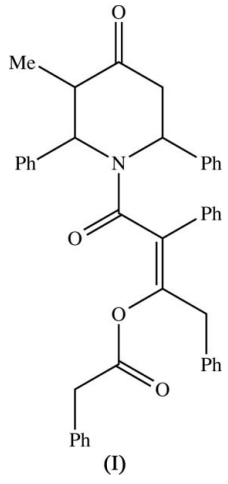
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 8 March 2006
Accepted 8 March 2006

Molecules of the title compound, $C_{42}H_{37}NO_4$, are weakly linked into chains by a C—H···O hydrogen bond and pairs of such chains are linked by a single aromatic π–π stacking interaction.

Comment

The reactions of acyl chlorides with piperidines in a 1:1 molar ratio in the presence of a suitable base yields the *N*-acyl derivatives, but the yields are sometimes low. This observation prompted us to carry out the reaction of 3-methyl-2,6-diphenylpiperidin-4-one with an excess of phenylacetyl chloride. The ^{13}C NMR spectrum of the product contained three absorptions at 168, 170 and 210 p.p.m. indicative of the presence of three different carbonyl environments. The present investigation was therefore undertaken to establish the constitution, configuration and conformation of the product, the title compound, (I) (Fig. 1).



(I)

The piperidone ring adopts a boat conformation with a pseudo-mirror plane containing atoms C33 and C36; the ring-puckering parameters (Cremer & Pople, 1975) for the atom sequence N31–C32–C33–C34–C35–C36 are $\theta = 94.1\ (2)^\circ$ and $\varphi = 122.0\ (2)^\circ$, very close to the ideal values for a boat conformation of $\theta = 90^\circ$ and $\varphi = (60n)^\circ$. The substituents at C32 and C33 occupy equatorial sites and that at C36 occupies an axial site. In the selected reference molecule, the configuration at C22 is *R* and those at C33 and C36 are both *S*. The centrosymmetric space group accommodates equal numbers of the *RSS* and *SRR* enantiomers.

The presence of a vinylic ester is shown clearly by the geometry involving atoms C1 and C2 (Table 1). There are two C—H···O hydrogen bonds within the molecule, both involving atom O1 as the acceptor (Table 2), and these may have

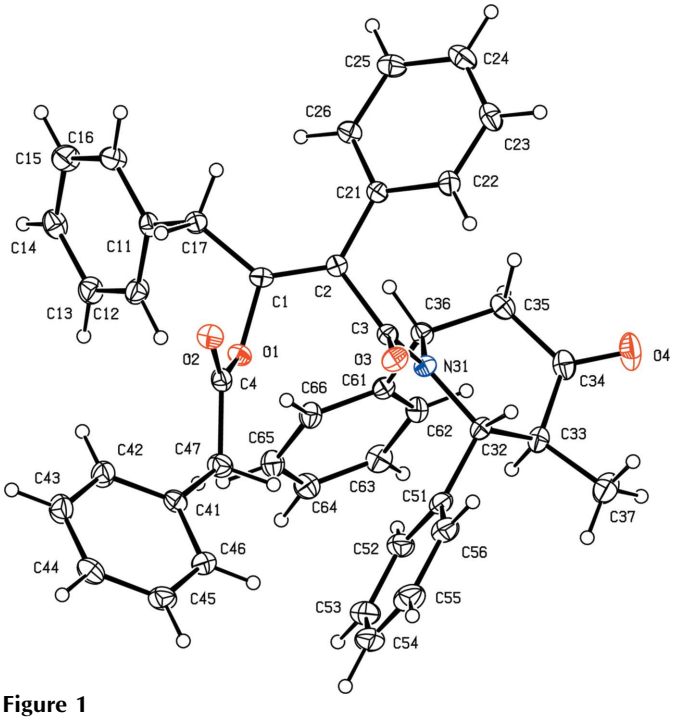


Figure 1
The *RSS* enantiomer of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

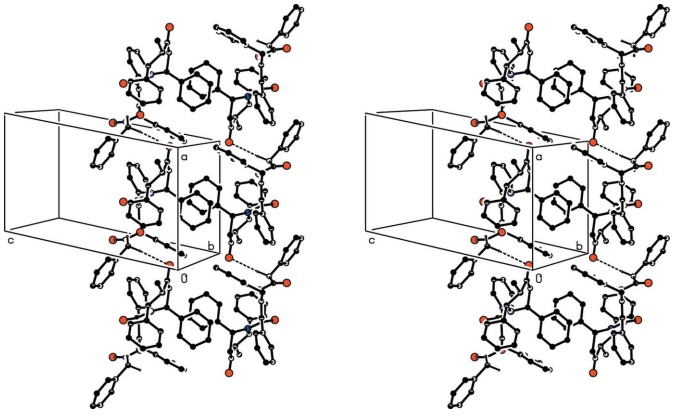


Figure 2
A stereoscopic view of part of the crystal structure of (I), showing the formation of a π-stacked pair of $C(12)$ chains along [100]. For the sake of clarity, H atoms not involved in the motif shown have been omitted.

some influence on the overall molecular conformation. A third hydrogen bond (Table 2) links the molecules into chains: atom C47 at (x, y, z) acts as donor, *via* H47*A*, to atom O4 at $(-1+x, y, z)$, so generating by translation a $C(12)$ (Bernstein *et al.*, 1995) chain running parallel to the [100] direction (Fig. 2). Two such chains, related to one another by inversion, pass through each unit cell and these chains are linked by a π–π stacking interaction. The C61–C66 rings in the molecules at (x, y, z) and $(1-x, 1-y, -z)$ are strictly parallel, with an interplanar spacing of 3.523 (2) Å; the ring-centroid separation is 3.868 (2) Å, corresponding to a ring offset of 1.597 (2) Å. Propagation of this interaction by translation and inversion then links a pair of [100] chains into a molecular ladder (Fig. 2).

Experimental

The title compound was prepared by heating under reflux a benzene solution of 3-methyl-2,6-diphenylpiperidin-4-one with a fourfold molar excess of phenylacetyl chloride in the presence of triethylamine. The resulting solid product was crystallized from aqueous ethanol, yielding crystals suitable for single-crystal X-ray diffraction (yield 48%, m.p. 419–421 K).

Crystal data

$C_{42}H_{37}NO_4$
$M_r = 619.73$
Triclinic, $P\overline{1}$
$a = 9.3027$ (3) Å
$b = 12.1333$ (5) Å
$c = 14.5927$ (6) Å
$\alpha = 88.7510$ (17)°
$\beta = 78.574$ (3)°
$\gamma = 85.653$ (2)°
$V = 1609.79$ (11) Å^3
$Z = 2$
$D_x = 1.279$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7258 reflections
$\theta = 2.9$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 120$ (2) K
Plate, colourless
0.13 × 0.07 × 0.03 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.985$, $T_{max} = 0.997$
34829 measured reflections
7423 independent reflections
4485 reflections with $I > 2\sigma(I)$
$R_{int} = 0.091$
$\theta_{max} = 27.7°$
$h = -12 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = -19 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.153$
$S = 1.01$
7423 reflections
425 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.6078P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.332 (3)	C2—C3	1.519 (3)
C1—O1	1.415 (2)	C2—C21	1.485 (3)
C1—C17	1.494 (3)		
C2—C1—O1	114.70 (18)	C1—C2—C3	116.53 (19)
C2—C1—C17	130.9 (2)	C1—C2—C21	127.73 (19)
O1—C1—C17	114.38 (17)	C3—C2—C21	115.55 (17)
O1—C1—C2—C3	−7.7 (3)	C36—N31—C32—C51	−115.6 (2)
O1—C1—C2—C21	177.47 (18)	N31—C32—C33—C37	165.83 (19)
C17—C1—C2—C21	−1.3 (4)	C34—C35—C36—C61	−77.2 (2)
C17—C1—C2—C3	173.5 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1	0.95	2.42	3.075 (3)	126
C47—H47*A*···O4^{i}	0.99	2.59	3.571 (3)	172
C66—H66···O1	0.95	2.53	3.469 (3)	172

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

All H atoms were located in difference maps and then treated as riding atoms, with C—H = 0.95 (aromatic), 0.98 (CH_3), 0.99 (CH_2) or

1.00 Å (aliphatic CH), and with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England. The authors thank the staff for all their help and advice.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
Hooft, R. W. W. (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.
McArdle, P. (2003). *OSCAIL for Windows*. Version 10. Crystallography Centre, Chemistry Department, NUI Galway, Ireland.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supporting information

Acta Cryst. (2006). E**62**, o1443–o1445 [https://doi.org/10.1107/S1600536806008804]

1-Benzyl-2-[(2*RS*,3*SR*,6*SR*)-3-methyl-4-oxo-2,6-diphenylpiperidin-1-yl-carbonyl]-2-phenylethenyl phenylacetate

Kanagasabapathy Thanikasalam, Ramasubbu Jeyaraman, Krishnaswamy Panchanatheswaran, John N. Low and Christopher Glidewell

1-Benzyl-2-[(2RS,3SR,6SR)-3-methyl-4-oxo-2,6-diphenylpiperidin-1-ylcarbonyl]- 2-phenylethenyl phenylacetate

Crystal data

$C_{42}H_{37}NO_4$
$M_r = 619.73$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.3027$ (3) Å
$b = 12.1333$ (5) Å
$c = 14.5927$ (6) Å
$\alpha = 88.7510$ (17)°
$\beta = 78.574$ (3)°
$\gamma = 85.653$ (2)°
$V = 1609.79$ (11) Å^3
$Z = 2$
$F(000) = 656$
$D_x = 1.279$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7258 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.08$ mm^{-1}
$T = 120$ K
Plate, colourless
0.13 × 0.07 × 0.03 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.985$, $T_{max} = 0.997$
34829 measured reflections
7423 independent reflections
4485 reflections with $I > 2\sigma(I)$
$R_{int} = 0.091$
$\theta_{max} = 27.7°$, $\theta_{min} = 3.0°$
$h = -12 \rightarrow 10$
$k = -15 \rightarrow 15$
$l = -19 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.153$
$S = 1.02$
7423 reflections
425 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
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.6078P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2356 (2)	0.43745 (17)	0.33712 (14)	0.0236 (5)
C17	0.1334 (2)	0.53735 (18)	0.36474 (14)	0.0255 (5)
C11	0.0816 (2)	0.60214 (18)	0.28508 (15)	0.0260 (5)
C12	0.0602 (3)	0.5520 (2)	0.20435 (16)	0.0335 (6)
C13	0.0064 (3)	0.6143 (2)	0.13589 (17)	0.0358 (6)
C14	−0.0288 (3)	0.7261 (2)	0.14681 (17)	0.0353 (6)
C15	−0.0094 (3)	0.7758 (2)	0.22692 (18)	0.0395 (6)
C16	0.0463 (3)	0.7141 (2)	0.29531 (17)	0.0340 (6)
O1	0.16875 (15)	0.34768 (12)	0.30674 (10)	0.0256 (3)
C4	0.0938 (2)	0.28122 (18)	0.37423 (16)	0.0261 (5)
O2	0.06080 (17)	0.30686 (13)	0.45492 (11)	0.0314 (4)
C47	0.0658 (3)	0.17689 (19)	0.32941 (16)	0.0319 (5)
C41	−0.0574 (2)	0.11470 (18)	0.38370 (15)	0.0267 (5)
C42	−0.1918 (3)	0.1688 (2)	0.42342 (17)	0.0351 (6)
C43	−0.3067 (3)	0.1106 (2)	0.47079 (18)	0.0392 (6)
C44	−0.2887 (3)	−0.0033 (2)	0.47951 (17)	0.0364 (6)
C45	−0.1564 (3)	−0.0582 (2)	0.44091 (17)	0.0367 (6)
C46	−0.0423 (3)	0.00086 (19)	0.39343 (17)	0.0337 (6)
C2	0.3793 (2)	0.41933 (17)	0.33484 (14)	0.0224 (5)
C21	0.4813 (2)	0.49775 (18)	0.35853 (14)	0.0232 (5)
C22	0.5994 (2)	0.4542 (2)	0.39704 (15)	0.0292 (5)
C23	0.7003 (3)	0.5218 (2)	0.41917 (16)	0.0352 (6)
C24	0.6862 (3)	0.6342 (2)	0.40385 (16)	0.0375 (6)
C25	0.5708 (3)	0.6784 (2)	0.36463 (16)	0.0346 (6)
C26	0.4689 (2)	0.61127 (18)	0.34235 (15)	0.0277 (5)
C3	0.4454 (2)	0.30227 (18)	0.31279 (15)	0.0240 (5)
O3	0.42009 (17)	0.22856 (12)	0.37155 (10)	0.0298 (4)
N31	0.53379 (19)	0.28357 (14)	0.22807 (12)	0.0237 (4)
C32	0.6095 (2)	0.17035 (17)	0.21216 (15)	0.0257 (5)
C51	0.5041 (2)	0.08504 (18)	0.19800 (16)	0.0287 (5)
C52	0.4433 (3)	0.0850 (2)	0.11868 (18)	0.0366 (6)
C53	0.3486 (3)	0.0059 (2)	0.1075 (2)	0.0452 (7)
C54	0.3135 (3)	−0.0740 (2)	0.1749 (2)	0.0490 (7)
C55	0.3730 (3)	−0.0747 (2)	0.2543 (2)	0.0461 (7)
C56	0.4681 (3)	0.0039 (2)	0.26610 (18)	0.0370 (6)
C33	0.7436 (2)	0.17069 (19)	0.13054 (16)	0.0301 (5)
C37	0.8405 (3)	0.0642 (2)	0.13068 (19)	0.0457 (7)
C34	0.8295 (3)	0.2720 (2)	0.12965 (16)	0.0338 (6)
O4	0.96224 (18)	0.26760 (16)	0.11562 (14)	0.0520 (5)
C35	0.7406 (2)	0.3808 (2)	0.14707 (16)	0.0316 (5)
C36	0.5752 (2)	0.37259 (18)	0.15922 (15)	0.0253 (5)
C61	0.5154 (2)	0.35869 (17)	0.07048 (14)	0.0243 (5)
C62	0.6029 (2)	0.35915 (19)	−0.01801 (15)	0.0295 (5)
C63	0.5429 (3)	0.3476 (2)	−0.09700 (16)	0.0342 (6)
C64	0.3938 (3)	0.3367 (2)	−0.08813 (16)	0.0340 (6)

C65	0.3051 (3)	0.3382 (2)	0.00000 (16)	0.0326 (5)
C66	0.3652 (2)	0.34915 (19)	0.07836 (16)	0.0296 (5)
H17A	0.1828	0.5878	0.3988	0.031*
H17B	0.0457	0.5139	0.4089	0.031*
H12	0.0826	0.4747	0.1961	0.040*
H13	−0.0063	0.5792	0.0808	0.043*
H14	−0.0658	0.7683	0.0998	0.042*
H15	−0.0342	0.8528	0.2356	0.047*
H16	0.0601	0.7498	0.3499	0.041*
H47A	0.0437	0.1957	0.2671	0.038*
H47B	0.1571	0.1274	0.3197	0.038*
H42	−0.2049	0.2469	0.4180	0.042*
H43	−0.3979	0.1488	0.4973	0.047*
H44	−0.3674	−0.0433	0.5120	0.044*
H45	−0.1433	−0.1362	0.4468	0.044*
H46	0.0486	−0.0378	0.3669	0.040*
H22	0.6106	0.3769	0.4082	0.035*
H23	0.7802	0.4905	0.4452	0.042*
H24	0.7549	0.6807	0.4200	0.045*
H25	0.5613	0.7557	0.3528	0.042*
H26	0.3898	0.6430	0.3158	0.033*
H32	0.6475	0.1485	0.2699	0.031*
H52	0.4668	0.1397	0.0716	0.044*
H53	0.3075	0.0067	0.0528	0.054*
H54	0.2486	−0.1282	0.1669	0.059*
H55	0.3487	−0.1295	0.3011	0.055*
H56	0.5090	0.0025	0.3209	0.044*
H33	0.7049	0.1723	0.0713	0.036*
H37A	0.9232	0.0644	0.0773	0.069*
H37B	0.7828	0.0010	0.1259	0.069*
H37C	0.8781	0.0585	0.1889	0.069*
H35A	0.7730	0.4312	0.0940	0.038*
H35B	0.7613	0.4140	0.2040	0.038*
H36	0.5272	0.4432	0.1885	0.030*
H62	0.7052	0.3675	−0.0249	0.035*
H63	0.6045	0.3471	−0.1573	0.041*
H64	0.3527	0.3282	−0.1420	0.041*
H65	0.2024	0.3317	0.0067	0.039*
H66	0.3032	0.3502	0.1385	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0292 (12)	0.0210 (11)	0.0216 (11)	−0.0050 (9)	−0.0061 (9)	−0.0001 (9)
C17	0.0254 (12)	0.0272 (12)	0.0237 (11)	−0.0018 (9)	−0.0046 (9)	−0.0010 (9)
C11	0.0192 (11)	0.0290 (13)	0.0299 (12)	−0.0029 (9)	−0.0048 (9)	0.0031 (10)
C12	0.0340 (13)	0.0337 (14)	0.0328 (13)	0.0056 (10)	−0.0093 (10)	−0.0022 (11)
C13	0.0361 (14)	0.0428 (15)	0.0304 (13)	0.0023 (11)	−0.0132 (11)	−0.0018 (11)

C14	0.0327 (13)	0.0391 (15)	0.0364 (14)	−0.0060 (11)	−0.0127 (11)	0.0114 (11)
C15	0.0467 (15)	0.0276 (14)	0.0475 (16)	−0.0042 (11)	−0.0177 (12)	0.0083 (12)
C16	0.0392 (14)	0.0305 (14)	0.0352 (13)	−0.0064 (11)	−0.0125 (11)	−0.0007 (11)
O1	0.0261 (8)	0.0255 (8)	0.0259 (8)	−0.0076 (6)	−0.0048 (6)	0.0006 (6)
C4	0.0216 (11)	0.0270 (12)	0.0297 (13)	−0.0027 (9)	−0.0049 (9)	0.0030 (10)
O2	0.0341 (9)	0.0326 (9)	0.0269 (9)	−0.0061 (7)	−0.0034 (7)	0.0005 (7)
C47	0.0313 (13)	0.0311 (13)	0.0335 (13)	−0.0067 (10)	−0.0049 (10)	−0.0009 (10)
C41	0.0285 (12)	0.0285 (13)	0.0252 (12)	−0.0057 (9)	−0.0093 (9)	0.0019 (10)
C42	0.0309 (13)	0.0271 (13)	0.0473 (15)	−0.0012 (10)	−0.0087 (11)	0.0053 (11)
C43	0.0276 (13)	0.0425 (16)	0.0462 (15)	−0.0024 (11)	−0.0038 (11)	0.0011 (12)
C44	0.0339 (14)	0.0398 (15)	0.0374 (14)	−0.0151 (11)	−0.0073 (11)	0.0045 (11)
C45	0.0413 (14)	0.0257 (13)	0.0446 (15)	−0.0090 (11)	−0.0097 (12)	0.0020 (11)
C46	0.0316 (13)	0.0287 (13)	0.0405 (14)	−0.0020 (10)	−0.0053 (10)	−0.0058 (11)
C2	0.0235 (11)	0.0232 (12)	0.0203 (11)	−0.0021 (8)	−0.0043 (8)	0.0020 (9)
C21	0.0229 (11)	0.0251 (12)	0.0213 (11)	−0.0033 (9)	−0.0034 (8)	−0.0005 (9)
C22	0.0248 (12)	0.0332 (13)	0.0293 (12)	−0.0007 (9)	−0.0054 (9)	−0.0004 (10)
C23	0.0268 (12)	0.0494 (17)	0.0318 (13)	−0.0053 (11)	−0.0105 (10)	−0.0003 (12)
C24	0.0332 (14)	0.0483 (17)	0.0326 (13)	−0.0167 (11)	−0.0048 (11)	−0.0055 (12)
C25	0.0417 (14)	0.0307 (14)	0.0319 (13)	−0.0118 (11)	−0.0045 (11)	−0.0012 (11)
C26	0.0307 (12)	0.0274 (13)	0.0264 (12)	−0.0052 (9)	−0.0078 (9)	0.0009 (10)
C3	0.0237 (11)	0.0239 (12)	0.0253 (11)	−0.0021 (9)	−0.0070 (9)	−0.0008 (9)
O3	0.0367 (9)	0.0245 (9)	0.0268 (8)	−0.0008 (7)	−0.0032 (7)	0.0038 (7)
N31	0.0265 (10)	0.0216 (10)	0.0225 (9)	−0.0010 (7)	−0.0039 (7)	−0.0001 (7)
C32	0.0268 (12)	0.0226 (12)	0.0266 (12)	0.0032 (9)	−0.0046 (9)	0.0000 (9)
C51	0.0298 (12)	0.0223 (12)	0.0320 (12)	0.0016 (9)	−0.0023 (10)	−0.0040 (10)
C52	0.0406 (14)	0.0281 (13)	0.0430 (15)	−0.0047 (11)	−0.0123 (11)	−0.0015 (11)
C53	0.0504 (16)	0.0360 (16)	0.0533 (17)	−0.0042 (12)	−0.0191 (13)	−0.0095 (13)
C54	0.0417 (16)	0.0321 (15)	0.073 (2)	−0.0103 (12)	−0.0070 (14)	−0.0118 (14)
C55	0.0496 (16)	0.0298 (15)	0.0543 (18)	−0.0061 (12)	0.0018 (13)	0.0003 (13)
C56	0.0411 (14)	0.0278 (13)	0.0403 (14)	−0.0004 (11)	−0.0043 (11)	−0.0018 (11)
C33	0.0266 (12)	0.0317 (13)	0.0310 (12)	0.0045 (10)	−0.0060 (10)	0.0003 (10)
C37	0.0413 (15)	0.0459 (17)	0.0470 (16)	0.0107 (12)	−0.0064 (12)	−0.0050 (13)
C34	0.0268 (13)	0.0456 (15)	0.0289 (13)	−0.0013 (10)	−0.0060 (10)	0.0050 (11)
O4	0.0228 (10)	0.0608 (13)	0.0701 (13)	−0.0009 (8)	−0.0059 (8)	0.0136 (10)
C35	0.0294 (13)	0.0370 (14)	0.0294 (12)	−0.0078 (10)	−0.0066 (10)	0.0026 (10)
C36	0.0256 (11)	0.0245 (12)	0.0259 (11)	−0.0043 (9)	−0.0050 (9)	0.0028 (9)
C61	0.0277 (12)	0.0216 (12)	0.0234 (11)	−0.0007 (9)	−0.0052 (9)	0.0026 (9)
C62	0.0262 (12)	0.0320 (13)	0.0296 (12)	−0.0011 (10)	−0.0044 (10)	0.0025 (10)
C63	0.0375 (14)	0.0386 (15)	0.0243 (12)	0.0001 (11)	−0.0020 (10)	0.0026 (10)
C64	0.0386 (14)	0.0370 (14)	0.0282 (13)	−0.0008 (11)	−0.0118 (10)	0.0002 (11)
C65	0.0304 (13)	0.0369 (14)	0.0310 (13)	−0.0007 (10)	−0.0084 (10)	0.0032 (11)
C66	0.0276 (12)	0.0343 (13)	0.0261 (12)	0.0014 (10)	−0.0051 (9)	0.0017 (10)

Geometric parameters (Å, º)

C1—C2	1.332 (3)	C25—H25	0.95
C1—O1	1.415 (2)	C26—H26	0.95
C1—C17	1.494 (3)	C3—O3	1.228 (2)

C17—C11	1.525 (3)	C3—N31	1.355 (3)
C17—H17A	0.99	N31—C36	1.479 (3)
C17—H17B	0.99	N31—C32	1.497 (3)
C11—C16	1.377 (3)	C32—C51	1.523 (3)
C11—C12	1.393 (3)	C32—C33	1.545 (3)
C12—C13	1.389 (3)	C32—H32	1.00
C12—H12	0.95	C51—C52	1.385 (3)
C13—C14	1.377 (3)	C51—C56	1.395 (3)
C13—H13	0.95	C52—C53	1.384 (3)
C14—C15	1.376 (3)	C52—H52	0.95
C14—H14	0.95	C53—C54	1.376 (4)
C15—C16	1.393 (3)	C53—H53	0.95
C15—H15	0.95	C54—C55	1.379 (4)
C16—H16	0.95	C54—H54	0.95
O1—C4	1.373 (3)	C55—C56	1.384 (4)
C4—O2	1.198 (3)	C55—H55	0.95
C4—C47	1.500 (3)	C56—H56	0.95
C47—C41	1.502 (3)	C33—C34	1.514 (3)
C47—H47A	0.99	C33—C37	1.518 (3)
C47—H47B	0.99	C33—H33	1.00
C41—C46	1.385 (3)	C37—H37A	0.98
C41—C42	1.391 (3)	C37—H37B	0.98
C42—C43	1.386 (3)	C37—H37C	0.98
C42—H42	0.95	C34—O4	1.209 (3)
C43—C44	1.385 (3)	C34—C35	1.502 (3)
C43—H43	0.95	C35—C36	1.524 (3)
C44—C45	1.376 (3)	C35—H35A	0.99
C44—H44	0.95	C35—H35B	0.99
C45—C46	1.386 (3)	C36—C61	1.525 (3)
C45—H45	0.95	C36—H36	1.00
C46—H46	0.95	C61—C62	1.384 (3)
C2—C3	1.519 (3)	C61—C66	1.392 (3)
C2—C21	1.485 (3)	C62—C63	1.390 (3)
C21—C26	1.392 (3)	C62—H62	0.95
C21—C22	1.395 (3)	C63—C64	1.384 (3)
C22—C23	1.380 (3)	C63—H63	0.95
C22—H22	0.95	C64—C65	1.383 (3)
C23—C24	1.378 (3)	C64—H64	0.95
C23—H23	0.95	C65—C66	1.382 (3)
C24—C25	1.382 (3)	C65—H65	0.95
C24—H24	0.95	C66—H66	0.95
C25—C26	1.386 (3)		

C2—C1—O1	114.70 (18)	O3—C3—N31	122.12 (19)
C2—C1—C17	130.9 (2)	O3—C3—C2	119.98 (19)
O1—C1—C17	114.38 (17)	N31—C3—C2	117.89 (18)
C1—C17—C11	115.99 (18)	C3—N31—C36	123.03 (18)
C1—C17—H17A	108.3	C3—N31—C32	116.59 (17)

C11—C17—H17A	108.3	C36—N31—C32	119.65 (17)
C1—C17—H17B	108.3	N31—C32—C51	112.01 (17)
C11—C17—H17B	108.3	N31—C32—C33	111.09 (17)
H17A—C17—H17B	107.4	C51—C32—C33	111.77 (18)
C16—C11—C12	118.3 (2)	N31—C32—H32	107.2
C16—C11—C17	118.8 (2)	C51—C32—H32	107.2
C12—C11—C17	122.8 (2)	C33—C32—H32	107.2
C13—C12—C11	120.4 (2)	C52—C51—C56	118.7 (2)
C13—C12—H12	119.8	C52—C51—C32	121.8 (2)
C11—C12—H12	119.8	C56—C51—C32	119.5 (2)
C14—C13—C12	120.9 (2)	C53—C52—C51	120.5 (2)
C14—C13—H13	119.5	C53—C52—H52	119.7
C12—C13—H13	119.5	C51—C52—H52	119.7
C15—C14—C13	118.9 (2)	C54—C53—C52	120.5 (3)
C15—C14—H14	120.5	C54—C53—H53	119.7
C13—C14—H14	120.5	C52—C53—H53	119.7
C14—C15—C16	120.5 (2)	C53—C54—C55	119.5 (3)
C14—C15—H15	119.8	C53—C54—H54	120.2
C16—C15—H15	119.8	C55—C54—H54	120.2
C11—C16—C15	121.1 (2)	C54—C55—C56	120.4 (3)
C11—C16—H16	119.5	C54—C55—H55	119.8
C15—C16—H16	119.5	C56—C55—H55	119.8
C4—O1—C1	117.44 (16)	C55—C56—C51	120.3 (2)
O2—C4—O1	122.7 (2)	C55—C56—H56	119.8
O2—C4—C47	128.5 (2)	C51—C56—H56	119.8
O1—C4—C47	108.83 (18)	C34—C33—C37	112.1 (2)
C4—C47—C41	115.13 (19)	C34—C33—C32	112.89 (19)
C4—C47—H47A	108.5	C37—C33—C32	110.36 (19)
C41—C47—H47A	108.5	C34—C33—H33	107.1
C4—C47—H47B	108.5	C37—C33—H33	107.1
C41—C47—H47B	108.5	C32—C33—H33	107.1
H47A—C47—H47B	107.5	C33—C37—H37A	109.5
C46—C41—C42	117.7 (2)	C33—C37—H37B	109.5
C46—C41—C47	120.9 (2)	H37A—C37—H37B	109.5
C42—C41—C47	121.3 (2)	C33—C37—H37C	109.5
C43—C42—C41	121.1 (2)	H37A—C37—H37C	109.5
C43—C42—H42	119.5	H37B—C37—H37C	109.5
C41—C42—H42	119.5	O4—C34—C35	120.7 (2)
C44—C43—C42	120.0 (2)	O4—C34—C33	122.9 (2)
C44—C43—H43	120.0	C35—C34—C33	116.39 (19)
C42—C43—H43	120.0	C34—C35—C36	114.1 (2)
C45—C44—C43	119.7 (2)	C34—C35—H35A	108.7
C45—C44—H44	120.1	C36—C35—H35A	108.7
C43—C44—H44	120.1	C34—C35—H35B	108.7
C44—C45—C46	119.7 (2)	C36—C35—H35B	108.7
C44—C45—H45	120.1	H35A—C35—H35B	107.6
C46—C45—H45	120.1	N31—C36—C35	107.59 (17)
C41—C46—C45	121.7 (2)	N31—C36—C61	111.84 (17)

C41—C46—H46	119.2	C35—C36—C61	116.73 (18)
C45—C46—H46	119.2	N31—C36—H36	106.7
C1—C2—C3	116.53 (19)	C35—C36—H36	106.7
C1—C2—C21	127.73 (19)	C61—C36—H36	106.7
C3—C2—C21	115.55 (17)	C62—C61—C66	118.4 (2)
C26—C21—C22	118.0 (2)	C62—C61—C36	122.89 (19)
C26—C21—C2	124.25 (18)	C66—C61—C36	118.71 (19)
C22—C21—C2	117.74 (19)	C61—C62—C63	120.8 (2)
C23—C22—C21	121.0 (2)	C61—C62—H62	119.6
C23—C22—H22	119.5	C63—C62—H62	119.6
C21—C22—H22	119.5	C64—C63—C62	120.2 (2)
C24—C23—C22	120.6 (2)	C64—C63—H63	119.9
C24—C23—H23	119.7	C62—C63—H63	119.9
C22—C23—H23	119.7	C65—C64—C63	119.3 (2)
C23—C24—C25	119.1 (2)	C65—C64—H64	120.3
C23—C24—H24	120.4	C63—C64—H64	120.3
C25—C24—H24	120.4	C66—C65—C64	120.3 (2)
C24—C25—C26	120.7 (2)	C66—C65—H65	119.9
C24—C25—H25	119.7	C64—C65—H65	119.9
C26—C25—H25	119.7	C65—C66—C61	121.0 (2)
C25—C26—C21	120.6 (2)	C65—C66—H66	119.5
C25—C26—H26	119.7	C61—C66—H66	119.5
C21—C26—H26	119.7		

C2—C1—C17—C11	107.1 (3)	O3—C3—N31—C36	−175.01 (18)
O1—C1—C17—C11	−71.6 (2)	C2—C3—N31—C36	3.7 (3)
C1—C17—C11—C16	−151.1 (2)	O3—C3—N31—C32	−4.9 (3)
C1—C17—C11—C12	32.8 (3)	C2—C3—N31—C32	173.88 (17)
C16—C11—C12—C13	0.8 (3)	C3—N31—C32—C51	73.9 (2)
C17—C11—C12—C13	177.0 (2)	C36—N31—C32—C51	−115.6 (2)
C11—C12—C13—C14	−0.9 (4)	C3—N31—C32—C33	−160.30 (18)
C12—C13—C14—C15	0.2 (4)	C36—N31—C32—C33	10.2 (3)
C13—C14—C15—C16	0.7 (4)	N31—C32—C51—C52	69.2 (3)
C12—C11—C16—C15	0.0 (3)	C33—C32—C51—C52	−56.2 (3)
C17—C11—C16—C15	−176.3 (2)	N31—C32—C51—C56	−111.3 (2)
C14—C15—C16—C11	−0.7 (4)	C33—C32—C51—C56	123.2 (2)
C2—C1—O1—C4	99.5 (2)	C56—C51—C52—C53	0.1 (4)
C17—C1—O1—C4	−81.5 (2)	C32—C51—C52—C53	179.5 (2)
C1—O1—C4—O2	13.8 (3)	C51—C52—C53—C54	0.0 (4)
C1—O1—C4—C47	−165.53 (17)	C52—C53—C54—C55	0.2 (4)
O2—C4—C47—C41	21.6 (3)	C53—C54—C55—C56	−0.3 (4)
O1—C4—C47—C41	−159.19 (18)	C54—C55—C56—C51	0.3 (4)
C4—C47—C41—C46	−136.4 (2)	C52—C51—C56—C55	−0.2 (3)
C4—C47—C41—C42	45.5 (3)	C32—C51—C56—C55	−179.7 (2)
C46—C41—C42—C43	−0.3 (3)	N31—C32—C33—C34	39.5 (2)
C47—C41—C42—C43	177.8 (2)	C51—C32—C33—C34	165.45 (18)
C41—C42—C43—C44	0.2 (4)	N31—C32—C33—C37	165.83 (19)
C42—C43—C44—C45	0.0 (4)	C51—C32—C33—C37	−68.3 (2)

C43—C44—C45—C46	−0.2 (4)	C37—C33—C34—O4	11.0 (3)
C42—C41—C46—C45	0.1 (3)	C32—C33—C34—O4	136.4 (2)
C47—C41—C46—C45	−178.1 (2)	C37—C33—C34—C35	−169.2 (2)
C44—C45—C46—C41	0.1 (4)	C32—C33—C34—C35	−43.8 (3)
O1—C1—C2—C3	−7.7 (3)	O4—C34—C35—C36	177.4 (2)
O1—C1—C2—C21	177.47 (18)	C33—C34—C35—C36	−2.4 (3)
C17—C1—C2—C21	−1.3 (4)	C3—N31—C36—C35	114.8 (2)
C17—C1—C2—C3	173.5 (2)	C32—N31—C36—C35	−55.0 (2)
C1—C2—C21—C26	−33.8 (3)	C3—N31—C36—C61	−115.7 (2)
C3—C2—C21—C26	151.4 (2)	C32—N31—C36—C61	74.4 (2)
C1—C2—C21—C22	148.1 (2)	C34—C35—C36—N31	49.4 (2)
C3—C2—C21—C22	−26.7 (3)	C34—C35—C36—C61	−77.2 (2)
C26—C21—C22—C23	0.5 (3)	N31—C36—C61—C62	−127.9 (2)
C2—C21—C22—C23	178.8 (2)	C35—C36—C61—C62	−3.4 (3)
C21—C22—C23—C24	0.2 (3)	N31—C36—C61—C66	54.5 (3)
C22—C23—C24—C25	−1.0 (4)	C35—C36—C61—C66	179.0 (2)
C23—C24—C25—C26	1.1 (4)	C66—C61—C62—C63	−1.6 (3)
C24—C25—C26—C21	−0.4 (3)	C36—C61—C62—C63	−179.2 (2)
C22—C21—C26—C25	−0.4 (3)	C61—C62—C63—C64	0.8 (4)
C2—C21—C26—C25	−178.5 (2)	C62—C63—C64—C65	0.4 (4)
C1—C2—C3—O3	−72.9 (3)	C63—C64—C65—C66	−0.8 (4)
C21—C2—C3—O3	102.6 (2)	C64—C65—C66—C61	−0.1 (4)
C1—C2—C3—N31	108.3 (2)	C62—C61—C66—C65	1.3 (3)
C21—C2—C3—N31	−76.2 (2)	C36—C61—C66—C65	178.9 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1	0.95	2.42	3.075 (3)	126
C47—H47*A*···O4^{i}	0.99	2.59	3.571 (3)	172
C66—H66···O1	0.95	2.53	3.469 (3)	172

Symmetry code: (i) *x*−1, *y*, *z*.