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Conformational isomers in (2*RS*,3*SR*,6*SR*)-3-ethyl-2,6-diphenyl-1-phenylacetyl-4-piperidone determined from synchrotron data at 120 K

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Key indicators

Single-crystal synchrotron study
T = 120 K
Mean σ(C–C) = 0.013 Å
R factor = 0.108
wR factor = 0.338
Data-to-parameter ratio = 10.2

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

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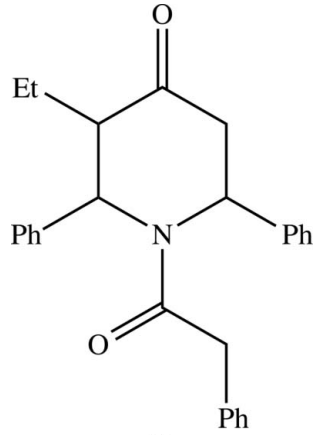
In the title compound, $C_{27}H_{27}NO_2$, the conformations of the two independent molecules differ in the shape of the piperidone rings, the orientation of the *N*-acyl substituents and the axial/equatorial disposition of the ethyl and phenyl groups. The molecules are linked into complex chains by four independent C—H···O hydrogen bonds.

Comment

In piperidone derivatives containing *N*-chloroacetyl or *N*-ethoxycarbonyl substituents, the piperidone rings generally adopt distorted boat conformations (Ponnuswamy *et al.*, 2002; Nallini *et al.*, 2003). This study of the title compound, (I) (Figs. 1 and 2), was undertaken to investigate the effect of an *N*-phenylacetyl substituent upon the molecular conformation of the piperidone ring.



(I)

Compound (I) forms extremely small crystals of rather indifferent quality, and synchrotron radiation was necessary to obtain usable diffraction data. It crystallizes with $Z' = 2$ in the space group $P2_1/c$ (Figs. 1 and 2): molecules 1 and 2 contain atoms N11 and N21, respectively. The conformations of the two independent molecules are significantly different. Firstly, the ring-puckering parameters (Cremer & Pople, 1975) for the piperidone rings are, for the atom sequences N*n*1—C*n*2—C*n*3—C*n*4—C*n*5—C*n*6, $\theta = 94.0\ (8)^\circ$ and $\varphi = 254.6\ (8)^\circ$ when $n = 1$, and $\theta = 88.3\ (7)^\circ$ and $\varphi = 124.1\ (7)^\circ$ when $n = 2$, indicating an almost ideal twist-boat conformation in molecule 2, but a conformation midway between boat and twist-boat for molecule 1. Secondly, the orientation of the *N*-acyl substituent is entirely different in the two molecules, as indicated by the leading torsion angles (Table 1). Finally, in molecule 1, the substituents at C12 and C13 are axial and that at C16 is equatorial; in molecule 2 the substituents at C22 and C23 are

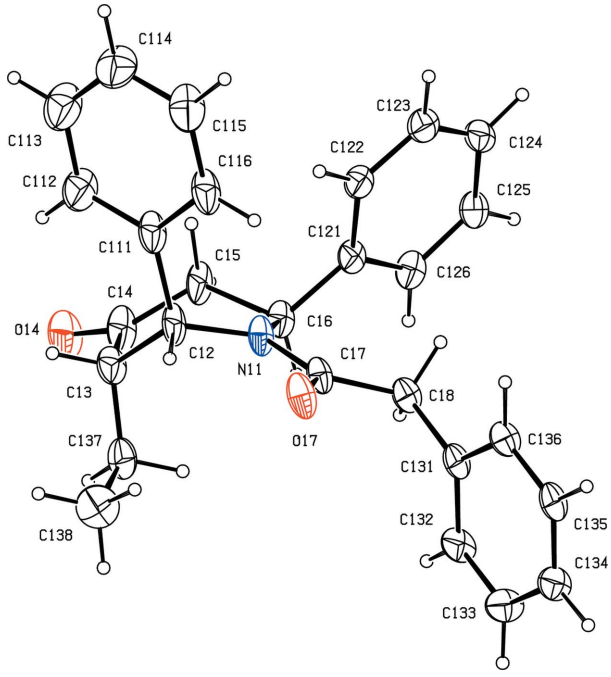


Figure 1
The *RSS* enantiomer of molecule 1 of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 20% probability level.

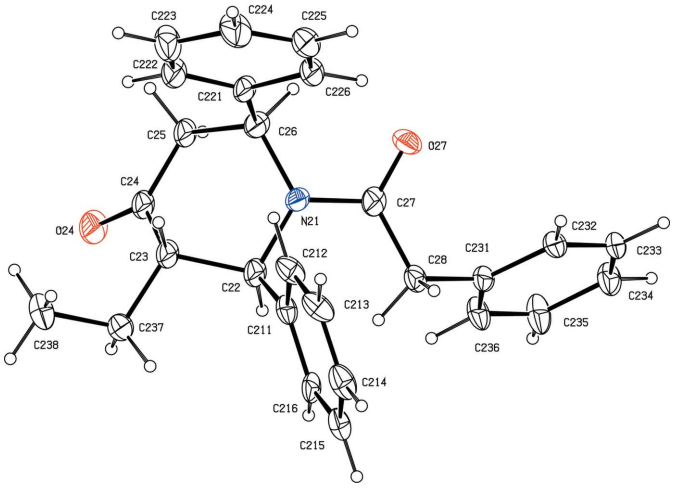


Figure 2
The *RSS* enantiomer of molecule 2 of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 20% probability level.

equatorial and that at C26 is axial (Figs. 1 and 2, and Table 1). The two molecules are thus conformational isomers and this in itself precludes the possibility of any additional crystallographic symmetry.

The molecules are linked into rather complex chains by a combination of four C—H···O hydrogen bonds (Table 2). Atoms C22 and C28 in the type 2 molecule at (x, y, z) both act as hydrogen-bond donors to atom O24 in the type 2 molecule at $(x, \frac{3}{2} - y, \frac{1}{2} + z)$, so forming a $C(4)C(5)[R_2^1(7)]$ chain of rings (Bernstein *et al.*, 1995) along [001]. This chain is formed by type 2 molecule only and the type 1 molecules are pendent

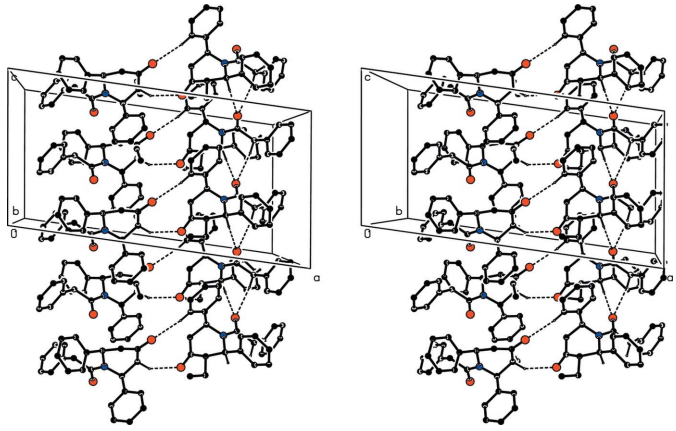


Figure 3
Stereoview of part of the crystal structure of compound (I), showing the formation of a [001] chain of rings containing only type 2 molecules, with type 1 molecules pendent from the chain. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

from it. Atoms C13 and *C*222 at (x, y, z) act as hydrogen-bond donors respectively to atoms O24 and O14 at $(1 - x, 1 - y, 1 - z)$, so forming a non-centrosymmetric $R_2^2(11)$ ring (Fig. 3). Antiparallel pairs of these chains, related to one another by inversion, are weakly linked by a single C—H···π(arene) interaction (Table 2).

Experimental

The title compound was prepared by the condensation of equimolar quantities of phenylacetyl chloride and 3-ethyl-2,6-diphenylpiperidin-4-one in anhydrous benzene, in the presence of triethylamine as the base. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in aqueous ethanol (m.p. 368–371 K).

Crystal data

$C_{27}H_{27}NO_2$
$M_r = 397.50$
Monoclinic, $P2_1/c$
$a = 19.355\ (6)$ Å
$b = 22.944\ (8)$ Å
$c = 9.959\ (3)$ Å
$\beta = 97.905\ (6)^\circ$
$V = 4381\ (2)$ Å^3
$Z = 8$
$D_x = 1.205$ Mg m^{-3}
Synchrotron radiation
$\lambda = 0.6712$ Å
Cell parameters from 1257 reflections
$\theta = 2.6–18.5^\circ$
$\mu = 0.08$ mm^{-1}
$T = 120\ (2)$ K
Lath, colourless
0.08 × 0.02 × 0.002 mm

Data collection

Bruker SMART APEX2 CCD diffractometer
Fine–slice ω scans
Absorption correction: none
16316 measured reflections
5346 independent reflections
2533 reflections with $I > 2\sigma(I)$
$R_{int} = 0.123$
$\theta_{max} = 20.7^\circ$
$h = -19 \rightarrow 20$
$k = -24 \rightarrow 24$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.108$
$wR(F^2) = 0.338$
$S = 1.04$
5346 reflections
526 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.195P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.81$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.030 (5)

Table 1
Selected torsion angles (°).

C12—C13—C137—C138	67.5 (11)	C22—C23—C237—C238	154.9 (7)
N11—C12—C111—C112	130.2 (10)	N21—C22—C211—C212	57.0 (10)
N11—C16—C121—C122	−41.2 (10)	N21—C26—C221—C222	−118.6 (9)
C12—N11—C17—C18	173.3 (7)	C22—N21—C27—C28	−4.2 (9)
N11—C17—C18—C131	−162.0 (7)	N21—C27—C28—C231	158.6 (6)
C17—C18—C131—C132	98.1 (9)	C27—C28—C231—C232	79.2 (8)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O24^{i}	1.00	2.35	3.232 (10)	147
C22—H22···O27^{ii}	1.00	2.19	3.190 (10)	175
C28—H28*A*···O27^{ii}	0.99	2.52	3.486 (9)	165
C222—H222···O14^{i}	0.95	2.54	3.478 (10)	169
C123—H123···*Cg*^{iii}	0.95	2.89	3.811 (10)	165

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iii) $x, y, z+1$. *Cg* is the centroid of ring (C221–C226)

Diffraction data were collected at the Daresbury SRS, station 9.8, UK (Cernik *et al.*, 1997; Clegg, 2000). The very small crystal size and weak diffraction led to a relatively high R_{int} and to a low $2\theta_{max}$ for the data used in the refinement. 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 $1.5U_{eq}$(C) for the methyl groups.

Data collection: *APEX2* (Bruker, 2003); cell refinement: *APEX2* and *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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 through the EPSRC X-ray Crystallographic Service at Daresbury SRS station 9.8, UK. The authors thank the staff for all their help and advice.

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Crystal data

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$V = 4381\ (2)$ Å^3
$Z = 8$
$F(000) = 1696$
$D_x = 1.205$ Mg m^{-3}
Synchrotron radiation, $\lambda = 0.6712$ Å
Cell parameters from 1257 reflections
$\theta = 2.6–18.5°$
$\mu = 0.08$ mm^{-1}
$T = 120$ K
Lath, colourless
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Data collection

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Radiation source: Daresbury SRS station 9.8
Silicon 111 monochromator
fine–slice ω scans
16316 measured reflections
5346 independent reflections
2533 reflections with $I > 2\sigma(I)$
$R_{int} = 0.123$
$\theta_{max} = 20.7°$, $\theta_{min} = 2.5°$
$h = -19\rightarrow20$
$k = -24\rightarrow24$
$l = -10\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.108$
$wR(F^2) = 0.338$
$S = 1.04$
5346 reflections
526 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
$w = 1/[\sigma^2(F_o^2) + (0.195P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.81$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.030 (5)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N11	0.6763 (3)	0.3221 (3)	0.9826 (6)	0.0563 (19)
C12	0.6095 (4)	0.3205 (5)	1.0347 (8)	0.069 (3)
C111	0.5988 (4)	0.3713 (5)	1.1295 (8)	0.075 (3)
C116	0.6501 (4)	0.3807 (5)	1.2418 (9)	0.088 (3)
C115	0.6409 (6)	0.4260 (6)	1.3317 (10)	0.104 (4)
C114	0.5824 (7)	0.4597 (6)	1.3165 (11)	0.110 (4)
C113	0.5314 (6)	0.4504 (6)	1.2081 (12)	0.118 (4)
C112	0.5390 (5)	0.4058 (5)	1.1154 (11)	0.101 (4)
C13	0.5503 (4)	0.3123 (5)	0.9160 (9)	0.071 (3)
C137	0.5544 (5)	0.2550 (5)	0.8515 (10)	0.079 (3)
C138	0.5403 (6)	0.2030 (5)	0.9362 (11)	0.104 (4)
C14	0.5511 (4)	0.3595 (5)	0.8117 (9)	0.082 (3)
O14	0.4994 (3)	0.3714 (3)	0.7273 (6)	0.099 (2)
C15	0.6148 (4)	0.3930 (4)	0.8189 (8)	0.075 (3)
C16	0.6838 (4)	0.3582 (4)	0.8596 (8)	0.058 (2)
C121	0.7431 (4)	0.4012 (3)	0.8795 (8)	0.054 (2)
C122	0.7574 (4)	0.4338 (4)	1.0009 (8)	0.059 (2)
C123	0.8106 (5)	0.4738 (4)	1.0139 (10)	0.070 (3)
C124	0.8494 (4)	0.4850 (4)	0.9134 (10)	0.063 (2)
C125	0.8363 (4)	0.4526 (4)	0.7949 (9)	0.062 (2)
C126	0.7838 (4)	0.4103 (4)	0.7785 (8)	0.060 (2)
C17	0.7275 (4)	0.2832 (4)	1.0293 (8)	0.054 (2)
O17	0.7193 (3)	0.2498 (3)	1.1245 (6)	0.0755 (19)
C18	0.7936 (4)	0.2809 (3)	0.9635 (8)	0.056 (2)
C131	0.8359 (4)	0.2263 (4)	0.9909 (8)	0.054 (2)
C132	0.8311 (4)	0.1809 (4)	0.8988 (9)	0.066 (3)
C133	0.8698 (5)	0.1317 (4)	0.9191 (9)	0.075 (3)
C134	0.9187 (4)	0.1258 (4)	1.0353 (9)	0.068 (3)
C135	0.9243 (4)	0.1694 (4)	1.1271 (9)	0.061 (2)
C136	0.8838 (4)	0.2203 (4)	1.1092 (8)	0.059 (2)
N21	0.7780 (3)	0.7033 (3)	0.3650 (6)	0.0510 (17)
C22	0.7804 (4)	0.6604 (4)	0.2513 (8)	0.0643 (13)
C211	0.8336 (4)	0.6127 (3)	0.2872 (8)	0.048 (2)
C212	0.8316 (5)	0.5773 (4)	0.3979 (8)	0.061 (2)
C213	0.8800 (5)	0.5343 (4)	0.4243 (9)	0.072 (3)
C214	0.9309 (5)	0.5248 (4)	0.3449 (10)	0.068 (3)
C215	0.9344 (4)	0.5603 (3)	0.2308 (8)	0.056 (2)
C216	0.8855 (4)	0.6039 (3)	0.2027 (8)	0.050 (2)
C23	0.7057 (4)	0.6369 (4)	0.2075 (8)	0.0643 (13)
C237	0.7021 (4)	0.6025 (4)	0.0778 (8)	0.064 (2)
C238	0.6417 (5)	0.5574 (5)	0.0659 (10)	0.096 (3)
C24	0.6532 (4)	0.6883 (4)	0.1948 (8)	0.0643 (13)
O24	0.6163 (3)	0.6997 (3)	0.0870 (5)	0.082 (2)
C25	0.6523 (4)	0.7261 (4)	0.3121 (7)	0.0643 (13)
C26	0.7137 (4)	0.7120 (4)	0.4259 (7)	0.057 (2)

C221	0.7030 (4)	0.6645 (4)	0.5269 (7)	0.062 (2)
C226	0.7553 (4)	0.6584 (4)	0.6401 (8)	0.063 (2)
C225	0.7513 (5)	0.6144 (4)	0.7334 (9)	0.072 (3)
C224	0.6975 (5)	0.5751 (5)	0.7195 (9)	0.088 (3)
C223	0.6433 (5)	0.5819 (5)	0.6104 (9)	0.095 (4)
C222	0.6475 (4)	0.6263 (5)	0.5169 (8)	0.080 (3)
C27	0.8332 (4)	0.7413 (4)	0.4046 (8)	0.052 (2)
O27	0.8300 (3)	0.7766 (3)	0.4954 (5)	0.0628 (16)
C28	0.8982 (4)	0.7358 (3)	0.3368 (7)	0.047 (2)
C231	0.9411 (4)	0.7912 (4)	0.3484 (8)	0.051 (2)
C232	0.9853 (4)	0.8049 (4)	0.4697 (7)	0.055 (2)
C233	1.0237 (4)	0.8554 (4)	0.4775 (8)	0.059 (2)
C234	1.0202 (4)	0.8941 (4)	0.3692 (8)	0.065 (2)
C235	0.9773 (5)	0.8794 (4)	0.2489 (9)	0.077 (3)
C236	0.9397 (4)	0.8286 (4)	0.2419 (8)	0.060 (2)
H12	0.6101	0.2844	1.0910	0.083*
H116	0.6905	0.3567	1.2563	0.106*
H115	0.6764	0.4335	1.4054	0.124*
H114	0.5769	0.4895	1.3804	0.132*
H113	0.4909	0.4743	1.1962	0.142*
H112	0.5030	0.3990	1.0422	0.121*
H13	0.5049	0.3145	0.9529	0.085*
H13A	0.6016	0.2507	0.8250	0.095*
H13B	0.5206	0.2544	0.7673	0.095*
H13C	0.4948	0.2074	0.9674	0.156*
H13D	0.5767	0.2001	1.0148	0.156*
H13E	0.5404	0.1674	0.8815	0.156*
H15A	0.6128	0.4249	0.8852	0.090*
H15B	0.6166	0.4111	0.7292	0.090*
H16	0.6910	0.3314	0.7835	0.069*
H122	0.7305	0.4281	1.0727	0.071*
H123	0.8207	0.4946	1.0969	0.083*
H124	0.8846	0.5142	0.9237	0.076*
H125	0.8635	0.4592	0.7238	0.074*
H126	0.7762	0.3878	0.6977	0.073*
H18A	0.7808	0.2851	0.8642	0.067*
H18B	0.8232	0.3147	0.9955	0.067*
H132	0.7993	0.1844	0.8176	0.080*
H133	0.8636	0.1011	0.8543	0.089*
H134	0.9471	0.0920	1.0493	0.082*
H135	0.9570	0.1655	1.2070	0.073*
H136	0.8887	0.2502	1.1760	0.071*
H22	0.7945	0.6824	0.1727	0.077*
H212	0.7968	0.5828	0.4553	0.073*
H213	0.8783	0.5101	0.5011	0.086*
H214	0.9638	0.4943	0.3663	0.082*
H215	0.9697	0.5545	0.1745	0.068*
H216	0.8867	0.6282	0.1258	0.060*

H23	0.6937	0.6101	0.2801	0.077*
H23A	0.7469	0.5819	0.0755	0.076*
H23B	0.6949	0.6295	−0.0005	0.076*
H23C	0.6516	0.5280	0.1373	0.144*
H23D	0.6374	0.5385	−0.0230	0.144*
H23E	0.5979	0.5774	0.0759	0.144*
H25A	0.6075	0.7212	0.3482	0.077*
H25B	0.6557	0.7673	0.2838	0.077*
H26	0.7215	0.7486	0.4807	0.069*
H226	0.7935	0.6848	0.6521	0.076*
H225	0.7868	0.6113	0.8092	0.087*
H224	0.6966	0.5440	0.7822	0.106*
H223	0.6044	0.5563	0.6008	0.114*
H222	0.6108	0.6305	0.4437	0.096*
H28A	0.8847	0.7262	0.2398	0.057*
H28B	0.9271	0.7033	0.3791	0.057*
H232	0.9884	0.7793	0.5452	0.066*
H233	1.0535	0.8642	0.5590	0.071*
H234	1.0460	0.9294	0.3766	0.078*
H235	0.9743	0.9045	0.1725	0.093*
H236	0.9115	0.8190	0.1590	0.072*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N11	0.033 (4)	0.101 (5)	0.031 (4)	0.002 (4)	−0.009 (3)	0.010 (3)
C12	0.031 (5)	0.127 (8)	0.044 (5)	0.000 (5)	−0.012 (4)	−0.004 (5)
C111	0.042 (5)	0.134 (9)	0.045 (5)	−0.002 (5)	−0.009 (4)	−0.014 (6)
C116	0.047 (6)	0.160 (10)	0.055 (6)	−0.012 (6)	−0.004 (5)	−0.016 (7)
C115	0.078 (8)	0.185 (12)	0.047 (6)	−0.011 (8)	0.005 (5)	−0.032 (7)
C114	0.102 (9)	0.154 (12)	0.075 (8)	0.012 (9)	0.014 (7)	−0.044 (7)
C113	0.086 (8)	0.176 (13)	0.090 (9)	0.022 (8)	0.003 (7)	−0.027 (9)
C112	0.068 (7)	0.148 (10)	0.078 (8)	0.019 (7)	−0.019 (6)	−0.039 (7)
C13	0.046 (5)	0.110 (8)	0.051 (6)	−0.004 (5)	−0.007 (4)	−0.014 (6)
C137	0.046 (5)	0.098 (8)	0.089 (7)	0.004 (5)	−0.006 (5)	0.006 (7)
C138	0.111 (9)	0.103 (9)	0.097 (9)	0.001 (7)	0.007 (7)	0.016 (7)
C14	0.046 (6)	0.158 (10)	0.037 (5)	0.009 (6)	−0.016 (4)	0.000 (6)
O14	0.061 (4)	0.154 (7)	0.070 (4)	0.003 (4)	−0.031 (4)	0.007 (4)
C15	0.033 (5)	0.137 (9)	0.048 (5)	0.015 (5)	−0.020 (4)	−0.006 (5)
C16	0.041 (5)	0.094 (7)	0.034 (5)	0.004 (4)	−0.008 (4)	0.002 (4)
C121	0.042 (5)	0.071 (6)	0.044 (5)	0.009 (4)	−0.014 (4)	0.003 (4)
C122	0.050 (5)	0.081 (6)	0.045 (5)	0.010 (5)	−0.002 (4)	−0.011 (4)
C123	0.055 (6)	0.082 (7)	0.066 (6)	0.013 (5)	−0.012 (5)	−0.011 (5)
C124	0.050 (5)	0.060 (6)	0.072 (7)	0.003 (4)	−0.018 (5)	0.000 (5)
C125	0.047 (5)	0.080 (6)	0.056 (6)	0.005 (5)	−0.001 (4)	0.014 (5)
C126	0.053 (5)	0.086 (7)	0.039 (5)	0.016 (5)	−0.005 (4)	0.011 (4)
C17	0.039 (5)	0.087 (6)	0.032 (4)	−0.009 (4)	−0.007 (4)	0.006 (5)
O17	0.047 (4)	0.123 (5)	0.051 (4)	−0.015 (3)	−0.012 (3)	0.023 (4)

C18	0.050 (5)	0.068 (6)	0.046 (5)	−0.006 (4)	−0.009 (4)	0.012 (4)
C131	0.039 (5)	0.069 (6)	0.050 (5)	−0.006 (4)	−0.012 (4)	0.016 (5)
C132	0.060 (6)	0.075 (7)	0.053 (5)	0.002 (5)	−0.031 (4)	0.006 (5)
C133	0.080 (6)	0.069 (6)	0.064 (6)	0.013 (5)	−0.028 (5)	−0.004 (5)
C134	0.061 (6)	0.064 (6)	0.073 (7)	0.006 (5)	−0.015 (5)	0.005 (5)
C135	0.041 (5)	0.081 (7)	0.055 (5)	−0.008 (5)	−0.015 (4)	0.019 (5)
C136	0.054 (5)	0.075 (6)	0.045 (5)	−0.007 (5)	−0.008 (4)	0.006 (4)
N21	0.032 (4)	0.089 (5)	0.031 (3)	0.007 (3)	0.000 (3)	0.004 (3)
C22	0.037 (2)	0.115 (4)	0.037 (2)	−0.006 (2)	−0.0076 (19)	0.001 (2)
C211	0.046 (5)	0.055 (5)	0.037 (5)	−0.004 (4)	−0.013 (4)	0.003 (4)
C212	0.076 (6)	0.069 (6)	0.034 (5)	0.005 (5)	−0.006 (4)	−0.001 (4)
C213	0.096 (7)	0.082 (7)	0.034 (5)	0.001 (6)	−0.004 (5)	−0.004 (5)
C214	0.077 (7)	0.055 (6)	0.064 (6)	0.009 (5)	−0.023 (5)	−0.007 (5)
C215	0.055 (5)	0.053 (5)	0.054 (5)	−0.002 (4)	−0.017 (4)	−0.007 (4)
C216	0.033 (4)	0.067 (6)	0.044 (5)	−0.006 (4)	−0.015 (4)	0.007 (4)
C23	0.037 (2)	0.115 (4)	0.037 (2)	−0.006 (2)	−0.0076 (19)	0.001 (2)
C237	0.050 (5)	0.098 (7)	0.041 (5)	−0.002 (5)	0.000 (4)	−0.005 (4)
C238	0.072 (7)	0.127 (9)	0.082 (7)	−0.017 (6)	−0.014 (5)	−0.036 (6)
C24	0.037 (2)	0.115 (4)	0.037 (2)	−0.006 (2)	−0.0076 (19)	0.001 (2)
O24	0.056 (4)	0.139 (6)	0.045 (3)	0.001 (3)	−0.014 (3)	0.013 (3)
C25	0.037 (2)	0.115 (4)	0.037 (2)	−0.006 (2)	−0.0076 (19)	0.001 (2)
C26	0.039 (5)	0.099 (7)	0.032 (4)	0.006 (4)	−0.002 (4)	0.012 (4)
C221	0.032 (5)	0.120 (8)	0.031 (5)	−0.003 (5)	−0.004 (4)	0.004 (5)
C226	0.048 (5)	0.104 (7)	0.034 (5)	−0.004 (5)	−0.007 (4)	0.001 (5)
C225	0.066 (6)	0.108 (8)	0.040 (5)	−0.002 (6)	−0.004 (4)	0.002 (5)
C224	0.097 (8)	0.123 (9)	0.041 (6)	−0.022 (7)	−0.002 (5)	0.016 (5)
C223	0.085 (7)	0.151 (10)	0.047 (6)	−0.051 (7)	−0.002 (5)	0.005 (7)
C222	0.052 (6)	0.153 (10)	0.033 (5)	−0.018 (6)	−0.005 (4)	0.004 (6)
C27	0.041 (5)	0.076 (6)	0.036 (5)	0.006 (4)	−0.011 (4)	0.016 (5)
O27	0.065 (4)	0.084 (4)	0.038 (3)	0.009 (3)	0.002 (3)	−0.014 (3)
C28	0.044 (5)	0.056 (5)	0.038 (4)	0.001 (4)	−0.008 (4)	−0.001 (4)
C231	0.034 (4)	0.069 (6)	0.045 (5)	0.006 (4)	−0.007 (4)	−0.012 (4)
C232	0.044 (5)	0.084 (6)	0.033 (4)	0.005 (5)	−0.008 (4)	−0.004 (4)
C233	0.038 (5)	0.091 (7)	0.046 (5)	−0.006 (5)	−0.006 (4)	−0.021 (5)
C234	0.056 (5)	0.080 (6)	0.053 (6)	−0.007 (5)	−0.015 (4)	−0.001 (5)
C235	0.075 (6)	0.082 (7)	0.065 (6)	−0.022 (5)	−0.027 (5)	0.011 (5)
C236	0.063 (6)	0.070 (6)	0.039 (5)	−0.010 (5)	−0.020 (4)	0.005 (5)

Geometric parameters (Å, º)

N11—C17	1.366 (10)	N21—C27	1.393 (10)
N11—C12	1.457 (9)	N21—C26	1.473 (9)
N11—C16	1.502 (9)	N21—C22	1.504 (10)
C12—C111	1.531 (13)	C22—C211	1.512 (11)
C12—C13	1.541 (10)	C22—C23	1.549 (10)
C12—H12	1.00	C22—H22	1.00
C111—C112	1.393 (13)	C211—C212	1.374 (11)
C111—C116	1.407 (11)	C211—C216	1.410 (11)

C116—C115	1.400 (14)	C212—C213	1.360 (12)
C116—H116	0.95	C212—H212	0.95
C115—C114	1.362 (14)	C213—C214	1.363 (12)
C115—H115	0.95	C213—H213	0.95
C114—C113	1.376 (15)	C214—C215	1.408 (12)
C114—H114	0.95	C214—H214	0.95
C113—C112	1.399 (15)	C215—C216	1.379 (10)
C113—H113	0.95	C215—H215	0.95
C112—H112	0.95	C216—H216	0.95
C13—C137	1.469 (12)	C23—C237	1.507 (11)
C13—C14	1.501 (13)	C23—C24	1.552 (12)
C13—H13	1.00	C23—H23	1.00
C137—C138	1.509 (13)	C237—C238	1.554 (12)
C137—H13A	0.99	C237—H23A	0.99
C137—H13B	0.99	C237—H23B	0.99
C138—H13C	0.98	C238—H23C	0.98
C138—H13D	0.98	C238—H23D	0.98
C138—H13E	0.98	C238—H23E	0.98
C14—O14	1.246 (9)	C24—O24	1.233 (9)
C14—C15	1.446 (12)	C24—C25	1.457 (11)
C15—C16	1.562 (11)	C25—C26	1.559 (10)
C15—H15A	0.99	C25—H25A	0.99
C15—H15B	0.99	C25—H25B	0.99
C16—C121	1.506 (11)	C26—C221	1.517 (11)
C16—H16	1.00	C26—H26	1.00
C121—C126	1.376 (11)	C221—C222	1.380 (12)
C121—C122	1.417 (11)	C221—C226	1.415 (10)
C122—C123	1.372 (12)	C226—C225	1.382 (11)
C122—H122	0.95	C226—H226	0.95
C123—C124	1.356 (12)	C225—C224	1.370 (12)
C123—H123	0.95	C225—H225	0.95
C124—C125	1.389 (11)	C224—C223	1.411 (13)
C124—H124	0.95	C224—H224	0.95
C125—C126	1.400 (11)	C223—C222	1.390 (13)
C125—H125	0.95	C223—H223	0.95
C126—H126	0.95	C222—H222	0.95
C17—O17	1.246 (9)	C27—O27	1.221 (9)
C17—C18	1.517 (11)	C27—C28	1.514 (11)
C18—C131	1.502 (11)	C28—C231	1.514 (10)
C18—H18A	0.99	C28—H28A	0.99
C18—H18B	0.99	C28—H28B	0.99
C131—C132	1.382 (11)	C231—C236	1.361 (11)
C131—C136	1.401 (10)	C231—C232	1.417 (10)
C132—C133	1.354 (11)	C232—C233	1.373 (11)
C132—H132	0.95	C232—H232	0.95
C133—C134	1.397 (11)	C233—C234	1.390 (11)
C133—H133	0.95	C233—H233	0.95
C134—C135	1.349 (11)	C234—C235	1.402 (11)

C134—H134	0.95	C234—H234	0.95
C135—C136	1.405 (11)	C235—C236	1.371 (11)
C135—H135	0.95	C235—H235	0.95
C136—H136	0.95	C236—H236	0.95
C17—N11—C12	120.0 (7)	C27—N21—C26	117.1 (7)
C17—N11—C16	119.3 (6)	C27—N21—C22	121.4 (6)
C12—N11—C16	119.6 (6)	C26—N21—C22	121.0 (6)
N11—C12—C111	113.8 (7)	N21—C22—C211	112.7 (6)
N11—C12—C13	109.4 (7)	N21—C22—C23	108.4 (6)
C111—C12—C13	114.6 (7)	C211—C22—C23	113.1 (7)
N11—C12—H12	106.1	N21—C22—H22	107.5
C111—C12—H12	106.1	C211—C22—H22	107.5
C13—C12—H12	106.1	C23—C22—H22	107.5
C112—C111—C116	118.6 (9)	C212—C211—C216	119.7 (7)
C112—C111—C12	123.5 (7)	C212—C211—C22	121.7 (8)
C116—C111—C12	117.7 (9)	C216—C211—C22	118.6 (7)
C115—C116—C111	118.9 (10)	C213—C212—C211	119.3 (9)
C115—C116—H116	120.5	C213—C212—H212	120.4
C111—C116—H116	120.5	C211—C212—H212	120.4
C114—C115—C116	121.9 (9)	C212—C213—C214	122.5 (9)
C114—C115—H115	119.0	C212—C213—H213	118.7
C116—C115—H115	119.0	C214—C213—H213	118.7
C115—C114—C113	119.6 (11)	C213—C214—C215	119.6 (8)
C115—C114—H114	120.2	C213—C214—H214	120.2
C113—C114—H114	120.2	C215—C214—H214	120.2
C114—C113—C112	120.1 (11)	C216—C215—C214	118.4 (8)
C114—C113—H113	119.9	C216—C215—H215	120.8
C112—C113—H113	119.9	C214—C215—H215	120.8
C111—C112—C113	120.7 (9)	C215—C216—C211	120.5 (7)
C111—C112—H112	119.7	C215—C216—H216	119.8
C113—C112—H112	119.7	C211—C216—H216	119.8
C137—C13—C14	109.7 (8)	C237—C23—C22	110.8 (7)
C137—C13—C12	111.5 (8)	C237—C23—C24	112.1 (6)
C14—C13—C12	111.4 (8)	C22—C23—C24	109.4 (7)
C137—C13—H13	108.0	C237—C23—H23	108.1
C14—C13—H13	108.0	C22—C23—H23	108.1
C12—C13—H13	108.0	C24—C23—H23	108.1
C13—C137—C138	116.0 (9)	C23—C237—C238	111.0 (7)
C13—C137—H13A	108.3	C23—C237—H23A	109.4
C138—C137—H13A	108.3	C238—C237—H23A	109.4
C13—C137—H13B	108.3	C23—C237—H23B	109.4
C138—C137—H13B	108.3	C238—C237—H23B	109.4
H13A—C137—H13B	107.4	H23A—C237—H23B	108.0
C137—C138—H13C	109.5	C237—C238—H23C	109.5
C137—C138—H13D	109.5	C237—C238—H23D	109.5
H13C—C138—H13D	109.5	H23C—C238—H23D	109.5
C137—C138—H13E	109.5	C237—C238—H23E	109.5

H13C—C138—H13E	109.5	H23C—C238—H23E	109.5
H13D—C138—H13E	109.5	H23D—C238—H23E	109.5
O14—C14—C15	121.2 (10)	O24—C24—C25	120.1 (8)
O14—C14—C13	122.8 (9)	O24—C24—C23	121.8 (8)
C15—C14—C13	116.0 (7)	C25—C24—C23	117.8 (7)
C14—C15—C16	115.8 (8)	C24—C25—C26	111.6 (7)
C14—C15—H15A	108.3	C24—C25—H25A	109.3
C16—C15—H15A	108.3	C26—C25—H25A	109.3
C14—C15—H15B	108.3	C24—C25—H25B	109.3
C16—C15—H15B	108.3	C26—C25—H25B	109.3
H15A—C15—H15B	107.4	H25A—C25—H25B	108.0
N11—C16—C121	114.3 (6)	N21—C26—C221	112.0 (6)
N11—C16—C15	108.5 (6)	N21—C26—C25	109.4 (6)
C121—C16—C15	107.9 (7)	C221—C26—C25	118.1 (7)
N11—C16—H16	108.6	N21—C26—H26	105.4
C121—C16—H16	108.6	C221—C26—H26	105.4
C15—C16—H16	108.6	C25—C26—H26	105.4
C126—C121—C122	118.6 (8)	C222—C221—C226	117.4 (8)
C126—C121—C16	120.3 (7)	C222—C221—C26	125.8 (7)
C122—C121—C16	121.0 (8)	C226—C221—C26	116.8 (8)
C123—C122—C121	119.4 (9)	C225—C226—C221	120.5 (8)
C123—C122—H122	120.3	C225—C226—H226	119.8
C121—C122—H122	120.3	C221—C226—H226	119.8
C124—C123—C122	122.8 (8)	C224—C225—C226	121.7 (8)
C124—C123—H123	118.6	C224—C225—H225	119.2
C122—C123—H123	118.6	C226—C225—H225	119.2
C123—C124—C125	118.1 (8)	C225—C224—C223	118.7 (10)
C123—C124—H124	120.9	C225—C224—H224	120.7
C125—C124—H124	120.9	C223—C224—H224	120.7
C124—C125—C126	121.0 (9)	C222—C223—C224	119.4 (9)
C124—C125—H125	119.5	C222—C223—H223	120.3
C126—C125—H125	119.5	C224—C223—H223	120.3
C121—C126—C125	120.0 (8)	C221—C222—C223	122.3 (8)
C121—C126—H126	120.0	C221—C222—H222	118.9
C125—C126—H126	120.0	C223—C222—H222	118.9
O17—C17—N11	119.9 (7)	O27—C27—N21	120.7 (7)
O17—C17—C18	120.7 (7)	O27—C27—C28	120.9 (7)
N11—C17—C18	119.4 (7)	N21—C27—C28	118.2 (8)
C131—C18—C17	115.0 (7)	C27—C28—C231	112.1 (6)
C131—C18—H18A	108.5	C27—C28—H28A	109.2
C17—C18—H18A	108.5	C231—C28—H28A	109.2
C131—C18—H18B	108.5	C27—C28—H28B	109.2
C17—C18—H18B	108.5	C231—C28—H28B	109.2
H18A—C18—H18B	107.5	H28A—C28—H28B	107.9
C132—C131—C136	117.5 (8)	C236—C231—C232	118.0 (8)
C132—C131—C18	121.5 (7)	C236—C231—C28	121.2 (7)
C136—C131—C18	121.0 (8)	C232—C231—C28	120.8 (8)
C133—C132—C131	122.9 (7)	C233—C232—C231	119.6 (8)

C133—C132—H132	118.6	C233—C232—H232	120.2
C131—C132—H132	118.6	C231—C232—H232	120.2
C132—C133—C134	120.1 (9)	C232—C233—C234	121.7 (7)
C132—C133—H133	119.9	C232—C233—H233	119.1
C134—C133—H133	119.9	C234—C233—H233	119.1
C135—C134—C133	118.2 (8)	C233—C234—C235	118.0 (8)
C135—C134—H134	120.9	C233—C234—H234	121.0
C133—C134—H134	120.9	C235—C234—H234	121.0
C134—C135—C136	122.6 (7)	C236—C235—C234	119.7 (8)
C134—C135—H135	118.7	C236—C235—H235	120.1
C136—C135—H135	118.7	C234—C235—H235	120.1
C131—C136—C135	118.7 (8)	C231—C236—C235	122.9 (7)
C131—C136—H136	120.6	C231—C236—H236	118.6
C135—C136—H136	120.6	C235—C236—H236	118.6

C17—N11—C12—C111	107.5 (9)	C27—N21—C22—C211	68.9 (9)
C16—N11—C12—C111	−84.3 (8)	C26—N21—C22—C211	−119.5 (7)
C17—N11—C12—C13	−122.9 (8)	C27—N21—C22—C23	−165.2 (6)
C16—N11—C12—C13	45.3 (11)	C26—N21—C22—C23	6.4 (10)
C12—C13—C137—C138	67.5 (11)	C22—C23—C237—C238	154.9 (7)
N11—C12—C111—C112	130.2 (10)	N21—C22—C211—C212	57.0 (10)
N11—C16—C121—C122	−41.2 (10)	N21—C26—C221—C222	−118.6 (9)
C12—N11—C17—C18	173.3 (7)	C22—N21—C27—C28	−4.2 (9)
N11—C17—C18—C131	−162.0 (7)	N21—C27—C28—C231	158.6 (6)
C17—C18—C131—C132	98.1 (9)	C27—C28—C231—C232	79.2 (8)
C13—C12—C111—C112	3.3 (14)	C23—C22—C211—C212	−66.4 (9)
N11—C12—C111—C116	−54.3 (11)	N21—C22—C211—C216	−124.2 (7)
C13—C12—C111—C116	178.7 (9)	C23—C22—C211—C216	112.4 (8)
C112—C111—C116—C115	−2.9 (16)	C216—C211—C212—C213	0.1 (11)
C12—C111—C116—C115	−178.6 (9)	C22—C211—C212—C213	178.9 (7)
C111—C116—C115—C114	2.6 (17)	C211—C212—C213—C214	0.0 (13)
C116—C115—C114—C113	−2 (2)	C212—C213—C214—C215	0.1 (13)
C115—C114—C113—C112	1 (2)	C213—C214—C215—C216	−0.4 (11)
C116—C111—C112—C113	2.5 (17)	C214—C215—C216—C211	0.5 (10)
C12—C111—C112—C113	177.9 (11)	C212—C211—C216—C215	−0.4 (11)
C114—C113—C112—C111	−1.6 (19)	C22—C211—C216—C215	−179.2 (6)
N11—C12—C13—C137	65.9 (11)	N21—C22—C23—C237	169.5 (7)
C111—C12—C13—C137	−165.0 (8)	C211—C22—C23—C237	−64.8 (9)
N11—C12—C13—C14	−57.0 (10)	N21—C22—C23—C24	45.4 (8)
C111—C12—C13—C14	72.1 (10)	C211—C22—C23—C24	171.1 (7)
C14—C13—C137—C138	−168.7 (8)	C24—C23—C237—C238	−82.5 (9)
C137—C13—C14—O14	75.7 (12)	C237—C23—C24—O24	−3.3 (12)
C12—C13—C14—O14	−160.4 (9)	C22—C23—C24—O24	120.1 (8)
C137—C13—C14—C15	−107.3 (9)	C237—C23—C24—C25	−178.5 (7)
C12—C13—C14—C15	16.6 (12)	C22—C23—C24—C25	−55.2 (10)
O14—C14—C15—C16	−146.4 (9)	O24—C24—C25—C26	−167.3 (7)
C13—C14—C15—C16	36.6 (11)	C23—C24—C25—C26	8.0 (11)
C17—N11—C16—C121	−66.1 (9)	C27—N21—C26—C221	−108.4 (8)

C12—N11—C16—C121	125.7 (7)	C22—N21—C26—C221	79.6 (8)
C17—N11—C16—C15	173.4 (7)	C27—N21—C26—C25	118.6 (7)
C12—N11—C16—C15	5.2 (10)	C22—N21—C26—C25	−53.3 (10)
C14—C15—C16—N11	−48.2 (9)	C24—C25—C26—N21	43.3 (10)
C14—C15—C16—C121	−172.7 (7)	C24—C25—C26—C221	−86.3 (9)
N11—C16—C121—C126	139.8 (7)	C25—C26—C221—C222	9.9 (13)
C15—C16—C121—C126	−99.3 (8)	N21—C26—C221—C226	60.5 (10)
C15—C16—C121—C122	79.7 (8)	C25—C26—C221—C226	−171.0 (8)
C126—C121—C122—C123	0.8 (11)	C222—C221—C226—C225	2.3 (13)
C16—C121—C122—C123	−178.2 (7)	C26—C221—C226—C225	−176.9 (8)
C121—C122—C123—C124	1.9 (12)	C221—C226—C225—C224	0.5 (14)
C122—C123—C124—C125	−2.9 (12)	C226—C225—C224—C223	−3.0 (15)
C123—C124—C125—C126	1.1 (11)	C225—C224—C223—C222	2.8 (16)
C122—C121—C126—C125	−2.4 (11)	C226—C221—C222—C223	−2.5 (15)
C16—C121—C126—C125	176.6 (7)	C26—C221—C222—C223	176.6 (9)
C124—C125—C126—C121	1.5 (11)	C224—C223—C222—C221	0.0 (17)
C12—N11—C17—O17	−6.0 (11)	C26—N21—C27—O27	6.7 (10)
C16—N11—C17—O17	−174.2 (7)	C22—N21—C27—O27	178.7 (7)
C16—N11—C17—C18	5.1 (10)	C26—N21—C27—C28	−176.1 (6)
O17—C17—C18—C131	17.3 (11)	O27—C27—C28—C231	−24.2 (9)
C17—C18—C131—C136	−84.2 (9)	C27—C28—C231—C236	−102.5 (8)
C136—C131—C132—C133	0.7 (13)	C236—C231—C232—C233	1.6 (11)
C18—C131—C132—C133	178.4 (8)	C28—C231—C232—C233	179.9 (7)
C131—C132—C133—C134	−2.0 (15)	C231—C232—C233—C234	0.4 (12)
C132—C133—C134—C135	2.0 (14)	C232—C233—C234—C235	−1.7 (13)
C133—C134—C135—C136	−0.8 (13)	C233—C234—C235—C236	1.1 (14)
C132—C131—C136—C135	0.6 (12)	C232—C231—C236—C235	−2.3 (13)
C18—C131—C136—C135	−177.2 (7)	C28—C231—C236—C235	179.4 (8)
C134—C135—C136—C131	−0.5 (12)	C234—C235—C236—C231	1.0 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···O24^{i}	1.00	2.35	3.232 (10)	147
C22—H22···O27^{ii}	1.00	2.19	3.190 (10)	175
C28—H28*A*···O27^{ii}	0.99	2.52	3.486 (9)	165
C222—H222···O14^{i}	0.95	2.54	3.478 (10)	169
C123—H123···*Cg*^{iii}	0.95	2.89	3.811 (10)	165

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*, −*y*+3/2, *z*−1/2; (iii) *x*, *y*, *z*+1.