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

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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.

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)
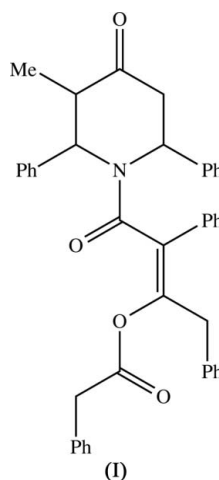
The piperidone ring adopts a boat conformation with a pseudo-mirror plane containing atoms C33 and C36; the ring-puckering parameters (Cremer & Pople, 1975) for the atom sequence N31–C32–C33–C34–C35–C36 are θ = 94.1 (2)° and φ = 122.0 (2)°, very close to the ideal values for a boat conformation of θ = 90° and φ = (60n)°. 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
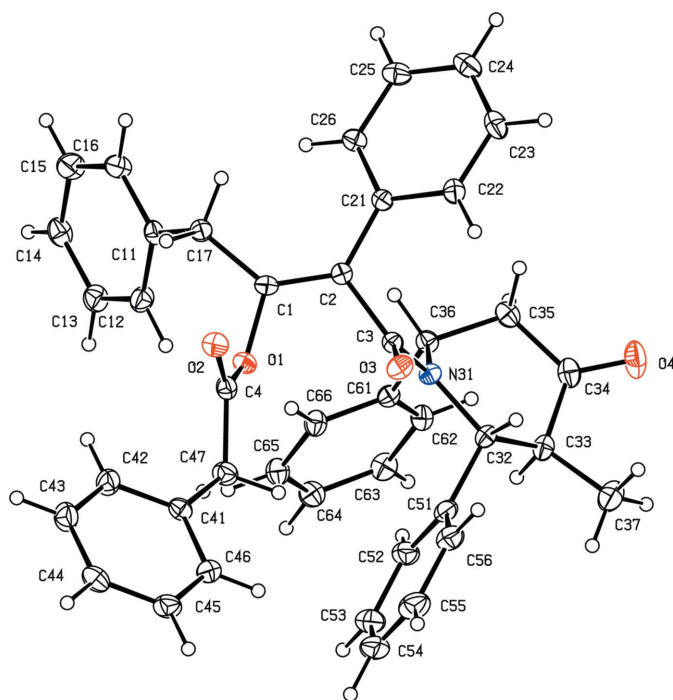
Figure 1
The *RSS* enantiomer of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.
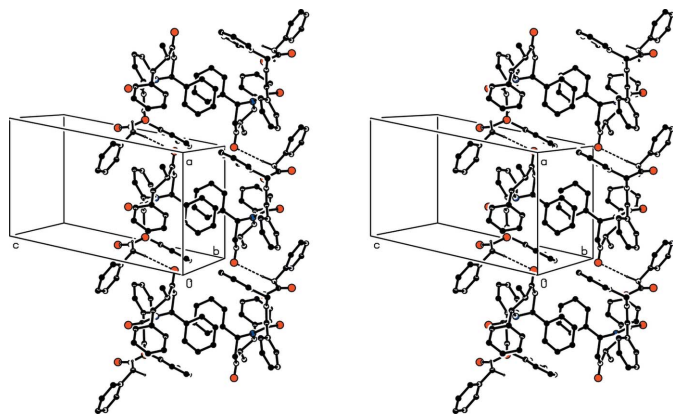

Figure 2
A stereoscopic view of part of the crystal structure of (I), showing the formation of a π-stacked pair of $C(12)$ chains along [100]. For the sake of clarity, H atoms not involved in the motif shown have been omitted.

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

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

Data collection

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

Refinement

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

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—H47A⋯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).

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