(−)-(4*R*,5*S*)-4-Methyl-3-[2(*S*)-phenoxypropionyl]-5-phenyloxazolidin-2-one

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The title compound, $C_{19}H_{19}NO_4$, formed from enantiomerically pure (+)-(4*R*,5*S*)-4-methyl-5-phenyl-2-oxazolidinone and racemic 2-phenoxypropanoyl chloride, crystallises with $Z' = 2$. The two carbonyl groups in each molecule are oriented *anti* to each other, while the two methyl groups are oriented *syn* to each other.

Comment

The title compound, (I), is the sixth in a series of structurally related compounds, introduced in our earlier report (Coumbarides, Eames *et al.*, 2006*a*). With $R^1 = C_6H_5O$, the reaction shown in that report yielded the *anti–syn* and *syn–syn* diastereomers in 44 and 45% yields, respectively. The *syn–syn* diastereomer was described previously (Coumbarides, Eames *et al.*, 2006*b*). The title compound, (I), is the *anti–syn* diastereomer.

(I)
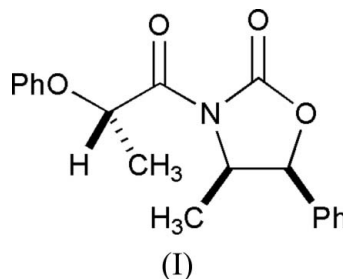
Compound (I) contains two molecules in the asymmetric unit in the space group $P2_1$, displaying closely comparable conformations (Fig. 1). In line with our previous reports (Coumbarides, Eames *et al.*, 2006*a*,*b*; Coumbarides, Dingjan *et al.*, 2006; Chavda *et al.*, 2006*a*,*b*), the carbonyl groups (C3*A*=O2*A*/C11*A*=O3*A* and C3*B*=O2*B*/C11*B*=O3*B*) are oriented *anti* to each other, with torsion angles O3—C11—N1—C3 = 176.5 (2) and 175.9 (2)° for molecules *A* and *B*, respectively. The two methyl groups (C4*A*/C19*A* and C4*B*/C19*B*) lie to the same side of the molecule. The principal distinction between the two independent molecules of (I) lies in the orientations of the phenoxypropionyl substituent with respect to the central portion of the molecule, with torsion angles O3—C11—C12—O4 = −25.6 (3) and −14.6 (3)° for molecules *A* and *B*, respectively. This subtle difference can be attributed to the influence of intermolecular interactions in the solid state. As observed in the phenylpropionyl derivative (Coumbarides, Eames *et al.*, 2006*a*), adjacent molecules approach each other in a 'side-on' manner (Fig. 2), and the shortest intermolecular contacts are C—H⋯O interactions (Table 1).

Experimental

The title compound was obtained from the same synthesis as the *syn–syn* diastereomer, as reported previously (Coumbarides, Eames *et al.*, 2006*b*). The *anti–syn* diastereomer, (I), was obtained as colourless crystals {4.03 g, 44% yield, m.p. 362–365 K, R_F 0.61 [light petroleum (b.p 313–333 K)/diethyl ether, 1:1]}. Spectroscopic analysis: $[\alpha]_D^{22}$ = −35.5 (CHCl$_3$, 293 K, concentration 2.0 g per 100 ml); IR (CHCl$_3$, ν_{max}, cm^{-1}): 1776 (C=O), 1714 (C=O); ^{1}H NMR (270 MHz, CDCl$_3$): δ 7.45–7.21 (7H, *m*, 7 × CH; Ph$_a$ and Ph$_b$), 6.97–6.86 (3H, *m*, 3 × CH; Ph$_a$ or Ph$_b$), 5.94 (1H, *q*, *J* = 6.7 Hz, PhOCH), 5.75 (1H, *d*, *J* = 7.4 Hz, PhCHO), 4.81 (1H, *m*, CHN), 1.66 (3H, *d*, *J* = 6.7 Hz, CH$_3$CHCO), 0.89 (3H, *d*, *J* = 6.6 Hz, CH$_3$CHN); ^{13}C NMR (67.9 MHz, CDCl$_3$, δ, p.p.m.): 172.0 (NC=O), 157.4 (*i*-CO; Ph), 152.8 (OC=O), 133.1 (*i*-C; Ph), 129.7, 129.0, 128.9, 125.7, 121.6, 115.1 (6 × CH; Ph$_a$ and Ph$_b$), 79.8 (PhCHO), 71.8 (PhOCH), 54.7 (CHN), 18.3 (CH$_3$), 14.5 (CH$_3$); found: MH$^+$ 326.1393; C$_{19}$H$_{20}$NO$_4$ requires 326.1392.

Crystal data

C$_{19}$H$_{19}$NO$_4$	$Z = 4$
$M_r = 325.35$	$D_x = 1.250$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 11.268\ (7)$ Å	$\mu = 0.09$ mm^{-1}
$b = 10.493\ (5)$ Å	$T = 160\ (2)$ K
$c = 15.008\ (9)$ Å	Block, colourless
$\beta = 102.96\ (4)°$	$0.44 \times 0.22 \times 0.20$ mm
$V = 1729.3\ (17)$ Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	2662 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{int} = 0.020$
Absorption correction: none	$\theta_{max} = 25.0°$
8490 measured reflections	2 standard reflections
3223 independent reflections	every 100 reflections
	intensity decay: 2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0293P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.029$	$+ 0.3025P]$
$wR(F^2) = 0.067$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
3223 reflections	$\Delta\rho_{max} = 0.13$ e Å^{-3}
438 parameters	$\Delta\rho_{min} = -0.14$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL97*
	Extinction coefficient: 0.0167 (13)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C1A—H1A⋯O3B	1.00	2.61	3.373 (3)	133
C1B—H1B⋯O3A	1.00	2.57	3.568 (3)	174
C18A—H18A⋯O4B^{i}	0.95	2.52	3.445 (4)	165
C18B—H18B⋯O4A^{ii}	0.95	2.69	3.564 (4)	154

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

H atoms were placed in geometrically idealised positions and constrained to ride on their parent atoms, with C—H = 0.95–1.00 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate about their local threefold axes. In the absence of significant anomalous scattering effects, the few measured Friedel pairs have been merged. The absolute configuration is assigned on the basis of the known configuration of the starting material (Coumbarides, Eames *et al.*, 2006*a*).
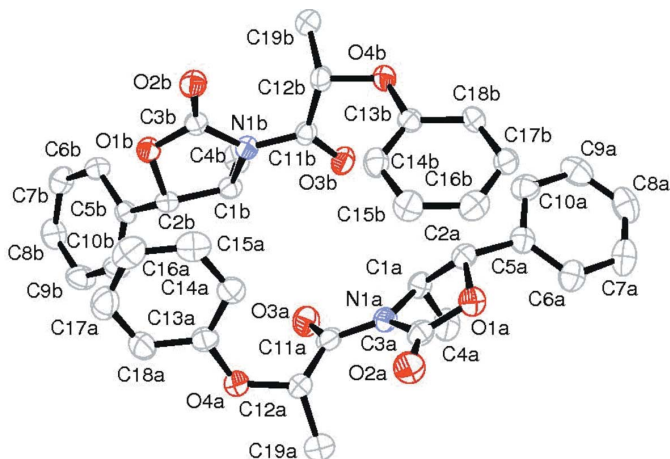

Figure 1
Two molecules in the asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted.
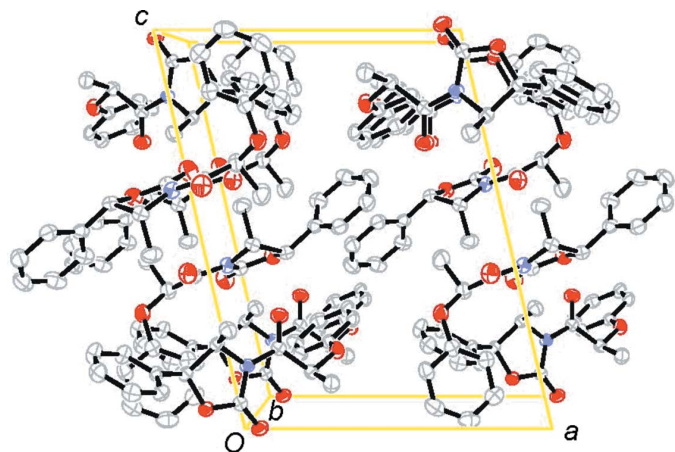

Figure 2
The crystal packing of (I) viewed along the *b* axis. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted.

Data collection: *CAD-4-PC* Software (Enraf–Nonius, 1994); cell refinement: *CAD-4-PC* Software; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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