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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$

R factor = 0.040

wR factor = 0.089

Data-to-parameter ratio = 7.6

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

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

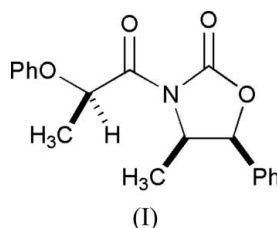
In the title compound, $\text{C}_{19}\text{H}_{19}\text{NO}_4$, formed from enantiomerically pure (+)-(4*R*,5*S*)-4-methyl-5-phenyl-2-oxazolidinone and racemic 2-phenoxypropanoyl chloride, the two carbonyl groups are oriented *anti* to each other, and the two methyl groups are oriented *anti* to each other.

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Comment

The title compound, (I), is the fifth in a series of structurally related compounds, introduced in our earlier report (Coumbarides *et al.*, 2006). With $R^1 = \text{C}_6\text{H}_5\text{O}$, the reaction shown in that report yielded the *anti-syn* and *syn-syn* diastereomers in 44 and 45% yields, respectively. The title compound, (I), is the *syn-syn* diastereomer.



The conformation of (I) (Fig. 1) is closely comparable with that of the phenylpropionyl derivative (Coumbarides *et al.*, 2006). The five-membered ring displays a twist conformation in which atoms C1 and C2 lie, respectively, 0.286 (5) Å above and 0.291 (5) Å below the plane defined by atoms O1, O2, N1 and C3. The two methyl groups (C4 and C19) lie *anti* to each other, on either side of the five-membered ring. The carbonyl groups (C3=O2 and C11=O3) are also oriented *anti* to each other [torsion angle O3–C11–N1–C3 = $-171.2(3)^\circ$], avoiding electrostatic repulsion between the two O atoms. The shortest intermolecular contacts (Fig. 2) are C–H...O interactions [$\text{H16}\cdots\text{O2}^i = 2.66\text{ \AA}$; symmetry code: (i) $\frac{1}{2} - x, 2 - y, \frac{1}{2} + z$] and edge-to-face C–H... π interactions [$\text{H9}\cdots\text{centroid}(\text{C13}-\text{C18}) = 2.92\text{ \AA}$; symmetry code: (ii) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$].

Experimental

The experimental procedure is comparable with that reported previously (Coumbarides *et al.*, 2006). The actual quantities used for preparation of (I) were: *n*-butyllithium (12.42 ml, 2.5 M in hexanes, 31.0 mmol) and (*R,S*)-oxazolidinone (5.00 g, 28.2 mmol) in 60 ml tetrahydrofuran (THF), combined with a solution of (*rac*)-2-phenoxypropanoyl chloride (5.71 g, 31.0 mmol) in 10 ml THF. The crude residue was purified by flash column chromatography on silica gel, eluting with light petroleum (b.p. 313–333 K)/diethyl ether (1:1)

to give a separable diastereoisomeric mixture in the approximate ratio *anti*-*syn*:*syn*-*syn* 50:50. The *syn*-*syn* diastereomer was isolated as colourless crystals [4.13 g, 45% yield, m.p. 403–404 K, R_F 0.42 [light petroleum b.p 313–333 K/diethyl ether, 1:1]]. Spectroscopic analysis: $[\alpha]_D^{25} = +69.8$ (CHCl₃, 295 K, concentration 1.9 g per 100 ml); IR (CHCl₃, ν_{\max} , cm⁻¹): 1770 (C=O), 1712 (C=O); ¹H NMR (250 MHz; CDCl₃): δ 7.44–7.21 (7H, *m*, 7 × CH; Ph_a and Ph_b), 6.99–6.84 (3H, *t*, $J = 7.3$ Hz, 3 × CH; Ph_a and/or Ph_b), 6.02 (1H, *q*, $J = 6.6$ Hz, PhCH), 5.75 (1H, *d*, $J = 7.2$ Hz, PhCHO), 5.69 (1H, *m*, CHN), 1.68 (3H, *d*, $J = 6.6$ Hz, CH₃CHCO), 0.90 (3H, *d*, $J = 6.8$ Hz, CH₃CHN); ¹³C NMR (67.9 MHz; CDCl₃): δ 172.2 (NC=O), 157.3 (*i*-CO; Ph), 152.9 (OC=O), 132.9 (*i*-C; Ph), 129.7, 129.1, 128.9, 125.7, 121.6, 115.0 (6 × CH; Ph_a and Ph_b), 80.8 (PhCHO), 71.7 (PhCH), 55.2 (CHN), 18.6 (CH₃), 14.5 (CH₃); found: MH⁺ 326.1393; C₁₉H₂₀NO₄ requires 326.1392.

Crystal data

C ₁₉ H ₁₉ NO ₄	Z = 4
$M_r = 325.35$	$D_x = 1.302$ Mg m ⁻³
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 16.915$ (12) Å	$\mu = 0.09$ mm ⁻¹
$b = 10.634$ (5) Å	$T = 293$ (2) K
$c = 9.226$ (6) Å	Prism, colourless
$V = 1659.5$ (18) Å ³	0.40 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	1115 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{\text{int}} = 0.032$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
3072 measured reflections	2 standard reflections
1681 independent reflections	every 100 reflections
	intensity decay: 3%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.089$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.14$ e Å ⁻³
1681 reflections	$\Delta\rho_{\text{min}} = -0.14$ e Å ⁻³
220 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.017 (2)

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H = 0.93–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$. 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 *et al.*, 2006).

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*

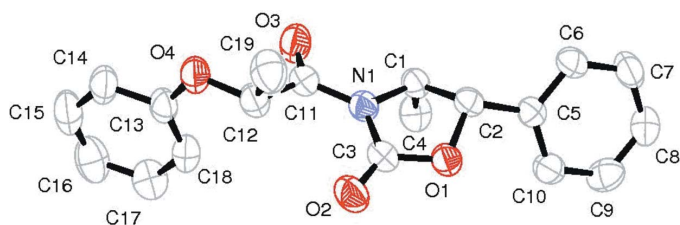


Figure 1 The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted.

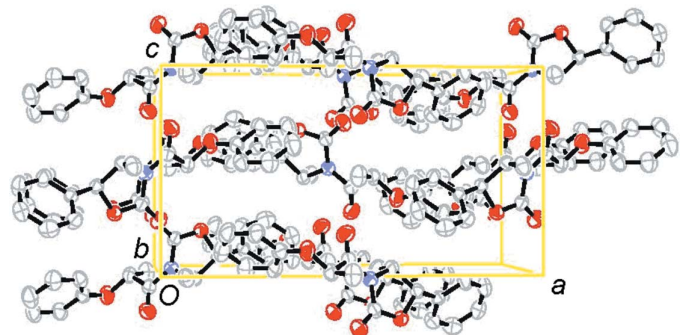


Figure 2 A view of (I) along the *b*-axis direction. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted.

(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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