# (−)-(4R,5S)-4-Methyl-3-[2(R)-(4-methyl-phenyl)propionyl]-5-phenyloxazolidin-2-one

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In the title compound, $C_{20}H_{21}NO_3$, formed from enantiomerically pure (+)-(4R,5S)-4-methyl-5-phenyl-2-oxazolidin-one and racemic 2-(4-methylphenyl)propanoyl chloride, the two carbonyl groups are oriented *anti* to each other, and the methyl group of the (4-methylphenyl)propionyl substituent lies close to the mean plane of the five-membered ring.

## Comment

The title compound is the third in a series of structurally related compounds, introduced in our earlier report (Coumbarides, Eames *et al.*, 2006). With $R^1$ = 4-(CH$_3$)C$_6$H$_4$, the reaction shown in that report yielded the *anti–syn* and *syn–syn* diastereomers in 38 and 38% yields, respectively. The title compound, (I), is the *anti–syn* diastereomer (Fig. 1). In the crystal structure, the conformation of the central portion of the molecule is closely comparable with that in the previously reported derivatives (Coumbarides, Eames *et al.*, 2006; Coumbarides, Dingjan *et al.*, 2006); in the twisted five-membered ring, atoms C1 and C2 lie, respectively, 0.245 (4) Å above and −0.202 (4) Å below the plane defined by atoms O1, O2, N1 and C3. The carbonyl groups (C3=O2 and C11=O3) are oriented *anti* to each other, with the torsion angle O3—C11—N1—C3 = −169.3 (2)°. The orientation of the 4-(CH$_3$)C$_6$H$_4$ substituent resembles most closely that in the 4-($^i$Bu)C$_6$H$_4$ derivative (Coumbarides, Dingjan *et al.*, 2006), with the C19 methyl group lying close to the mean plane of the five-membered ring [deviating by 0.138 (8) Å from it] and the torsion angle N1—C11—C12—C13 = 78.7 (3)°.



(I)
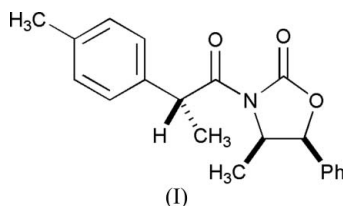
## Experimental

The experimental procedure is comparable with that reported previously (Coumbarides, Eames *et al.*, 2006). The actual quantities used for the preparation of (I) were: *n*-butyllithium (15.45 ml, 2.5 *M* in hexanes, 38.6 mmol) and (*R*,*S*)-oxazolidinone (4.89 g, 27.6 mmol) in 60 ml tetrahydrofuran (THF), combined with a solution of (*rac*)-2-(4-methylphenyl)propanoyl chloride (5.02 g, 27.6 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 ratio *anti–syn*:*syn*,*syn* 50:50. The *anti–syn* diastereomer was obtained as

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colourless crystals (3.39 g, 38% yield, m.p. 341–343 K, $R_F$ 0.71 [light petroleum (b.p 313–333 K)/diethyl ether, 7:3]. Spectroscopic analysis: $[\alpha]_D^{20} = -164.5$ (CHCl$_3$, 293 K, concentration 0.18 g per 100 ml); IR (CHCl$_3$, $\nu_{max}$/cm$^{-1}$): 1779 (C=O), 1710 (C=O); $^1$H NMR (270 MHz; CDCl$_3$): $\delta$ 7.36–7.24 (9H, $m$, 9 × CH; Ar and Ph), 5.46 (1H, $d$, $J$ = 6.9 Hz, CHO), 5.07 (1H, $q$, $J$ = 7.1 Hz, ArCH), 4.65 (1H, $m$, NCHCH$_3$), 2.31 (3H, $s$, CH$_3$; Ar), 1.46 (3H, $d$, $J$ = 7.1 Hz, CH$_3$CH), 0.91 (3H, $d$, $J$ = 6.9 Hz, CH$_3$CHN); $^{13}$C NMR (67.5 MHz; CDCl$_3$): $\delta$ 174.8 (NC=O), 152.9 (OC=O), 137.7, 137.1, 133.9 (3 × $i$-C; Ar and Ph), 129.7, 129.1, 128.3, 128.3, 126.1 (5 × CH; Ar and Ph), 79.1 (PhCHO), 55.0 (CHN), 43.6 (ArCH), 21.4 (CH$_3$; Ar), 19.8 (CH$_3$CH), 14.5 (CH$_3$CHN); found: MH$^+$ 324.1187; C$_{20}$H$_{22}$NO$_3$ requires 324.1194.

## Crystal data

C$_{20}$H$_{21}$NO$_3$
$M_r$ = 323.38
Monoclinic, $P2_1$
$a$ = 13.066 (8) Å
$b$ = 9.509 (8) Å
$c$ = 7.201 (4) Å
$\beta$ = 102.95 (4)°
$V$ = 871.9 (10) Å$^3$

$Z$ = 2
$D_x$ = 1.232 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 160 (2) K
Prism, colourless
0.40 × 0.30 × 0.30 mm

## Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1743 measured reflections
1630 independent reflections

1417 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.010
$\theta_{max}$ = 25.0°
2 standard reflections
 every 100 reflections
 intensity decay: 1%

## Refinement

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.078
$S$ = 1.06
1630 reflections
220 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.0761P]$
 where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$

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) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(methyl C). The methyl groups were allowed to rotate about their local threefold axes. In the absence
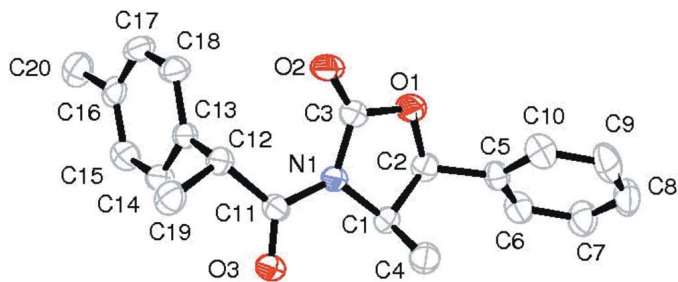


**Figure 1**
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted.

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).

Data collection: *CAD-4-PC* (Enraf–Nonius, 1994); cell refinement: *CAD-4-PC*; 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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