(−)-(4*R*,5*S*)-3-[2(*R*)-(4-Chlorophenyl)propionyl]-4-methyl-5-phenyloxazolidin-2-one

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Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.007 Å
R factor = 0.047
wR factor = 0.105
Data-to-parameter ratio = 8.2

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

The title compound, $C_{19}H_{18}ClNO_3$, is formed from enantiomerically pure (+)-(4R,5S)-4-methyl-5-phenyl-2-oxazolidinone and racemic 2-(4-chlorophenyl)propanoyl chloride. The crystal structure resembles closely that of the comparable (4-methylphenyl)propionyl derivative, although the two structures differ in the nature of the intermolecular contacts to the Cl atom and methyl group.

Comment

The title compound, (I), is the fourth in a series of structurally related compounds, introduced in our earlier report (Coumbarides *et al.*, 2006). With R^1 = 4-(Cl)C_6H_4, the reaction shown in that report yielded the *anti–syn* and *syn–syn* diastereomers in 38 and 39% yields, respectively. The title compound, (I), is the *anti–syn* diastereomer (Fig. 1). In the crystal structure, the conformation of the molecule is essentially indistinguishable from that of the (4-methylphenyl)propionyl derivative (Chavda *et al.*, 2006).

(I)
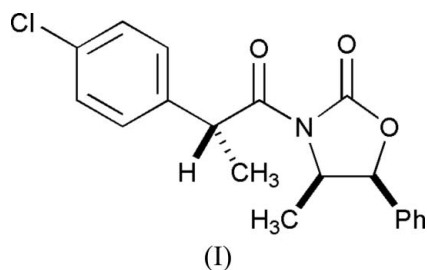
The crystal structure of (I) is closely related to that of the (4-methylphenyl)propionyl derivative. The two structures contain essentially identical two-dimensional layers, lying in the (010) planes for (I) and in the (100) planes for the methyl derivative (Fig. 2). In the methyl derivative, adjacent layers are related by translation along *a*, bringing the methyl groups of the 4-(CH_3)C_6H_4 substituent into the vicinity of O2 [H20B···O2 = 2.71 Å]. In (I), adjacent layers are related by 2_1 screw axes, and Cl1 forms its shortest intermolecular contacts between layers to the methyl group C19 [H19B···Cl1[i] = 3.35 Å; symmetry code: (i) $-\frac{1}{2} + x, \frac{3}{2} + y, \frac{3}{2} - z$]. Thus, chloro/methyl interchange (Edwards *et al.*, 2006) does not lead to isostructurality in this instance, and this can be attributed to the influence of the different charge distributions of the Cl atom and CH_3 group.

Experimental

The experimental procedure is comparable with that reported previously (Coumbarides *et al.*, 2006). The actual quantities used for

the preparation of (I) were: *n*-butyllithium (15.81 ml, 2.5 *M* in hexanes, 39.5 mmol) and (*R,S*)-oxazolidinone (5.00 g, 28.2 mmol) in 60 ml tetrahydrofuran (THF), combined with a solution of (*rac*)-2-(4-chlorophenyl)propanoyl chloride (5.73 g, 28.2 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 (7:3), to give a separable diastereoisomeric mixture in the approximate ratio *anti–syn:syn–syn* 50:50. The *anti–syn* diastereomer was obtained as colourless crystals {3.68 g, 38% yield, m.p. 362–364 K, R_F 0.58 [light petroleum (b.p 313–333 K)/diethyl ether, 7:3]}. Spectroscopic analysis: $[\alpha]_D^{22} = -60.0$ (CHCl₃, 293 K, concentration 0.60 g per 100 ml); IR (CHCl₃, ν_{max}/cm⁻¹): 1779 (C=O), 1713 (C=O); ¹H NMR (270 MHz; CDCl₃): 7.36–7.20 (9H, *m*, 9 × CH; Ar and Ph), 5.62 (1H, *d*, *J* = 6.4 Hz, CHPh), 5.01 (1H, *q*, *J* = 6.9 Hz, ArCH), 4.79 (1H, *m*, CHN), 1.48 (3H, *d*, *J* = 6.9 Hz, CH₃CH), 0.86 (3H, *d*, *J* = 6.4 Hz, CH₃CHN); ¹³C NMR (100 MHz; CDCl₃): δ 173.9 (NC=O), 152.5 (OC=O), 138.9 (*i*-CCl; Ar), 133.2, 133.1 (2 × *i*-C; Ar and Ph), 129.5, 128.9, 128.8, 128.7, 125.6 (5 × CH; Ar and Ph), 78.7 (PhCHO), 55.4 (CHN), 42.7 (ArCH), 19.2 (CH₃CH), 14.4 (CH₃CHN); found: MNH₄⁺ 361.1307; C₁₉H₂₂ClN₂O₃ requires 361.1313.

Crystal data

C₁₉H₁₈ClNO₃	$Z = 4$
$M_r = 343.79$	$D_x = 1.307$ Mg m⁻³
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 7.105\ (3)$ Å	$\mu = 0.24$ mm⁻¹
$b = 25.662\ (12)$ Å	$T = 160\ (2)$ K
$c = 9.580\ (8)$ Å	Prism, colourless
$V = 1746.7\ (18)$ Å³	$0.20 \times 0.10 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	1083 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{int} = 0.044$
Absorption correction: none	$\theta_{max} = 25.0°$
2316 measured reflections	2 standard reflections
1794 independent reflections	frequency: 60 min
	intensity decay: 2%

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2]$
$wR(F^2) = 0.105$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
1794 reflections	$\Delta\rho_{max} = 0.21$ e Å⁻³
219 parameters	$\Delta\rho_{min} = -0.22$ 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) = $1.2U_{eq}$(C) or $1.5U_{eq}$(methyl C). The methyl groups were allowed to rotate about their local threefold axes. The absolute configuration could not be established and is assigned on the basis of the known configuration of the starting material (Coumbarides *et al.*, 2006).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97*
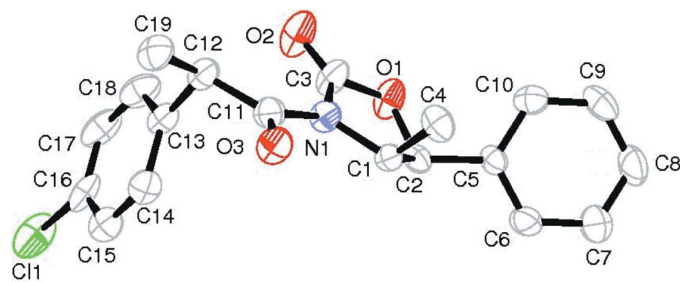

Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted.
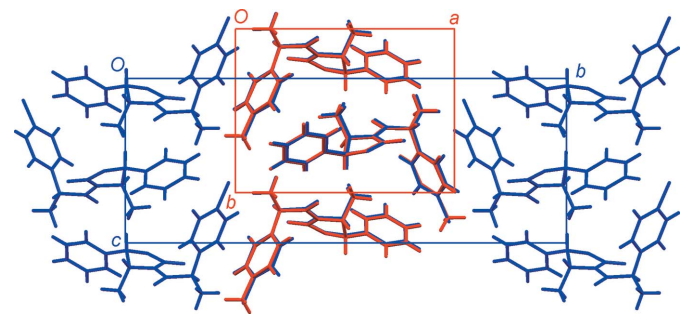

Figure 2
Overlay of the unit-cell contents of (I) (blue) and the (4-methylphenyl)propionyl derivative (red) (Chavda *et al.*, 2006), showing essentially identical layers of molecules in the (010) planes of (I) and (100) planes of the methyl derivative.

(Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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