

**(+)-(4*R*,5*S*)-3-[2(*S*)-(4-Isobutylphenyl)propionyl]-  
4-methyl-5-phenyloxazolidin-2-one**Gregory S. Coumbarides,<sup>a</sup> Marco Dingjan,<sup>a</sup> Jason Eames,<sup>b\*</sup> Majid Motevalli<sup>a</sup> and Nela Malatesti<sup>c</sup><sup>a</sup>Department of Chemistry, Queen Mary, University of London, Mile End Road, London E1 4NS, England, <sup>b</sup>Department of Chemistry, University of Hull, Cottingham Road, Kingston-upon-Hull HU6 7RX, England, and <sup>c</sup>Department of Chemistry, J. J. Strossmayer University of Osijek, Trg Sv. Trojstva 3, Osijek 31000, Croatia

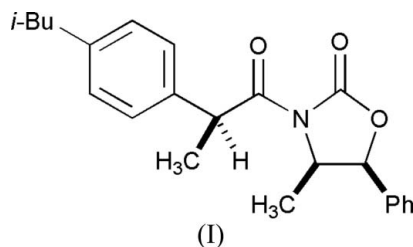
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**Key indicators**Single-crystal X-ray study  
*T* = 160 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$   
*R* factor = 0.042  
*wR* factor = 0.101  
Data-to-parameter ratio = 8.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

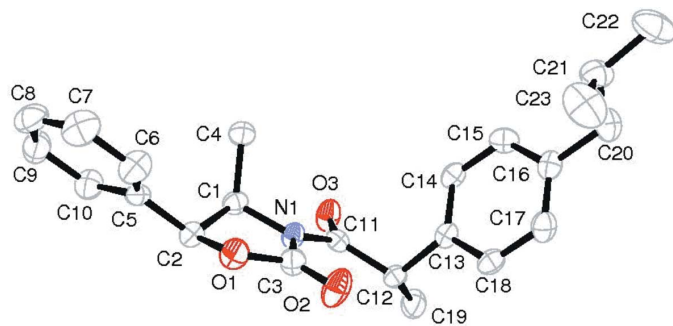
In the title compound,  $\text{C}_{23}\text{H}_{27}\text{NO}_3$ , formed from enantiomerically pure (+)-(4*R*,5*S*)-4-methyl-5-phenyl-2-oxazolidinone and racemic 2-(4-isobutylphenyl)propanoyl chloride, the two carbonyl groups are oriented *anti* to each other, and the methyl group of the (4-isobutylphenyl)propionyl substituent lies close to the mean plane of the five-membered ring.

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The title compound, (I), is the second in a series of structurally related compounds, introduced in our previous report (Coumbarides *et al.*, 2006). With  $R^1 = 4\text{-}(i\text{-Bu})\text{C}_6\text{H}_4$ , the reaction shown in that report yielded the *anti-syn* and *syn-syn* diastereomers in 34 and 32% yields, respectively. The title compound, (I), is the *syn-syn* diastereomer (Fig. 1). In the crystal structure, the conformation of the central portion of the molecule is closely comparable with that in the phenyl derivative (Coumbarides *et al.*, 2006). The conformation of the five-membered ring is similar, with atoms C1 and C2 lying respectively 0.170 (6) Å above and 0.298 (6) 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 =  $-179.7 (4)^\circ$ . The principal difference between the conformations of (I) and the phenyl derivative lies in the orientation of the (4-isobutylphenyl)propionyl substituent with respect to the remainder of the molecule: in (I), the torsion angle N1–C11–C12–C13 =  $-90.2 (4)^\circ$  compared with  $-166.97 (16)^\circ$  for the comparable measurement in the phenyl derivative. Thus, the C19 methyl group lies closer to the plane of the five-membered ring in (I), in contrast with the *anti* arrangement observed for the C4 and C19 methyl groups in the phenyl derivative.

**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 (0.22 ml, 2.5 M in hexanes, 0.56 mmol) and (*R,S*)-oxazolidinone (0.1 g, 0.56 mmol) in 10 ml tetrahydrofuran (THF), combined with a solution of (*rac*)-2-(4-



**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.

isobutylphenyl)propanoyl chloride (0.125 g, 0.56 mmol) in 1 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* 54:46). The *syn*–*syn* diastereomer was obtained as colourless crystals [67 mg, 32% yield, m.p. 394–396 K,  $R_F$  0.55 [light petroleum (b.p. 313–333 K)/diethyl ether, 1:1]]. Spectroscopic analysis:  $[\alpha]_D^{25} = +98.1$  (CHCl<sub>3</sub>, 293 K, concentration 1.3 g per 100 ml); IR (CHCl<sub>3</sub>,  $\nu_{\max}$ , cm<sup>-1</sup>): 1770 (C=O), 1699 (C=O); <sup>1</sup>H NMR (250 MHz; CDCl<sub>3</sub>):  $\delta$  7.38–7.16 (7H, *m*, 7 × CH; Ar and Ph), 7.08 (2H, *d*,  $J = 8.2$  Hz, 2 × CH; Ar), 5.63 (1H, *d*,  $J = 7.4$  Hz, CHO), 5.05 (1H, *q*,  $J = 7.1$  Hz, ArCH), 4.81 (1H, *m*, CHN), 2.43 (2H, *d*,  $J = 7.2$  Hz, CH<sub>2</sub>), 1.89–1.79 (1H, *m*, CH(CH<sub>3</sub>)<sub>2</sub>), 1.48 (3H, *d*,  $J = 7.1$  Hz, CH<sub>3</sub>CHAr), 0.88 (3H, *d*,  $J = 6.7$  Hz, CH<sub>3</sub><sup>a</sup>CHCH<sub>3</sub><sup>b</sup>), 0.87 (3H, *d*,  $J = 6.7$  Hz, CH<sub>3</sub><sup>a</sup>CHCH<sub>3</sub><sup>b</sup>), 0.72 (3H, *d*,  $J = 6.7$  Hz, CH<sub>3</sub>CHN); <sup>13</sup>C NMR (100.6 MHz; CDCl<sub>3</sub>):  $\delta$  174.6 (NC=O), 152.6 (OC=O), 140.5 (*i*-C; Ar), 137.5 (*i*-C; Ar), 133.6 (*i*-C; Ph), 129.3, 127.7 (2 × CH; Ar), 129.1, 128.7 and 125.8 (3 × CH; Ph), 78.8 (OCHPh), 54.7 (CHN), 45.1 [CH(CH<sub>3</sub>)<sub>2</sub>], 42.2 (ArCH), 30.1 (CH<sub>2</sub>), 22.5 (CH<sub>3</sub><sup>a</sup>CHCH<sub>3</sub><sup>b</sup>; isobutylphenyl), 19.4 (CH<sub>3</sub>CH), 14.2 (CH<sub>3</sub>CHN); found:  $M$  365.1986; C<sub>23</sub>H<sub>27</sub>NO<sub>3</sub> requires 365.1985.

#### Crystal data

C<sub>23</sub>H<sub>27</sub>NO<sub>3</sub>  
 $M_r = 365.46$   
 Orthorhombic,  $P2_12_12_1$   
 $a = 7.194$  (4) Å  
 $b = 14.208$  (8) Å  
 $c = 20.049$  (11) Å  
 $V = 2049$  (2) Å<sup>3</sup>

$Z = 4$   
 $D_x = 1.185$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 160$  (2) K  
 Block, colourless  
 $0.63 \times 0.38 \times 0.15$  mm

#### Data collection

Enraf–Nonius CAD-4  
 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 2141 measured reflections  
 2079 independent reflections

1340 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.004$   
 $\theta_{\text{max}} = 25.0^\circ$   
 2 standard reflections  
 frequency: 60 min  
 intensity decay: 1%

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.101$   
 $S = 1.00$   
 2079 reflections  
 248 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0466P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.18$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.20$  e Å<sup>-3</sup>

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  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  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, 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 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* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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