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**(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl  
(2*S*,3*S*)-3-phenyloxirane-2-carboxylate**

The title compound, C<sub>19</sub>H<sub>26</sub>O<sub>3</sub>, was synthesized from (1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl chloroacetate and benzaldehyde *via* the well known Darzen reaction. The absolute configuration was determined from the synthetic precursor. Non-classical C—H···O hydrogen bonds link the molecules in the crystal structure into a sheet parallel to (001).

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**Key indicators**

Single-crystal X-ray study

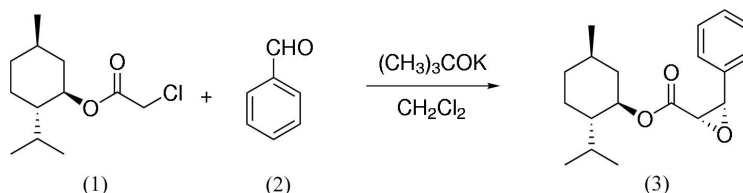
*T* = 293 KMean  $\sigma$ (C—C) = 0.004 Å*R* factor = 0.047*wR* factor = 0.123

Data-to-parameter ratio = 11.4

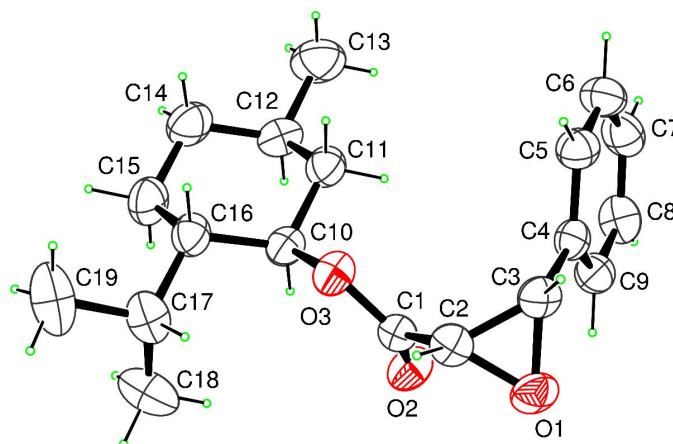
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

**Comment**

Fig. 1 shows the structure of the title compound, (3). Selected molecular parameters and hydrogen-bonding geometries are listed in Tables 1 and 2, respectively. The absolute configuration was chosen according to the known configuration of the starting material, (1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexanol [also known as (–)-*L*-menthol]. This was not unexpected, as the chiral centres were not affected by the reaction.



The compound crystallizes in the orthorhombic space group *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub> with four symmetry-equivalent molecules per unit cell. In the crystal structure, non-classical C—H···O hydrogen bonds play an important role, resulting in the formation of a polymeric sheet parallel to (001).

**Figure 1**

The molecule of compound (3) in the crystal structure. Displacement ellipsoids are drawn at the 30% probability level.

Experimental

To a mixture of benzaldehyde (2) (6.4 g, 60 mmol) and (–)-menthyl chloroacetate (1) (11.6 g, 50 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10 ml) was added dropwise a solution of potassium *tert*-butoxide (5.6 g, 50 mmol) in anhydrous *tert*-butyl alcohol (45 ml) with vigorous stirring over a period of 1 h, the temperature being kept below 283 K. The mixture was stirred for another 4 h, then the reaction mixture was extracted with diethyl ether (3 × 20 ml). The combined ethereal solutions were washed with saturated NaHSO<sub>3</sub> (3 × 30 ml), saturated sodium bicarbonate solution, and brine (3 × 30 ml), and dried over anhydrous sodium sulfate. An oily residue was obtained after distillation of the solvent under reduced pressure. Aqueous ethanol (90%, 5 ml) was added to the oily residue, and a white solid was obtained (yield 9.2 g, 61%) after being kept at 269 K for 2 h. Colourless crystals were obtained from a saturated 90% aqueous ethanol solution after allowing it to stand for 4 d. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.40 (*dd*, 2H, *J* = 7.8 and 1.7 Hz), 7.32 (3H, *m*), 4.58 (*dt*, 1H, *J* = 10.9 and 4.2 Hz), 4.26 (*d*, 1H, *J* = 4.6 Hz), 3.83 (*d*, 1H, *J* = 4.8 Hz), 1.6–0.85 (9H, *m*), 0.78 (*d*, 3H, *J* = 7 Hz), 0.75 (*d*, 3H, *J* = 6.4 Hz), 0.63 (*d*, 3H, *J* = 6.9 Hz).

Crystal data

C<sub>19</sub>H<sub>26</sub>O<sub>3</sub>  
*M<sub>r</sub>* = 302.4  
 Orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 5.6507 (6) Å  
*b* = 12.8745 (17) Å  
*c* = 24.386 (3) Å  
*V* = 1774.1 (4) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.132 Mg m<sup>-3</sup>

Mo Kα radiation  
 Cell parameters from 14 189 reflections  
 θ = 1.8–27.3°  
 μ = 0.08 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Prism, colorless  
 0.70 × 0.38 × 0.23 mm

Data collection

Rigaku R-Axis RAPID diffractometer  
 ω scans  
 14 189 measured reflections  
 2312 independent reflections  
 1675 reflections with *I* > 2σ(*I*)

*R*<sub>int</sub> = 0.054  
 θ<sub>max</sub> = 27.2°  
*h* = -7 → 7  
*k* = -16 → 16  
*l* = -31 → 30

Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.047  
*wR*(*F*<sup>2</sup>) = 0.124  
*S* = 1.02  
 2312 reflections  
 203 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.1274P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 (Δ/σ)<sub>max</sub> < 0.001  
 Δρ<sub>max</sub> = 0.13 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.12 e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.023 (3)

Table 1

Hydrogen-bonding geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
C2—H2...O2 <sup>ii</sup>	0.98	2.602	3.290 (4)	127
C3—H3...O2 <sup>ii</sup>	0.98	2.693	3.350 (4)	125
C6—H6...O2 <sup>ii</sup>	0.93	2.698	3.486 (4)	143
C7—H7...O1 <sup>ii</sup>	0.93	2.692	3.342 (5)	128

Symmetry codes: (i) *x* - 1, *y*, *z*; (ii) 1 - *x*, ½ + *y*, ½ - *z*.

The absolute stereochemistry could not be established from the diffraction experiment because of the lack of significant anomalous dispersion effects; Friedel pairs in the data set were merged. The

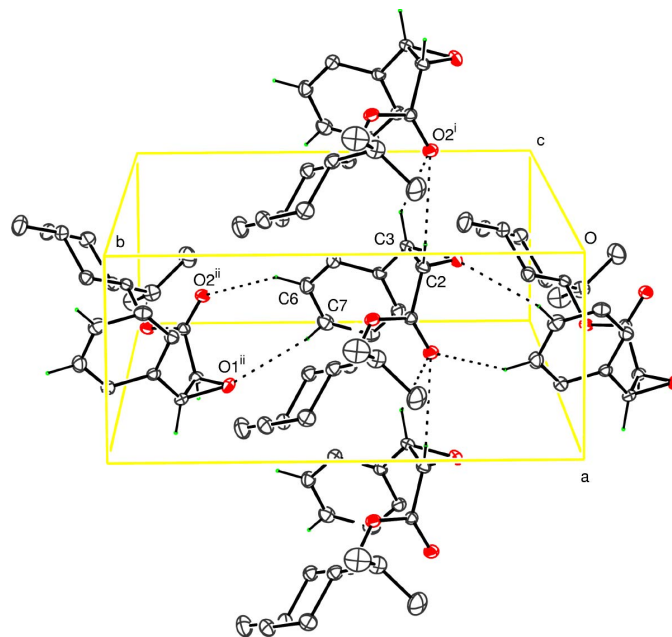


Figure 2

The molecular packing of (3). Dashed lines indicate the hydrogen-bonding interactions. H atoms not involved in hydrogen bonding have been omitted (see Table 1 for symmetry codes).

absolute configuration was therefore chosen on the basis of the known configuration of the synthetic precursor. The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with *U*<sub>iso</sub>(H) = 1.5*U*<sub>eq</sub>(C), and were allowed to rotate freely about the C—C bonds. The other H atoms were placed in calculated positions (C—H = 0.93–0.98 Å), with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub> of the carrier atoms, and included in the final cycles of refinement using a riding model.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); 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) and *PLATON* (Spek, 2003).

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