Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 2,5-Dioxopyrrolidin-1-yl (1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl carbonate

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Received 14 November 2006
Accepted 20 November 2006

The title compound, $C_{15}H_{23}NO_5$, was obtained as colourless crystals by the reaction of *N*,*N*′-disuccinimidyl carbonate (DSC) and L-(−)-menthol. The molecular packing is stabilized by weak C—H···O hydrogen-bonding interactions.

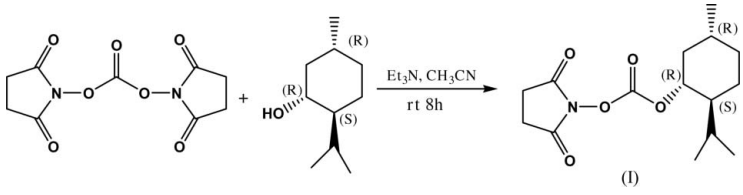
**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.006 Å
*R* factor = 0.062
*wR* factor = 0.126
Data-to-parameter ratio = 8.6

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

## Comment

The title compound, (I), is a key intermediate used in the synthesis of a series of biologically active polyfunctional molecules for enzyme active sites (Ghosh *et al.*, 1992). It can react with various chiral alcohols and amines to yield active esters of interest in medicinal chemistry. The absolute configuration of the three stereocentres remains unchanged during the synthetic procedure. The molecular packing is stabilized by weak C—H···O hydrogen-bonding interactions (Table 1).




## Experimental

*N*,*N*′-Disuccinimidyl carbonate (DSC), was prepared according to the procedure of Pereira *et al.* (1998). To a solution of DSC (1.5 mmol, 0.384 g) and L-(−)-menthol (1.0 mmol, 0.156 g) in dried acetonitrile (10 ml) at room temperature was added triethylamine (3 mmol) dropwise. The resulting mixture was stirred at room temperature

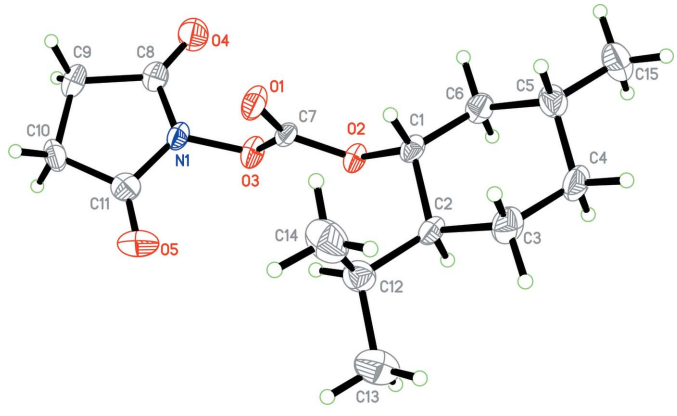



**Figure 1**
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by spheres of arbitrary radii.

until no starting L-(−)-menthol remained, as monitored by thin-layer chromatography (8 h). The mixture was concentrated under reduced pressure and the residue was diluted with a saturated aqueous $NaHCO_3$ solution (10 ml) and extracted thoroughly with ethyl acetate (3 × 20 ml). The combined extracts were washed with a saturated aqueous NaCl solution (8 ml) and dried over $Na_2SO_4$. Evaporation of the solvent provided the mixed carbonate (I) which was purified by column chromatography over silica gel (petroleum ether/ethyl acetate 5:1 *v*/*v*) to obtain a white solid. Single crystals were obtained by recrystallization from a mixture of petroleum ether/ethyl acetate (5:1 *v*/*v*).

*Crystal data*

$C_{15}H_{23}NO_5$
$M_r$ = 297.34
Orthorhombic, $P2_12_12_1$
$a$ = 6.5911 (14) Å
$b$ = 7.7815 (17) Å
$c$ = 31.125 (7) Å
$V$ = 1596.4 (6) Å$^3$
$Z$ = 4
$D_x$ = 1.237 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 (2) K
Block, colourless
0.40 × 0.27 × 0.20 mm

*Data collection*

Bruker APEX area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.964, $T_{max}$ = 0.982
7845 measured reflections
1652 independent reflections
1621 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028
$\theta_{max}$ = 25.0°

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.127
$S$ = 1.39
1652 reflections
193 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0276P)^2 + 0.8697P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C1—H1···O5$^{i}$ | 0.98 | 2.51 | 3.460 (5) | 164 |
| C10—H10$A$···O1$^{ii}$ | 0.97 | 2.41 | 3.255 (5) | 145 |

Symmetry codes: (i) $x-1, y, z$; (ii) $x+\frac{1}{2}, -y+\frac{5}{2}, -z+2$.

All H atoms were placed in geometrically idealized positions and treated as riding on their parent atoms, with C—H = 0.98 (CH), 0.97 ($CH_2$) or 0.96 Å ($CH_3$), and $U_{iso}$(H) = 1.2 $U_{eq}$(CH and $CH_2$) or 1.5$U_{eq}$(methyl C). In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration was assumed from the synthesis.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The project was supported by the National Natural Science Foundation of China (No. 20572061) and we also thank the Key Laboratory for the Physical Chemistry of the Solid Surface for providing the X-ray diffraction facilities.

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