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Cyclodec-2′-ynyl (1*S*,1′*R*)-(−)-4,7,7-trimethyl-3-oxo-2-oxabicyclo[2.2.1]heptane-1-carboxylate

Claudia Zelder, Markus Schürmann, Hans Preut* and Norbert Krause

Fachbereich Chemie, Universität Dortmund, Otto-Hahn-Straße 6, 44221 Dortmund, Germany

Correspondence e-mail: uch002@uxp1.hrz.uni-dortmund.de

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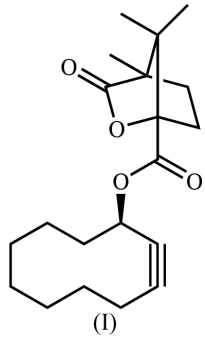
Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.005 Å
R factor = 0.040
wR factor = 0.085
Data-to-parameter ratio = 10.9

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

The synthesis and the crystal structure analysis *via* X-ray diffraction of the title compound, $C_{20}H_{28}O_4$, (I), are described. The title compound is a camphanic acid cyclodecynyl ester. This compound contains a ten-membered ring including a triple bond and is prepared by condensation of (1*S*)-(−)-camphanic acid chloride with (*R*)-(+)-cyclodec-2-yn-1-ol [(II), 90% ee]. This alcohol [Hanack & Wächter (1987). *Chem. Ber.* **120**, 727–734] has been isolated by kinetic deracemization of the corresponding acetate [Zelder (2001). PhD Thesis. In preparation] with the lipase from *mucor miehei*. With this structure analysis, the configuration of (II) was determined.



(I)

Experimental

(*R*)-(+)-Cyclodec-2-yn-1-ol was dissolved in pure pyridine. (1*S*)-(−)-Camphanic acid chloride was added in one portion and the mixture was stirred for 1 h at 313 K. After this process, the title compound was obtained in 85% yield. A small amount was dissolved in 1 ml of cyclohexane/diethyl ether (1:1) and crystals were obtained by vapor diffusion at room temperature.

Crystal data

$C_{20}H_{28}O_4$
M_r = 332.42
Orthorhombic, $P2_12_12_1$
a = 6.5640 (3) Å
b = 9.7316 (5) Å
c = 28.4686 (14) Å
V = 1818.52 (15) Å^3
Z = 4
D_x = 1.214 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 11414 reflections
θ = 3.0–27.5°
μ = 0.08 mm^{-1}
T = 291 (1) K
Plate, colourless
0.50 × 0.50 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
109 frames *via* ω-rotation (Δω = 1°) with two sets at different κ-angles and two times 20 s per frame
2407 measured reflections
2407 independent reflections
1036 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 27.5°
h = −8 → 8
k = −12 → 12
l = −36 → 36

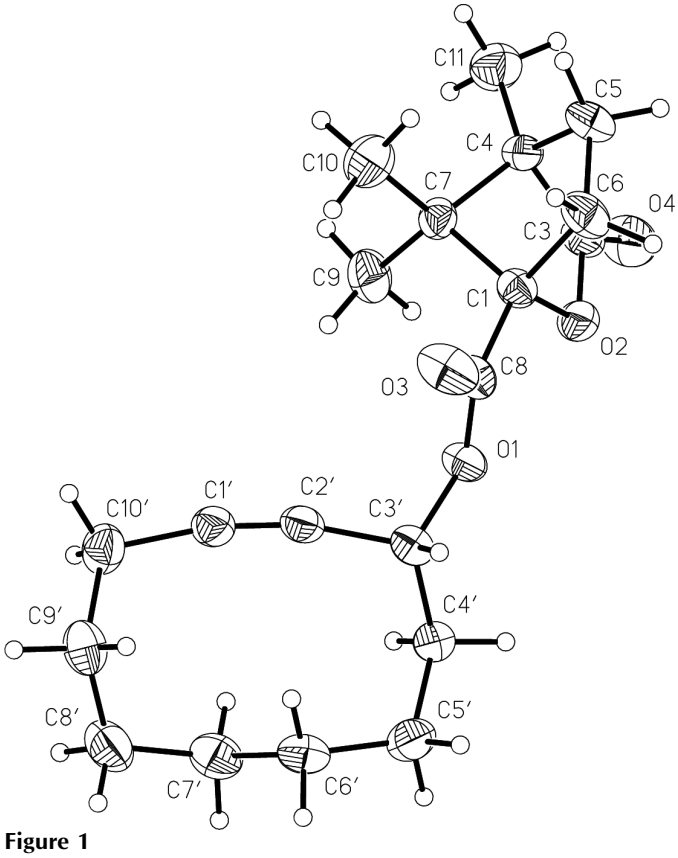


Figure 1
The structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0250P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.040$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.085$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
$S = 0.96$	$\Delta\rho_{min} = -0.13$ e Å^{-3}
2407 reflections	Extinction correction: *SHELXL*97
221 parameters	Extinction coefficient: 0.018 (2)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1′—C2′	1.167 (4)	C2′—C3′	1.467 (4)
C1′—C10′	1.471 (4)		
C2′—C1′—C10′	169.4 (3)	C1′—C2′—C3′	167.7 (3)
C10′—C1′—C2′—C3′	21 (3)	C5′—C6′—C7′—C8′	−166.7 (3)
C1′—C2′—C3′—C4′	27.9 (17)	C6′—C7′—C8′—C9′	61.1 (4)
C2′—C3′—C4′—C5′	−76.5 (3)	C7′—C8′—C9′—C10′	54.7 (4)
C3′—C4′—C5′—C6′	54.5 (4)	C2′—C1′—C10′—C9′	16 (2)
C4′—C5′—C6′—C7′	61.8 (4)	C8′—C9′—C10′—C1′	−74.8 (4)

H atoms were placed in calculated positions with U_{iso} constrained to be $1.5U_{eq}$ of the carrier atom for the methyl groups and with U_{iso} constrained to be $1.2U_{eq}$ of the carrier atom for the remaining positions. The absolute structure could not be determined reliably and the Friedel reflections were merged before final refinement.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PARST*95 (Nardelli, 1995).

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