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(–)-(4*S*)-1-Chloro-3-(2,2-dimethyl-1,3-dioxolan-4-yl)-propan-2-one

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Key indicators

Single-crystal X-ray study
T = 262 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.093
Data-to-parameter ratio = 15.1

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

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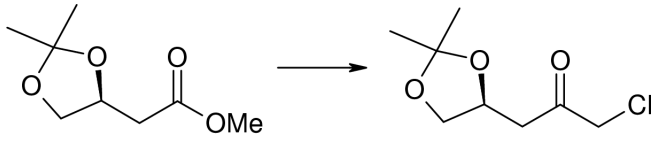
The crystal structure of the title compound, $C_8H_{13}ClO_3$, has been determined at 262 (2) K following *in-situ* growth from the liquid.

Comment

This work forms part of a continuing study devoted to improving the techniques for determining the crystal structures of substances which are liquids at room temperature [see, for example, Davies & Bond (2001)]. The structure of (–)-(4*S*)-1-chloro-3-(2,2-dimethyl-1,3-dioxolan-4-yl)propan-2-one, (2), is reported here.



(1) (2)

Experimental

The sample was obtained from methyl ester (1), prepared using the method of Steel & Thomas (1997). Treatment of (1) with *in-situ* generated chloromethyllithium (Barluenga *et al.*, 1995)) at 195 K in dichloromethane afforded the title compound (2) in 94% yield. The crystal was grown in a 0.3 mm glass capillary tube at 278 K. This temperature is less than 1 K below the observed melting point of the solid in the tube and allowed solid and liquid to exist together in the tube for a period of at least several hours. With the axis of the capillary parallel to the φ axis and horizontal on the instrument (a Nonius KappaCCD diffractometer equipped with an Oxford Cryosystems Cryostream cooler), the crystal was obtained overnight from the very slow movement of the solid/liquid interface (the tube remaining stationary during the period of crystal growth). Attempts to grow a suitable crystal by moving a plug of solid material up and down the tube using a method reported earlier (Davies & Bond, 2001) were unsuccessful, but one of the best of these attempts was used as the starting point for the stationary-tube method which produced the crystal used in the structure determination.

Crystal data

$C_8H_{13}ClO_3$
M_r = 192.63
Orthorhombic, $P2_12_12_1$
a = 5.3067 (2) Å
b = 9.3552 (6) Å
c = 20.0955 (12) Å
V = 997.65 (10) Å^3
Z = 4
D_x = 1.283 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3592 reflections
θ = 1.0–25.0°
μ = 0.35 mm^{-1}
T = 262 (2) K
Cylinder, colourless
0.125 mm (radius)
0.25 mm (length)

Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.090$
Fine-slice ω/φ scans	$\theta_{max} = 25.0°$
5899 measured reflections	$h = -5 \rightarrow 6$
1704 independent reflections	$k = -8 \rightarrow 11$
1385 reflections with $I > 2\sigma(I)$	$l = -23 \rightarrow 22$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.040$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
$wR(F^2) = 0.093$	$\Delta\rho_{min} = -0.18$ e Å^{-3}
$S = 1.20$	Extinction correction: *SHELXL*97
1701 reflections	Extinction coefficient: 0.019 (5)
113 parameters	Absolute structure: Flack (1983)
H-atom parameters constrained	Flack parameter = −0.17 (12)
$w = 1/[\sigma^2(F_o^2) + (0.0247P)^2 + 0.1484P]$ where $P = (F_o^2 + 2F_c^2)/3$	

H atoms were placed geometrically and refined using the usual riding model. The absolute configuration was determined satisfactorily from the X-ray data [Flack (1983) parameter = −0.2 (1), number of Friedel pairs = 649], confirming the known configuration of the starting material.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1993).

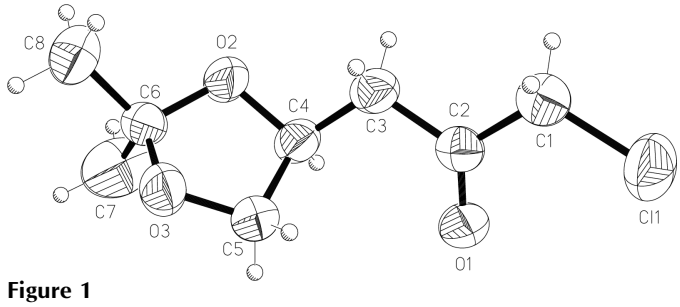


Figure 1
The molecular structure and atom-labelling scheme of (2). Displacement ellipsoids are drawn at the 50% probability level.

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