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Key indicators

Single-crystal X-ray study
T = 190 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.050
wR factor = 0.096
Data-to-parameter ratio = 20.5

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

Isopropyl 2,5-anhydro-3,4-di-O-tert-butyl-diphenylsilyl-L-ribonate

Determination of the crystal structure of the title compound, $\text{C}_{40}\text{H}_{50}\text{O}_5\text{Si}_2$, firmly established its relative configuration and hence that of some related tetrahydrofuran carboxylates. The material crystallizes with $Z' = 2$. Except for the chiral centres, the two independent molecules are related by a pseudo-centre of symmetry.

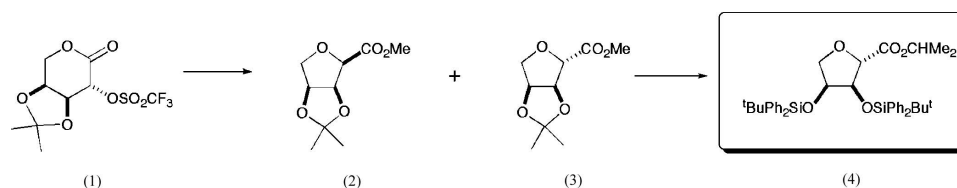
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Comment

The reaction of methanol with lactones containing 2-O-trifluoromethanesulfonates (trifluoromethanesulfonates) in the presence of either acid (Wheatley *et al.*, 1993) or base (Choi *et al.*, 1992) provides a general synthesis of methyl tetrahydrofuran-2-carboxylates. Such materials have been exploited in the preparation of sugar amino acids (SAAs) for use as peptidomimetics (Chakraborty *et al.*, 2004; Grotenberg *et al.*, 2004; Smith *et al.*, 2003). Many THF SAA scaffolds are predisposed to form secondary structures in short oligomers (Claridge *et al.*, 2005; Long *et al.*, 1999, 2002; Hungerford *et al.*, 2000). There are only limited reports of γ -peptides based on cyclic templates (Curran *et al.*, 1996; Crisma *et al.*, 2001). In a programme directed towards the synthesis of γ -THF SAAs, it was found that reaction of the δ -lactone trifluoromethanesulfonate (1) (Stewart *et al.*, 2002) with methanol in the presence of sodium carbonate gave a mixture of the THF carboxylates (2) and (3). In order to ensure the correct assignment of the stereochemistry at C-2 in the epimers, (3) was converted to the crystalline disilyl ether (4), the structure of which is reported in this paper (Fig. 1).



The structure of (4) contains two molecules in the asymmetric unit ($Z' = 2$). Except for the 1,4-anhydroribonate units (which are chiral and therefore cannot be related by an improper operator), the molecules are related by a pseudo-centre of symmetry at $(\frac{1}{2}, \frac{1}{2}, \frac{3}{4})$. The absolute configuration of the material was known unambiguously from the synthesis; the Flack (1983) parameter is in agreement with this assignment.

The structure consists of molecular layers (Fig. 2) lying parallel to the *bc* plane, and characterized by a hydrophilic and a hydrophobic surface. The hydrophobic surface of one layer faces the equivalent surface of the adjacent layer (Fig. 3).

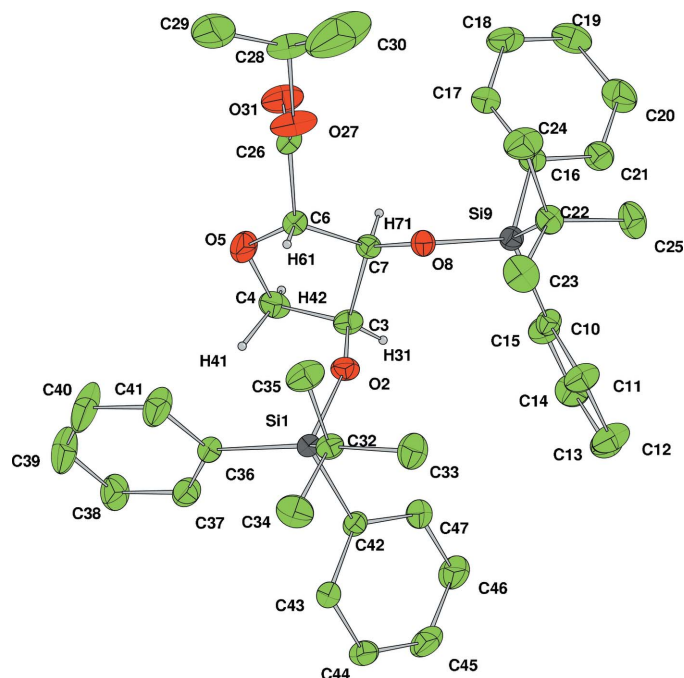


Figure 1

The structure of one molecule of the title compound with displacement ellipsoids drawn at the 50% probability level. All H atoms, except for H31, H41, H61 and H71, have been omitted for clarity. The H atoms are drawn with an arbitrary radius.

Experimental

Epimer (3) was converted to the corresponding disilyl ether by standard procedures (Sanjayan *et al.*, 2003) and was crystallized from ethyl acetate–hexane (1:4).

Crystal data

$C_{40}H_{50}O_5Si_2$	$D_x = 1.184 \text{ Mg m}^{-3}$
$M_r = 667.01$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 8312 reflections
$a = 17.2952 (2) \text{ \AA}$	$\theta = 5\text{--}30^\circ$
$b = 10.7468 (2) \text{ \AA}$	$\mu = 0.14 \text{ mm}^{-1}$
$c = 20.4914 (4) \text{ \AA}$	$T = 190 \text{ K}$
$\beta = 100.7864 (5)^\circ$	Prism, colourless
$V = 3741.40 (11) \text{ \AA}^3$	$0.40 \times 0.20 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	17372 independent reflections
ω scans	17372 reflections with $I > -3\sigma(I)$
Absorption correction: multi-scan (<i>DENZO/SCALEPACK</i> ; Otwinowski & Minor, 1997)	$R_{\text{int}} = 0.027$
$T_{\text{min}} = 0.70$, $T_{\text{max}} = 0.97$	$\theta_{\text{max}} = 30.0^\circ$
27685 measured reflections	$h = -24 \rightarrow 24$
	$k = -15 \rightarrow 15$
	$l = -28 \rightarrow 28$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F^2) + (0.02P)^2 + 2.21P]$
$R[F^2 > 2\sigma(F^2)] = 0.050$	where $P = [\max(F_o^2, 0) + 2F_c^2]/3$
$wR(F^2) = 0.096$	$(\Delta/\sigma)_{\text{max}} = 0.003$
$S = 0.97$	$\Delta\rho_{\text{max}} = 0.70 \text{ e \AA}^{-3}$
17372 reflections	$\Delta\rho_{\text{min}} = -0.47 \text{ e \AA}^{-3}$
848 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	17372 Friedel pairs
	Flack parameter: 0.05 (7)

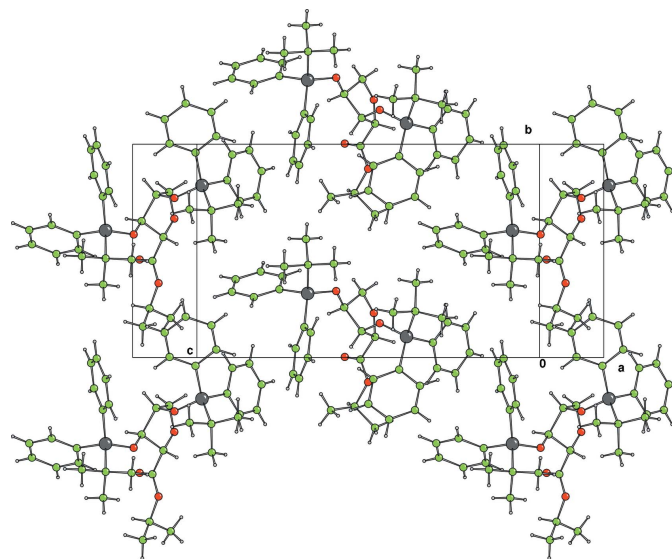


Figure 2

The crystal structure projected on to the bc plane.

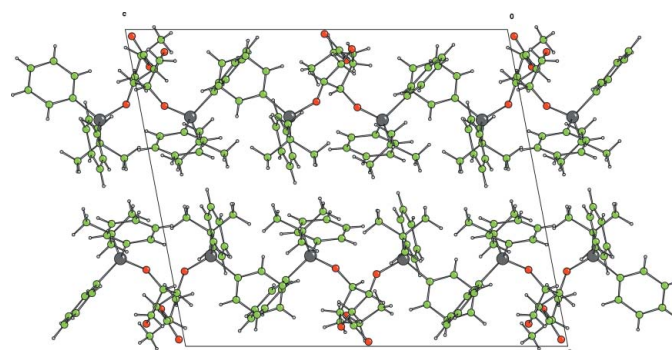


Figure 3

The crystal structure projected along the b axis, showing two hydrophobic faces opposing each other. By symmetry, pairs of hydrophilic faces also oppose each other.

The H atoms were all located in a difference map, but those attached to C atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry ($C-H = 0.93\text{--}0.98 \text{ \AA}$) and displacement parameters [$U_{\text{iso}}(H) = 1.2\text{--}1.5U_{\text{eq}}(\text{parent atom})$], after which they were refined with riding constraints. The pseudo-centre of inversion did not lead to any refinement problems.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO/SCALEPACK*; data reduction: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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