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(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl 3,5-bis-*O*-(4-chlorobenzoyl)-2-deoxy-α-D-*erythro*-ribofuranoside

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.072
Data-to-parameter ratio = 18.7

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

The molecule of the title compound, $C_{29}H_{34}Cl_2O_6$, possesses normal geometric parameters. The absolute configuration was determined from both the synthetic precursor and anomalous scattering effects. Non-classical C—H···O hydrogen bonds link the molecules in the crystal structure into infinite chains along the *a* axis.

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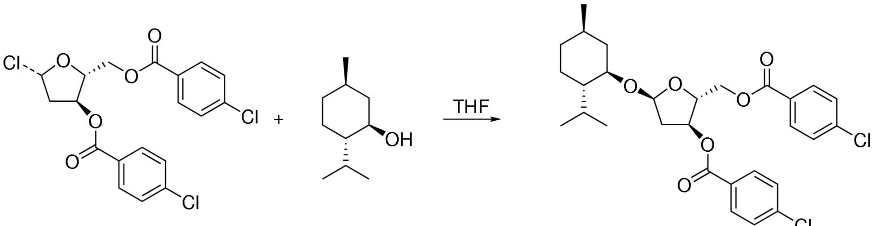
Comment

Fig. 1 shows the structure of the title compound, (3). Selected molecular parameters and hydrogen-bond geometric characteristics are listed in Tables 1 and 2, respectively.



(1) (2) (3)

The absolute configuration was found to be the same as that of the starting material, (1*R*,2*S*,5*R*)-2-(1-methylethyl)-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 $P2_12_12_1$, with one molecule in the asymmetric unit. In the crystal structure, non-classical C—H···O hydrogen bonds play an important role in the formation of polymeric chains running along the crystallographic *a* axis (Fig. 2). The dihedral angle between the two benzene rings is 27.40 (11)°.

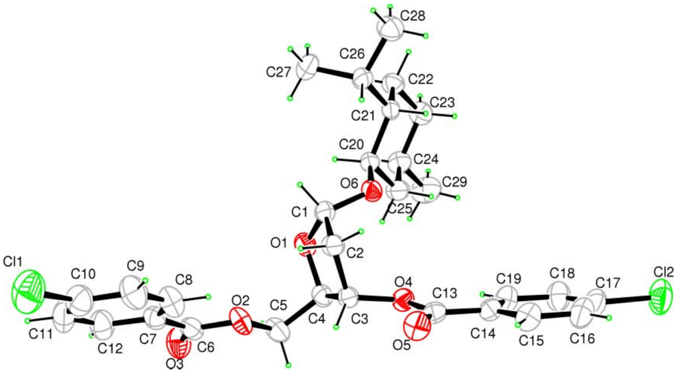


Figure 1
The molecular structure of compound (3). Displacement ellipsoids for the non-H atoms are drawn at the 30% probability level.

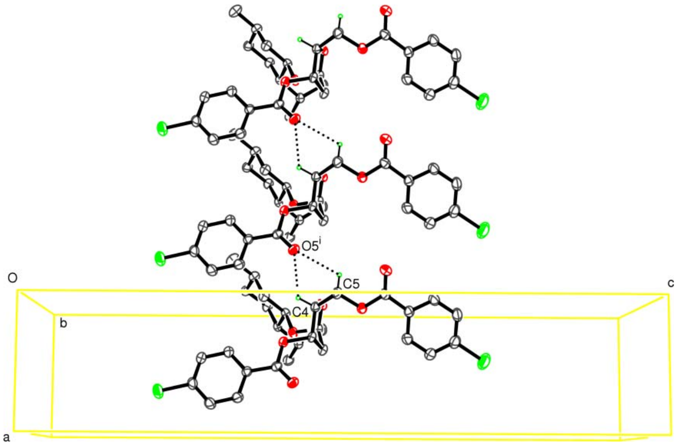


Figure 2
The molecular packing of (3), viewed approximately along the *b* axis. Dashed lines indicate the hydrogen-bonding interactions. H atoms not involved in the hydrogen bonding have been omitted (see Table 2 for symmetry code).

Experimental

The title compound was prepared by reaction of 2-deoxy-3,5-di-*O*-(*p*-chlorobenzoyl)-*D*-*erythro*-pentofuranosyl chloride (0.01 mol) with L-menthol (0.012 mol) in tetrahydrofuran (50 ml) at room temperature for 4 h (reaction monitored by thin-layer chromatography). The resulting mixture was concentrated to dryness. The crude product, which consists of the α and β anomers, was separated by chromatography on silica gel and eluted with 2:1 dichloromethane–petroleum ether, in a yield of 26 and 16%, respectively. The title compound (α anomer) was recrystallized by slow evaporation of a dichloromethane–petroleum ether (2:1) solution. M.p. 368–369 K. ^{1}H NMR ($CDCl_3$, p.p.m.): δ 7.99–7.95 (4H, *m*), 7.43–7.38 (4H, *m*), 5.39, 5.38 (1H, *d*, J = 5.6 Hz), 5.37 (1H, *m*), 4.62–4.49 (3H, *m*), 3.39–3.33 (1H, *m*), 2.55–2.48 (1H, *m*), 2.20–2.17 (2H, *m*), 2.11–2.07 (1H, *m*), 1.66–1.60 (2H, *m*), 1.42–1.19 (3H, *m*), 1.08–0.76 (13H, *m*); ^{13}C NMR ($CDCl_3$, p.p.m.): δ 165.55, 165.42, 139.80, 139.69, 131.22, 131.11, 128.83, 128.81, 128.32, 128.15, 104.93, 80.14, 79.38, 74.99, 71.59, 64.78, 50.18, 48.71, 45.13, 43.46, 39.53, 34.61, 34.41, 31.71, 25.89, 23.38, 22.37, 21.15, 16.42. MS *m*/*z* (%): 411 (0.14), 393 (1.58), 241 (0.76), 156 (2/3), 139 (27.84), 111 (8.13), 97 (4.41), 81 (100.0), 75 (4.11), 69 (12.03), 55 (15.12), 43 (9.12).

Crystal data

$C_{29}H_{34}Cl_2O_6$
M_r = 549.46
Orthorhombic, $P2_12_12_1$
a = 6.5720 (9) Å
b = 14.3913 (2) Å
c = 30.5468 (3) Å
V = 2889.1 (4) Å^3
Z = 4
D_x = 1.263 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 5980 reflections
θ = 1.3–27.5°
μ = 0.26 mm^{-1}
T = 295 (2) K
Prism, colourless
0.31 × 0.30 × 0.29 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.824, T_{max} = 0.926
23110 measured reflections

6328 independent reflections
4079 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.5°
h = −8 → 8
k = −18 → 18
l = −39 → 39

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.073
S = 0.99
6328 reflections
338 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0264P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0097 (5)
Absolute structure: Flack (1983), 2532 Friedel pairs
Flack parameter: −0.04 (6)

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.403 (2)	C10—Cl1	1.741 (2)
C1—O6	1.4036 (18)	C13—O5	1.2058 (19)
C3—O4	1.4460 (18)	C17—Cl2	1.738 (2)
O1—C1—O6	112.63 (14)	O5—C13—O4	123.47 (17)
O1—C1—C2	105.72 (13)	C18—C17—Cl2	119.10 (18)
O6—C1—C2	108.86 (14)	C13—O4—C3	115.46 (13)
O1—C4—C3	106.35 (13)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O5^{i}	0.98	2.48	3.142 (2)	124
C5—H5A···O5^{i}	0.97	2.60	3.038 (2)	107

Symmetry code: (i) $x - 1, y, z$.

The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with U_{iso}(H) = 1.5U_{eq}(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_{iso}(H) = 1.2U_{eq}(carrier atom), and included in the final cycles of refinement in the riding-model approximation.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 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).

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