Acta Crystallographica Section C
Structural Chemistry
ISSN 2053-2296

Three different fluoro- or chloro-substituted 1′-deoxy-1′-phenyl-*β*-D-ribofuranoses

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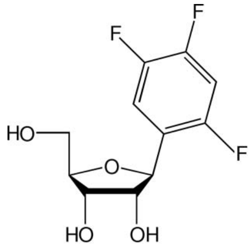
Received 3 February 2014
Accepted 4 March 2014

Crystal structures are reported for three fluoro- or chloro-substituted 1′-deoxy-1′-phenyl-*β*-D-ribofuranoses, namely 1′-deoxy-1′-(2,4,5-trifluorophenyl)-*β*-D-ribofuranose, $C_{11}H_{11}F_3O_4$, (I), 1′-deoxy-1′-(2,4,6-trifluorophenyl)-*β*-D-ribofuranose, $C_{11}H_{11}F_3O_4$, (II), and 1′-(4-chlorophenyl)-1′-deoxy-*β*-D-ribofuranose, $C_{11}H_{13}ClO_4$, (III). The five-membered furanose ring of the three compounds has a conformation between a C2′-*endo*,C3′-*exo* twist and a C2′-*endo* envelope. The ribofuranose groups of (I) and (III) are connected by intermolecular O—H⋯O hydrogen bonds to six symmetry-related molecules to form double layers, while the ribofuranose group of (II) is connected by O—H⋯O hydrogen bonds to four symmetry-related molecules to form single layers. The O⋯O contact distance of the O—H⋯O hydrogen bonds ranges from 2.7172 (15) to 2.8895 (19) Å. Neighbouring double layers of (I) are connected by a very weak intermolecular C—F⋯π contact. The layers of (II) are connected by one C—H⋯O and two C—H⋯F contacts, while the double layers of (III) are connected by a C—H⋯Cl contact. The conformations of the molecules are compared with those of seven related molecules. The orientation of the benzene ring is coplanar with the H—C1′ bond or bisecting the H—C1′—C2′ angle, or intermediate between these positions. The orientation of the benzene ring is independent of the substitution pattern of the ring and depends mainly on crystal-packing effects.

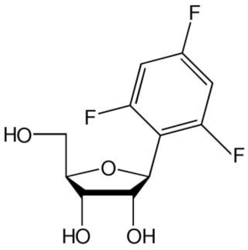
Keywords: crystal structure; nucleic acids; *β*-D-ribofuranose; hydrogen bonding; crystal-packing effects.

1. Introduction

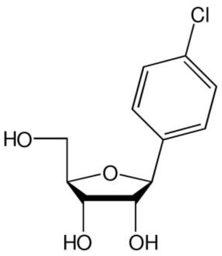
The stability of nucleic acids is mainly determined by hydrogen bonding, base stacking and solvation. In order to probe these interactions, a common approach is to replace the natural nucleobases by nonpolar nucleoside isosteres (Gohlke *et al.*, 2012). The shape and size of the modified base should be kept as similar as possible to that of the native base. This concept, introduced by Kool (2001), has been extended by us for RNA. A series of halogenated nucleosides was prepared and their ability to form duplexes was determined (Parsch & Engels, 2002). The crystal structures of a number of fluoro-substituted 1′-deoxy-1′-phenyl-*β*-D-ribofuranoses have already been reported (Bats *et al.*, 1999*a*,*b*, 2000). We report here the structures of three new compounds, namely 1′-deoxy-1′-(2,4,5-trifluorophenyl)-*β*-D-ribofuranose, (I), 1′-deoxy-1′-(2,4,6-trifluorophenyl)-*β*-D-ribofuranose, (II), and 1′-(4-chlorophenyl)-1′-deoxy-*β*-D-ribofuranose, (III).



(I)



(II)



(III)

2. Experimental

2.1. Synthesis and crystallization

The title compounds were prepared according to the procedure described by Krohn *et al.* (1992). Thus, 1-bromo-2,4,5-trifluorobenzene [for (I)], 2,4,6-trifluorobenzene [for (II)] or 1-bromo-4-chlorobenzene [for (III)] was reacted with *n*-butyllithium and subsequently with 2,3,5-tri-*O*-benzyl-D-ribono-1,4-lactone (Timpe *et al.*, 1975; Barker & Fletcher, 1961) to give the corresponding fluoro- or chloro-substituted 2′,3′,5′-tri-*O*-benzyl-1′-deoxy-1′-phenyl-*β*-D-ribofuranose. Next, the *O*-benzyl protecting groups were removed, using palladium hydroxide on a carbon matrix as catalyst. Compounds (I), (II) and (III) were obtained in yields of 94, 92 and 95%, respectively, and were all recrystallized from water. For full details of the syntheses, see Živković (2005).

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Friedel opposites were merged for (I) and (II), but not for (III), which contains a Cl atom as anomalous scatterer. The C-bound H atoms were positioned geometrically and treated as riding, with C—H = 0.95 Å for aromatic, 0.99 Å for methylene or 1.00 Å for methine C—H groups, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The

Table 1
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{11}H_{11}F_3O_4$	$C_{11}H_{11}F_3O_4$	$C_{11}H_{13}ClO_4$
M_r	264.20	264.20	244.66
Crystal system, space group	Monoclinic, $C2$	Orthorhombic, $P2_12_12$	Orthorhombic, $P2_12_12_1$
Temperature (K)	143	142	145
a, b, c (Å)	13.248 (2), 4.9913 (10), 16.312 (6)	11.209 (3), 20.221 (5), 4.9699 (12)	6.7097 (9), 6.8447 (9), 23.948 (4)
α, β, γ (°)	90, 100.18 (2), 90	90, 90, 90	90, 90, 90
V (Å^3)	1061.6 (5)	1126.4 (5)	1099.9 (3)
Z	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.16	0.15	0.34
Crystal size (mm)	0.60 × 0.38 × 0.10	0.55 × 0.30 × 0.14	0.50 × 0.20 × 0.14
Data collection			
Diffractometer	Siemens SMART 1K CCD area-detector diffractometer	Siemens SMART 1K CCD area-detector diffractometer	Siemens SMART 1K CCD area-detector diffractometer
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2000)	Numerical (*SHELXTL*; Sheldrick, 2008)	Numerical (*SHELXTL*; Sheldrick, 2008)
T_{min}, T_{max}	0.853, 0.984	0.930, 0.980	0.872, 0.955
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11403, 1924, 1774	19902, 2255, 2139	24334, 4164, 3470
R_{int}	0.036	0.033	0.041
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.735	0.746	0.777
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.080, 1.09	0.031, 0.082, 1.14	0.040, 0.091, 1.04
No. of reflections	1924	2255	4164
No. of parameters	175	175	157
No. of restraints	1	0	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.20	0.31, −0.17	0.31, −0.46
Absolute structure	Known from the synthesis	Known from the synthesis	Flack (1983), with 1707 Friedel pairs
Absolute structure parameter			−0.01 (6)

Computer programs: *SMART* (Siemens, 1995), *SAINT* (Siemens, 1995), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008) and *SHELXTL* (Sheldrick, 2008).

O-bound H atoms were taken from difference Fourier syntheses, and their coordinates and isotropic displacement parameters were refined.

3. Results and discussion

The molecular structure of (I) is shown in Fig. 1. The five-membered furanose ring has a conformation approximately between a C2′-*endo* envelope and a C2′-*endo*,C3′-*exo* twist. The phenyl group attached to C1′ and the hydroxy group attached to C2′ are in pseudo-equatorial positions, and the hydroxy group attached to C3′ is in a pseudo-axial position with respect to the furanose ring. The trifluorophenyl group is planar [r.m.s. deviation of the C and F atoms from the plane = 0.0044 (14) Å].

The ribofuranose groups of (I) are connected by intermolecular O—H⋯O hydrogen bonds (Table 2 and Fig. 2) to form double layers parallel to (001). Each molecule is hydrogen bonded to six symmetry-related molecules. The molecules within a double layer are further stabilized by a rather short intermolecular C—H⋯O contact, with an H⋯O distance of 2.32 Å, and by four weak C—H⋯F interactions (involving atoms F2 and F5), with H⋯F distances between 2.59 and 2.75 Å (Table 2). There is only one relevant intermolecular interaction among the double layers along the *c*-axis direction with a contact distance shorter than the van der Waals contact distance (Bondi, 1964); this is a weak intermolecular C—F⋯π contact [C5—F5⋯C4^{vi}; symmetry code: (vi) $-x + \frac{1}{2}$, $y + \frac{1}{2}$, $-z + 1$], with an F⋯C distance of 3.047 (2) Å and a C—F⋯C angle of 148.25 (10)°. This interaction is expected to be very weak. Therefore, the crystal habit is a (001) plate.

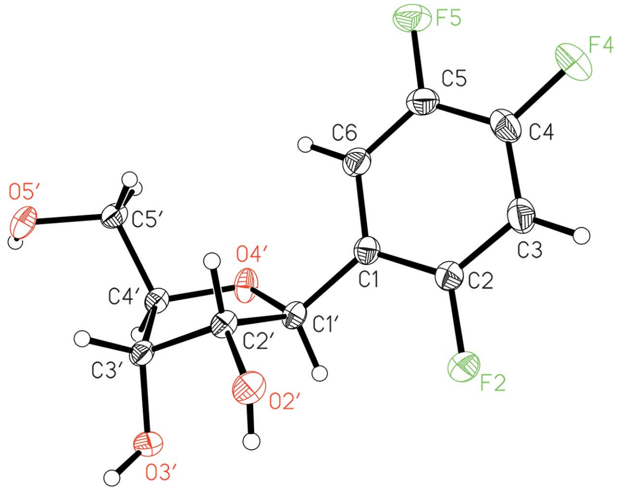


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

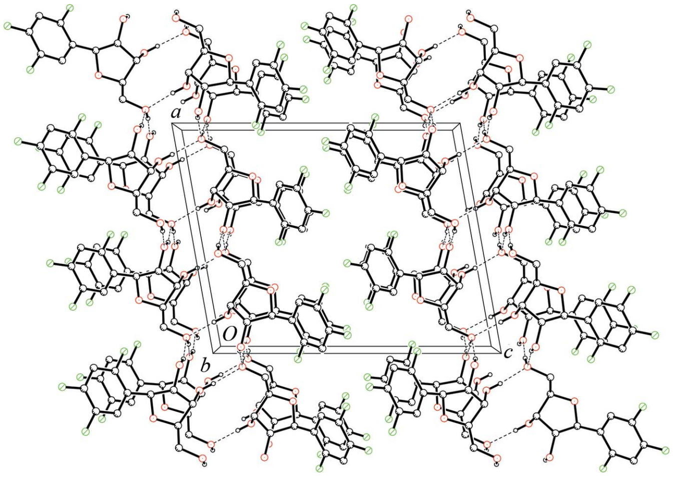


Figure 2
The crystal packing of (I), viewed down [010], showing the hydrogen-bonded double layers. C-bound H atoms have been omitted for clarity. Intermolecular O—H···O hydrogen bonds are shown as dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2′—H2*B*···O5′^{i}	0.80 (3)	2.10 (3)	2.8895 (19)	166 (3)
O3′—H3*B*···O5′^{ii}	0.88 (3)	1.92 (3)	2.7738 (19)	164 (4)
O5′—H5*C*···O2′^{iii}	0.80 (3)	1.98 (3)	2.7795 (18)	171 (3)
C2′—H2*A*···O3′^{iv}	1.00	2.32	3.166 (2)	142
C5′—H5*A*···F2^{v}	0.99	2.67	3.081 (2)	105
C5′—H5*B*···F2^{v}	0.99	2.59	3.081 (2)	110
C6—H6*A*···F2^{v}	0.95	2.74	3.6340 (19)	157
C3—H3*C*···F5^{i}	0.95	2.75	3.640 (2)	157

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

The crystal structure of (I) is isomorphous with that of 1′-deoxy-1′-(2-fluorophenyl)-β-D-ribofuranose (Bats *et al.*, 1999*a*). As the 2-fluoro substituent is the only F atom occuring in both compounds, this atom may be the stabilizing factor resulting in the observed crystal packing, while the 4-fluoro and 5-fluoro substituents have little effect on the packing of the structure. This is in agreement with the intermolecular contacts presented in Table 2. Thus, the related compound 1′-deoxy-1′-(2,4-difluorophenyl)-β-D-ribofuranose (Bats *et al.*, 2000) could also be expected to have the same crystal structure. The space group of this structure is also *C*2 and the unit-cell dimensions show some similarity with those of (I). However, the hydrogen bonding within the double layers of the compound differs from that observed in (I), showing the crystal structures to be different.

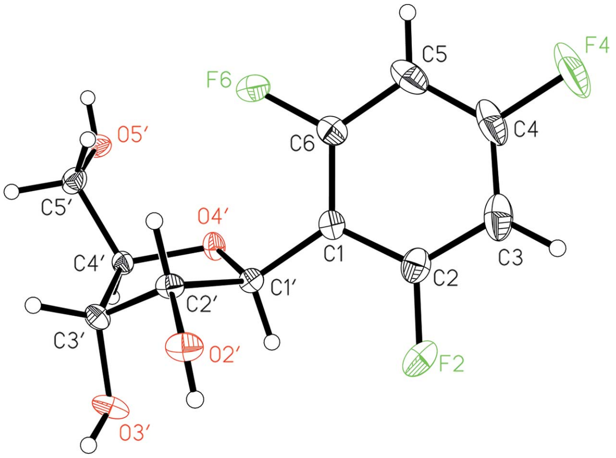


Figure 3
The molecular structure of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

The molecular structure of (II) is shown in Fig. 3. The five-membered furanose ring has an approximately C2′-*endo* envelope conformation. The trifluorophenyl group is in a pseudo-equatorial position with respect to the furanose ring. It adopts an orientation with the C2—F2 and C1′—H1*A* bonds in synperiplanar positions (torsion angle H1*A*—C1′—C1—C2 = −13°), resulting in an intramolecular H1*A*···F2 distance of 2.35 Å, which is shorter than the van der Waals contact distance. The trifluorophenyl group shows a small deviation from planarity [r.m.s. deviation of the C and F atoms from the plane = 0.0270 (13) Å]. Substituent atoms C1′ and F6 deviate from the benzene plane by 0.110 (2) Å in opposite directions. This deviation from planarity may result from steric repulsion between atoms F6 and O4′. The observed F6···O4′ distance of 2.8183 (15) Å is slightly shorter than the van der Waals contact distance.

The ribofuranose groups of (II) are connected by intermolecular O—H···O hydrogen bonds (Table 3 and Fig. 4) to four symmetry-related molecules to form layers parallel to (010). The molecules within a layer are also stabilized by a very weak intermolecular O—H···F and C—H···F contact (third and fifth entries in Table 3). The molecular layers are connected along the *b*-axis direction by an intermolecular C—H···O interaction, with an H···O distance of 2.55 Å, and by

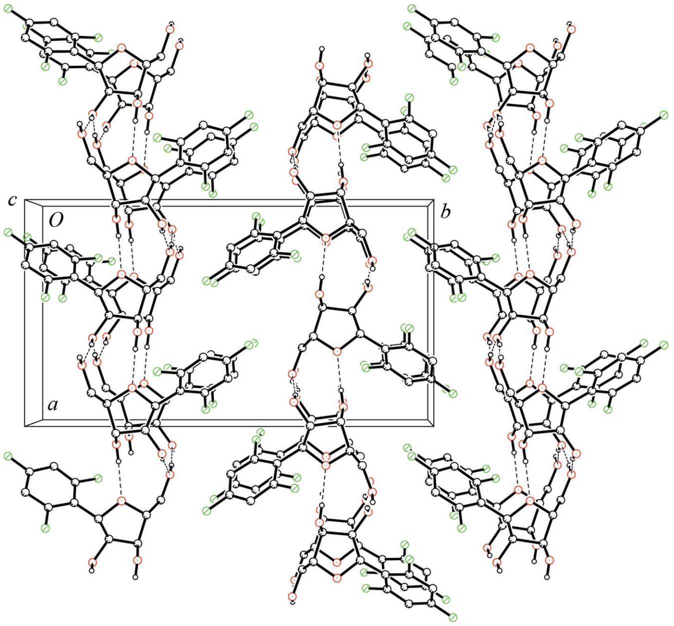


Figure 4
The crystal packing of (II), viewed down [001], showing the hydrogen-bonded layers. C-bound H atoms have been omitted for clarity. Intermolecular O—H···O hydrogen bonds are shown as dashed lines.

Table 3
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
O2′—H2B···O5′^{i}	0.78 (3)	2.05 (3)	2.8157 (17)	168 (2)
O3′—H3B···O4′^{i}	0.85 (3)	1.97 (3)	2.8151 (16)	175 (2)
O3′—H3B···F6^{i}	0.85 (3)	2.53 (2)	2.8865 (15)	106.1 (19)
O5′—H5C···O2′^{ii}	0.81 (3)	1.97 (3)	2.7719 (17)	175 (3)
C3′—H3A···F6^{iii}	1.00	2.48	3.4024 (17)	153
C4′—H4A···F4^{iv}	1.00	2.62	3.5489 (19)	154
C5′—H5A···F4^{v}	0.99	2.53	3.2788 (19)	132
C3—H3C···O5′^{vi}	0.95	2.55	3.448 (2)	159

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+2$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iv) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+2$; (v) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+1$; (vi) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+2$.

two intermolecular C—H···F interactions (both involving the 4-fluoro substituent), with H···F distances of 2.53 and 2.62 Å (last three entries of Table 3).

The molecular structure of (III) is shown in Fig. 5. The crystal structure is isomorphous with that of one of the two polymorphs of 1′-deoxy-1′-(4-fluorophenyl)-β-D-ribofuranose reported by Bats *et al.* (2000). The ribofuranose ring has a conformation near a C2′-*endo*,C3′-*exo* twist. The conformation of the molecule is rather similar to those observed in 1′-deoxy-1′-(3-fluorophenyl)-β-D-ribofuranose (Bats *et al.*, 1999*b*) and 1′-deoxy-1′-phenyl-β-D-ribofuranose (Matulic-Adamic *et al.*, 1996; Štambaský *et al.*, 2011). The phenyl group attached to C1′ and the hydroxy group attached to C2′ are in pseudo-equatorial positions, and the hydroxy group attached to C3′ is in a pseudo-axial position with respect to the five-membered ring. The shortest intramolecular contact distance is 2.43 Å between atoms O4′ and H6A. The benzene ring is planar [r.m.s. deviation of the C atoms from the plane = 0.0068 (11) Å]. The Cl atom is displaced by 0.064 (2) Å from the benzene plane as a result of crystal-packing effects.

The ribofuranose groups of (III) are connected by intermolecular O—H···O hydrogen bonds (Table 4 and Fig. 6) to six symmetry-related molecules to form double layers parallel to (001). The molecules within a double layer are also connected by a weak intermolecular C—H···O contact, with an H···O distance of 2.58 Å. The double layers are connected along the *c*-axis direction by a rather weak intermolecular C—H···Cl contact, with an H···Cl distance of 2.74 Å.

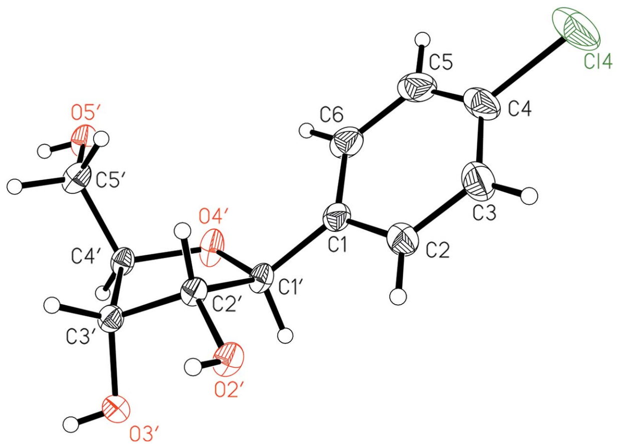


Figure 5
The molecular structure of (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

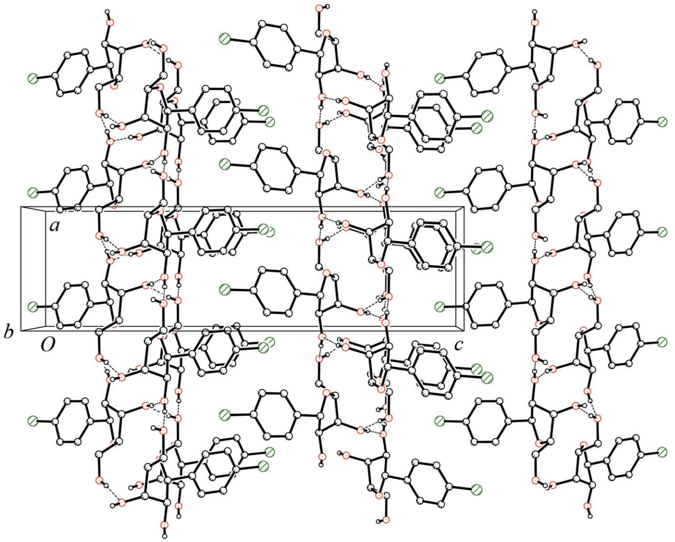


Figure 6
The crystal packing of (III), viewed down [010], showing the hydrogen-bonded double layers. C-bound H atoms have been omitted for clarity. Intermolecular O—H···O hydrogen bonds are shown as dashed lines.

Table 4
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
O2′—H2B···O5′^{i}	0.75 (3)	1.99 (3)	2.7172 (15)	162 (3)
O3′—H3B···O2′^{ii}	0.73 (2)	2.00 (2)	2.7220 (15)	172 (2)
O5′—H5C···O3′^{iii}	0.76 (2)	2.05 (2)	2.8077 (15)	174 (2)
C4′—H4A···O4′^{iii}	1.00	2.58	3.3584 (17)	135
C3—H3C···Cl4^{iv}	0.95	2.74	3.5902 (19)	150

Symmetry codes: (i) $x-1, y, z$; (ii) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $x-\frac{1}{2}, -y+\frac{5}{2}, -z+1$.

The conformations of the ribofuranose rings and the relative orientations of the phenyl substituents in (I), (II) and (III) are compared in Table 5 with the corresponding values observed in the crystal structures of related compounds. The conformation of the five-membered ring is characterized by the ring-puckering parameters q and φ as defined by Cremer & Pople (1975). The value of q (0.37–0.42 Å) is rather constant for all ten entries. The value of the rotation angle φ varies between 60 and 87° in nine structures. This corresponds to a conformation ranging from one intermediate between a C1′-*exo*,C2′-*endo* twist and a C2′-*endo* envelope to a C2′-*endo*,C3′-*exo* twist. Only one entry in Table 5 shows a very different conformation with φ = 275°, corresponding to an unsymmetrical C2′-*exo*,C3′-*endo* twist conformation. These findings are in agreement with the observation by Murray-Rust & Motherwell (1978) that conformations near C2′-*endo* and C3′-*endo* envelopes are most favoured. The torsion angle O4′—C1′—C1—C6 is in the range 2–50°. In three structures, a value between 45 and 50° is observed and the benzene ring is almost coplanar with the H—C1′ bond (the H—C1′—C1—C2 torsion angle ranges from −16 to −4°). Five structures show an O4′—C1′—C1—C6 torsion angle between 2 and 9°. In this case, the benzene ring has an orientation bisecting the H—C1′—C2′ angle. Two structures have an intermediate orientation of the benzene ring, with O4′—C1′—C1—C6 torsion angles of 27 and 29°. The relative orientation of the benzene ring does not depend on the substitution pattern of this ring. Even for the two polymorphs of the 4-fluorophenyl compound, large differences were observed (Bats *et al.*, 2000). Thus, the orientation of the benzene ring is mainly determined by crystal-packing effects.

Table 5

Ring-puckering parameters q (Å) and φ (°), as defined by Cremer & Pople (1975), and selected torsion angles (°) in (I)–(III) and in a number of related compounds (numbering scheme as in Figs. 1, 3 and 5).

	q	φ	O4′—C1′—C1—C6	H—C1′—C1—C2
(I)	0.3764 (16)	80.0 (2)	29.32 (19)	−33
(II)	0.4162 (14)	77.3 (2)	46.99 (17)	−13
(III)	0.3744 (14)	84.8 (2)	2.25 (18)	−57
(*a*), polymorph 1	0.3752 (11)	60.21 (17)	50.34 (14)	−4
(*a*), polymorph 2	0.3685 (12)	81.57 (17)	8.80 (15)	−50
(*a*), hemihydrate	0.4214 (12)	63.39 (16)	44.96 (15)	−16
(*b*)	0.3901 (18)	274.8 (2)	6.8 (2)	−57
(*c*)	0.3767 (13)	86.71 (18)	4.62 (16)	−53
(*d*)	0.3850 (10)	74.75 (15)	27.35 (13)	−35
(*e*)	0.3740 (17)	84.3 (2)	7.4 (2)	−52

Notes: (*a*) 1′-deoxy-1′-(4-fluorophenyl)-β-D-ribofuranose (Bats *et al.*, 2000); (*b*) 1′-deoxy-1′-(2,4-difluorophenyl)-β-D-ribofuranose (Bats *et al.*, 2000); (*c*) 1′-deoxy-1′-(3-fluorophenyl)-β-D-ribofuranose (Bats *et al.*, 1999*b*); (*d*) 1′-deoxy-1′-(2-fluorophenyl)-β-D-ribofuranose (Bats *et al.*, 1999*a*); (*e*) 1′-deoxy-1′-phenyl-β-D-ribofuranose (Štambaský *et al.*, 2011).

Supporting information for this paper is available from the IUCr electronic archives (Reference: EG3152).

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supplementary materials

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Three different fluoro- or chloro-substituted 1′-deoxy-1′-phenyl-*β*-D-ribofuranoses

Jan W. Bats, Aleksandra Živković, Jörg Parsch and Joachim W. Engels

Computing details

For all compounds, data collection: *SMART* (Siemens, 1995); cell refinement: *SMART* (Siemens, 1995); data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

(I) 1′-Deoxy-1′-(2,4,5-trifluorophenyl)-*β-D*-ribofuranose

Crystal data

$C_{11}H_{11}F_3O_4$	$F(000) = 544$
$M_r = 264.20$	$D_x = 1.653$ Mg m^{-3}
Monoclinic, *C*2	Mo *Kα* radiation, $\lambda = 0.71073$ Å
Hall symbol: C 2y	Cell parameters from 84 reflections
$a = 13.248$ (2) Å	$\theta = 3$–$23°$
$b = 4.9913$ (10) Å	$\mu = 0.16$ mm^{-1}
$c = 16.312$ (6) Å	$T = 143$ K
$\beta = 100.18$ (2)°	Plate, colourless
$V = 1061.6$ (5) Å^3	0.60 × 0.38 × 0.10 mm
$Z = 4$	

Data collection

Siemens SMART 1K CCD area-detector diffractometer	11403 measured reflections
Radiation source: normal-focus sealed tube	1924 independent reflections
Graphite monochromator	1774 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.036$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)	$\theta_{max} = 31.5°$, $\theta_{min} = 2.5°$
$T_{min} = 0.853$, $T_{max} = 0.984$	$h = -19 \rightarrow 19$
	$k = -7 \rightarrow 7$
	$l = -22 \rightarrow 23$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.030$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.080$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.09$	
1924 reflections	
175 parameters	
1 restraint	

$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.3P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.34$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
F2	−0.01656 (7)	0.2424 (2)	0.29412 (7)	0.0294 (3)
F4	0.10539 (9)	0.9400 (3)	0.47910 (7)	0.0363 (3)
F5	0.28911 (8)	0.9152 (3)	0.43078 (7)	0.0333 (3)
O2′	0.03839 (8)	0.3322 (3)	0.10934 (8)	0.0205 (2)
H2B	0.0174 (19)	0.183 (7)	0.0987 (16)	0.037 (7)*
O3′	0.16450 (8)	−0.0876 (3)	0.07279 (8)	0.0206 (2)
H3B	0.134 (2)	−0.083 (8)	0.0205 (18)	0.055 (8)*
O4′	0.27512 (8)	0.1283 (3)	0.24105 (7)	0.0208 (2)
O5′	0.43560 (8)	0.3197 (3)	0.08891 (8)	0.0212 (2)
H5C	0.4692 (19)	0.185 (7)	0.0912 (15)	0.037 (7)*
C1′	0.16895 (10)	0.2009 (3)	0.23188 (9)	0.0164 (3)
H1A	0.1261	0.0370	0.2338	0.020*
C2′	0.14429 (10)	0.3297 (3)	0.14487 (9)	0.0155 (3)
H2A	0.1699	0.5186	0.1489	0.019*
C3′	0.21087 (10)	0.1656 (3)	0.09543 (9)	0.0158 (3)
H3A	0.2264	0.2658	0.0460	0.019*
C4′	0.30682 (10)	0.1185 (3)	0.16077 (9)	0.0158 (3)
H4A	0.3355	−0.0627	0.1523	0.019*
C5′	0.38994 (10)	0.3280 (3)	0.16230 (10)	0.0186 (3)
H5A	0.3602	0.5079	0.1673	0.022*
H5B	0.4438	0.2987	0.2119	0.022*
C1	0.15409 (11)	0.3905 (3)	0.30037 (9)	0.0174 (3)
C2	0.06057 (11)	0.4104 (4)	0.32753 (10)	0.0204 (3)
C3	0.04071 (12)	0.5920 (4)	0.38678 (11)	0.0249 (3)
H3C	−0.0244	0.5999	0.4034	0.030*
C4	0.11933 (13)	0.7610 (4)	0.42062 (10)	0.0243 (3)
C5	0.21418 (12)	0.7470 (4)	0.39567 (10)	0.0228 (3)
C6	0.23187 (12)	0.5644 (3)	0.33689 (10)	0.0207 (3)
H6A	0.2974	0.5562	0.3209	0.025*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F2	0.0197 (4)	0.0350 (6)	0.0353 (6)	−0.0076 (4)	0.0097 (4)	−0.0119 (5)

F4	0.0459 (6)	0.0344 (6)	0.0304 (6)	−0.0013 (6)	0.0118 (5)	−0.0145 (5)
F5	0.0366 (5)	0.0330 (6)	0.0291 (5)	−0.0148 (5)	0.0024 (4)	−0.0079 (5)
O2′	0.0145 (4)	0.0224 (6)	0.0241 (6)	0.0021 (4)	0.0024 (4)	0.0015 (5)
O3′	0.0241 (5)	0.0171 (5)	0.0209 (5)	−0.0057 (4)	0.0048 (4)	−0.0021 (5)
O4′	0.0160 (4)	0.0288 (6)	0.0184 (5)	0.0059 (4)	0.0054 (4)	0.0059 (5)
O5′	0.0153 (4)	0.0239 (6)	0.0258 (6)	0.0016 (5)	0.0075 (4)	0.0072 (5)
C1′	0.0135 (5)	0.0179 (7)	0.0186 (7)	0.0005 (5)	0.0045 (5)	0.0020 (5)
C2′	0.0135 (5)	0.0146 (6)	0.0185 (7)	0.0011 (5)	0.0034 (5)	0.0021 (5)
C3′	0.0162 (5)	0.0145 (6)	0.0176 (7)	−0.0017 (5)	0.0051 (5)	0.0015 (5)
C4′	0.0151 (5)	0.0141 (6)	0.0191 (7)	0.0009 (5)	0.0051 (5)	0.0014 (5)
C5′	0.0163 (5)	0.0173 (6)	0.0227 (7)	−0.0022 (5)	0.0050 (5)	0.0004 (6)
C1	0.0187 (6)	0.0176 (7)	0.0162 (6)	0.0000 (5)	0.0039 (5)	0.0021 (5)
C2	0.0186 (6)	0.0227 (7)	0.0203 (7)	−0.0013 (6)	0.0045 (5)	−0.0017 (6)
C3	0.0233 (7)	0.0293 (8)	0.0238 (8)	0.0017 (6)	0.0089 (6)	−0.0038 (7)
C4	0.0327 (8)	0.0237 (8)	0.0167 (7)	0.0020 (6)	0.0050 (6)	−0.0022 (6)
C5	0.0267 (7)	0.0213 (7)	0.0194 (7)	−0.0062 (6)	0.0013 (6)	0.0001 (6)
C6	0.0206 (6)	0.0229 (7)	0.0186 (7)	−0.0034 (6)	0.0034 (5)	0.0016 (6)

Geometric parameters (Å, º)

F2—C2	1.3590 (19)	C2′—H2A	1.0000
F4—C4	1.343 (2)	C3′—C4′	1.526 (2)
F5—C5	1.3482 (19)	C3′—H3A	1.0000
O2′—C2′	1.4202 (17)	C4′—C5′	1.516 (2)
O2′—H2B	0.80 (3)	C4′—H4A	1.0000
O3′—C3′	1.4249 (19)	C5′—H5A	0.9900
O3′—H3B	0.88 (3)	C5′—H5B	0.9900
O4′—C1′	1.4347 (16)	C1—C2	1.3912 (19)
O4′—C4′	1.4450 (18)	C1—C6	1.397 (2)
O5′—C5′	1.4342 (19)	C2—C3	1.384 (2)
O5′—H5C	0.80 (3)	C3—C4	1.378 (3)
C1′—C1	1.503 (2)	C3—H3C	0.9500
C1′—C2′	1.540 (2)	C4—C5	1.389 (2)
C1′—H1A	1.0000	C5—C6	1.373 (2)
C2′—C3′	1.533 (2)	C6—H6A	0.9500
C2′—O2′—H2B	111.1 (19)	C5′—C4′—H4A	109.5
C3′—O3′—H3B	109 (2)	C3′—C4′—H4A	109.5
C1′—O4′—C4′	110.53 (11)	O5′—C5′—C4′	112.25 (13)
C5′—O5′—H5C	107.3 (18)	O5′—C5′—H5A	109.2
O4′—C1′—C1	109.66 (12)	C4′—C5′—H5A	109.2
O4′—C1′—C2′	104.25 (11)	O5′—C5′—H5B	109.2
C1—C1′—C2′	112.82 (13)	C4′—C5′—H5B	109.2
O4′—C1′—H1A	110.0	H5A—C5′—H5B	107.9
C1—C1′—H1A	110.0	C2—C1—C6	116.82 (14)
C2′—C1′—H1A	110.0	C2—C1—C1′	121.49 (13)
O2′—C2′—C3′	114.11 (12)	C6—C1—C1′	121.62 (13)
O2′—C2′—C1′	114.41 (11)	F2—C2—C3	117.51 (13)
C3′—C2′—C1′	102.34 (12)	F2—C2—C1	118.50 (14)
O2′—C2′—H2A	108.6	C3—C2—C1	123.99 (15)

C3′—C2′—H2A	108.6	C4—C3—C2	117.22 (14)
C1′—C2′—H2A	108.6	C4—C3—H3C	121.4
O3′—C3′—C4′	108.19 (12)	C2—C3—H3C	121.4
O3′—C3′—C2′	110.39 (11)	F4—C4—C3	120.17 (15)
C4′—C3′—C2′	101.57 (12)	F4—C4—C5	119.11 (15)
O3′—C3′—H3A	112.1	C3—C4—C5	120.71 (15)
C4′—C3′—H3A	112.1	F5—C5—C6	120.43 (15)
C2′—C3′—H3A	112.1	F5—C5—C4	118.70 (15)
O4′—C4′—C5′	106.77 (12)	C6—C5—C4	120.86 (15)
O4′—C4′—C3′	106.75 (11)	C5—C6—C1	120.40 (14)
C5′—C4′—C3′	114.73 (12)	C5—C6—H6A	119.8
O4′—C4′—H4A	109.5	C1—C6—H6A	119.8

C4′—O4′—C1′—C1	−139.83 (13)	C2′—C1′—C1—C2	90.39 (17)
C4′—O4′—C1′—C2′	−18.78 (15)	O4′—C1′—C1—C6	29.32 (19)
O4′—C1′—C2′—O2′	158.76 (13)	C2′—C1′—C1—C6	−86.40 (16)
C1—C1′—C2′—O2′	−82.33 (15)	C6—C1—C2—F2	−179.26 (14)
O4′—C1′—C2′—C3′	34.80 (14)	C1′—C1—C2—F2	3.8 (2)
C1—C1′—C2′—C3′	153.71 (11)	C6—C1—C2—C3	0.9 (2)
O2′—C2′—C3′—O3′	−46.41 (17)	C1′—C1—C2—C3	−176.06 (16)
C1′—C2′—C3′—O3′	77.75 (13)	F2—C2—C3—C4	179.76 (16)
O2′—C2′—C3′—C4′	−160.99 (12)	C1—C2—C3—C4	−0.4 (3)
C1′—C2′—C3′—C4′	−36.84 (13)	C2—C3—C4—F4	−179.28 (16)
C1′—O4′—C4′—C5′	118.09 (13)	C2—C3—C4—C5	0.0 (3)
C1′—O4′—C4′—C3′	−5.08 (16)	F4—C4—C5—F5	−0.4 (2)
O3′—C3′—C4′—O4′	−89.60 (13)	C3—C4—C5—F5	−179.64 (17)
C2′—C3′—C4′—O4′	26.60 (15)	F4—C4—C5—C6	179.14 (16)
O3′—C3′—C4′—C5′	152.34 (12)	C3—C4—C5—C6	−0.1 (3)
C2′—C3′—C4′—C5′	−91.46 (14)	F5—C5—C6—C1	−179.84 (15)
O4′—C4′—C5′—O5′	173.95 (12)	C4—C5—C6—C1	0.6 (2)
C3′—C4′—C5′—O5′	−68.01 (16)	C2—C1—C6—C5	−1.0 (2)
O4′—C1′—C1—C2	−153.89 (14)	C1′—C1—C6—C5	175.95 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2′—H2*B*···O5′^{i}	0.80 (3)	2.10 (3)	2.8895 (19)	166 (3)
O3′—H3*B*···O5′^{ii}	0.88 (3)	1.92 (3)	2.7738 (19)	164 (4)
O5′—H5*C*···O2′^{iii}	0.80 (3)	1.98 (3)	2.7795 (18)	171 (3)
C2′—H2*A*···O3′^{iv}	1.00	2.32	3.166 (2)	142
C5′—H5*A*···F2^{v}	0.99	2.67	3.081 (2)	105
C5′—H5*B*···F2^{v}	0.99	2.59	3.081 (2)	110
C6—H6*A*···F2^{v}	0.95	2.74	3.6340 (19)	157
C3—H3*C*···F5^{i}	0.95	2.75	3.640 (2)	157

Symmetry codes: (i) *x*−1/2, *y*−1/2, *z*; (ii) −*x*+1/2, *y*−1/2, −*z*; (iii) *x*+1/2, *y*−1/2, *z*; (iv) *x*, *y*+1, *z*; (v) *x*+1/2, *y*+1/2, *z*.

(II) 1′-Deoxy-1′-(2,4,6-trifluorophenyl)-*β*-*D*-ribofuranose

Crystal data

$C_{11}H_{11}F_3O_4$	$F(000) = 544$
$M_r = 264.20$	$D_x = 1.558$ Mg m^{-3}
Orthorhombic, $P2_12_12$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2 2ab	Cell parameters from 81 reflections
$a = 11.209$ (3) Å	$\theta = 3$–$23°$
$b = 20.221$ (5) Å	$\mu = 0.15$ mm^{-1}
$c = 4.9699$ (12) Å	$T = 142$ K
$V = 1126.4$ (5) Å^3	Rod, colourless
$Z = 4$	0.55 × 0.30 × 0.14 mm

Data collection

Siemens SMART 1K CCD area-detector diffractometer	19902 measured reflections
Radiation source: normal-focus sealed tube	2255 independent reflections
Graphite monochromator	2139 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.033$
Absorption correction: numerical (*SHELXTL*; Sheldrick, 2008)	$\theta_{max} = 32.0°$, $\theta_{min} = 2.0°$
$T_{min} = 0.930$, $T_{max} = 0.980$	$h = -16 \rightarrow 16$
	$k = -30 \rightarrow 29$
	$l = -7 \rightarrow 7$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.082$	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.25P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.14$	$(\Delta/\sigma)_{max} = 0.001$
2255 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
175 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
F2	0.56794 (9)	0.93713 (5)	1.2726 (2)	0.0287 (2)
F4	0.86049 (13)	1.05097 (6)	0.7823 (3)	0.0540 (4)
F6	0.74939 (8)	0.83724 (4)	0.52339 (19)	0.02270 (19)
O2′	0.38744 (9)	0.84093 (5)	0.8066 (2)	0.0192 (2)

H2B	0.361 (2)	0.8379 (11)	0.951 (5)	0.029 (6)*
O3′	0.41764 (10)	0.72085 (5)	1.1058 (2)	0.0222 (2)
H3B	0.343 (2)	0.7266 (11)	1.092 (5)	0.035 (6)*
O4′	0.67037 (8)	0.76384 (4)	0.9713 (2)	0.01536 (18)
O5′	0.77418 (9)	0.65233 (5)	0.6885 (2)	0.0196 (2)
H5C	0.810 (2)	0.6522 (12)	0.547 (5)	0.037 (7)*
C1′	0.59144 (11)	0.82024 (6)	0.9738 (3)	0.0134 (2)
H1A	0.5572	0.8247	1.1587	0.016*
C2′	0.49068 (11)	0.80116 (6)	0.7814 (3)	0.0145 (2)
H2A	0.5208	0.8045	0.5925	0.017*
C3′	0.47447 (11)	0.72775 (7)	0.8522 (3)	0.0160 (2)
H3A	0.4323	0.7027	0.7069	0.019*
C4′	0.60384 (11)	0.70527 (6)	0.8871 (3)	0.0139 (2)
H4A	0.6077	0.6713	1.0330	0.017*
C5′	0.65670 (12)	0.67670 (7)	0.6325 (3)	0.0182 (2)
H5A	0.6057	0.6403	0.5656	0.022*
H5B	0.6606	0.7113	0.4916	0.022*
C1	0.65950 (12)	0.88247 (6)	0.9098 (3)	0.0160 (2)
C2	0.64719 (14)	0.93884 (7)	1.0673 (3)	0.0219 (3)
C3	0.71151 (18)	0.99670 (8)	1.0297 (4)	0.0318 (4)
H3C	0.7001	1.0344	1.1406	0.038*
C4	0.79263 (17)	0.99621 (8)	0.8226 (4)	0.0332 (4)
C5	0.80965 (15)	0.94338 (8)	0.6513 (4)	0.0283 (3)
H5D	0.8663	0.9448	0.5093	0.034*
C6	0.73965 (13)	0.88817 (7)	0.6978 (3)	0.0190 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F2	0.0350 (5)	0.0259 (4)	0.0252 (4)	0.0076 (4)	0.0023 (4)	−0.0077 (4)
F4	0.0719 (9)	0.0297 (5)	0.0603 (8)	−0.0302 (6)	−0.0085 (8)	0.0141 (6)
F6	0.0219 (4)	0.0274 (4)	0.0188 (4)	0.0012 (3)	0.0038 (3)	−0.0005 (3)
O2′	0.0130 (4)	0.0255 (5)	0.0191 (5)	0.0059 (4)	−0.0005 (4)	0.0036 (4)
O3′	0.0131 (4)	0.0271 (5)	0.0266 (5)	0.0007 (4)	0.0062 (4)	0.0076 (4)
O4′	0.0113 (4)	0.0140 (4)	0.0208 (4)	0.0005 (3)	−0.0038 (4)	−0.0014 (4)
O5′	0.0147 (4)	0.0260 (5)	0.0181 (5)	0.0031 (4)	0.0027 (4)	−0.0010 (4)
C1′	0.0123 (5)	0.0146 (5)	0.0131 (5)	0.0017 (4)	−0.0013 (4)	0.0001 (4)
C2′	0.0106 (5)	0.0197 (5)	0.0133 (5)	0.0021 (4)	−0.0013 (4)	0.0012 (5)
C3′	0.0107 (5)	0.0186 (5)	0.0186 (6)	−0.0015 (4)	−0.0006 (4)	−0.0003 (5)
C4′	0.0120 (5)	0.0142 (5)	0.0155 (5)	−0.0011 (4)	−0.0004 (4)	−0.0015 (4)
C5′	0.0145 (5)	0.0224 (6)	0.0176 (5)	0.0017 (5)	−0.0015 (5)	−0.0051 (5)
C1	0.0152 (5)	0.0149 (5)	0.0179 (6)	0.0008 (4)	−0.0030 (5)	0.0010 (4)
C2	0.0257 (7)	0.0182 (6)	0.0217 (6)	0.0031 (5)	−0.0039 (6)	−0.0008 (5)
C3	0.0444 (9)	0.0164 (6)	0.0346 (8)	−0.0017 (6)	−0.0103 (8)	0.0000 (7)
C4	0.0412 (9)	0.0201 (6)	0.0384 (9)	−0.0122 (6)	−0.0109 (8)	0.0109 (7)
C5	0.0259 (7)	0.0308 (7)	0.0283 (8)	−0.0084 (6)	−0.0023 (6)	0.0117 (7)
C6	0.0182 (6)	0.0200 (5)	0.0189 (6)	−0.0003 (5)	−0.0019 (5)	0.0039 (5)

Geometric parameters (Å, º)

F2—C2	1.3532 (19)	C2′—H2A	1.0000
F4—C4	1.3582 (18)	C3′—C4′	1.5296 (18)
F6—C6	1.3506 (17)	C3′—H3A	1.0000
O2′—C2′	1.4147 (16)	C4′—C5′	1.5120 (19)
O2′—H2B	0.78 (3)	C4′—H4A	1.0000
O3′—C3′	1.4194 (18)	C5′—H5A	0.9900
O3′—H3B	0.85 (3)	C5′—H5B	0.9900
O4′—C1′	1.4435 (15)	C1—C6	1.389 (2)
O4′—C4′	1.4608 (15)	C1—C2	1.3898 (19)
O5′—C5′	1.4332 (16)	C2—C3	1.387 (2)
O5′—H5C	0.81 (3)	C3—C4	1.373 (3)
C1′—C1	1.5054 (18)	C3—H3C	0.9500
C1′—C2′	1.5293 (18)	C4—C5	1.379 (3)
C1′—H1A	1.0000	C5—C6	1.384 (2)
C2′—C3′	1.5363 (18)	C5—H5D	0.9500

C2′—O2′—H2B	110.3 (18)	C5′—C4′—H4A	109.1
C3′—O3′—H3B	111.0 (17)	C3′—C4′—H4A	109.1
C1′—O4′—C4′	109.28 (9)	O5′—C5′—C4′	109.19 (11)
C5′—O5′—H5C	106.6 (18)	O5′—C5′—H5A	109.8
O4′—C1′—C1	110.37 (10)	C4′—C5′—H5A	109.8
O4′—C1′—C2′	104.36 (10)	O5′—C5′—H5B	109.8
C1—C1′—C2′	116.94 (11)	C4′—C5′—H5B	109.8
O4′—C1′—H1A	108.3	H5A—C5′—H5B	108.3
C1—C1′—H1A	108.3	C6—C1—C2	115.04 (13)
C2′—C1′—H1A	108.3	C6—C1—C1′	123.86 (12)
O2′—C2′—C1′	113.91 (11)	C2—C1—C1′	121.08 (12)
O2′—C2′—C3′	115.61 (11)	F2—C2—C3	117.68 (13)
C1′—C2′—C3′	100.83 (10)	F2—C2—C1	117.96 (13)
O2′—C2′—H2A	108.7	C3—C2—C1	124.35 (15)
C1′—C2′—H2A	108.7	C4—C3—C2	116.05 (15)
C3′—C2′—H2A	108.7	C4—C3—H3C	122.0
O3′—C3′—C4′	107.18 (11)	C2—C3—H3C	122.0
O3′—C3′—C2′	110.58 (11)	F4—C4—C3	118.39 (17)
C4′—C3′—C2′	101.60 (10)	F4—C4—C5	117.58 (17)
O3′—C3′—H3A	112.3	C3—C4—C5	124.03 (15)
C4′—C3′—H3A	112.3	C4—C5—C6	116.32 (16)
C2′—C3′—H3A	112.3	C4—C5—H5D	121.8
O4′—C4′—C5′	110.46 (10)	C6—C5—H5D	121.8
O4′—C4′—C3′	105.99 (10)	F6—C6—C5	117.52 (14)
C5′—C4′—C3′	112.96 (11)	F6—C6—C1	118.40 (12)
O4′—C4′—H4A	109.1	C5—C6—C1	124.07 (14)

C4′—O4′—C1′—C1	−149.30 (10)	C2′—C1′—C1—C6	−72.05 (16)
C4′—O4′—C1′—C2′	−22.87 (13)	O4′—C1′—C1—C2	−131.69 (13)
O4′—C1′—C2′—O2′	163.85 (11)	C2′—C1′—C1—C2	109.27 (15)
C1—C1′—C2′—O2′	−73.93 (15)	C6—C1—C2—F2	178.17 (12)
O4′—C1′—C2′—C3′	39.36 (12)	C1′—C1—C2—F2	−3.0 (2)

C1—C1′—C2′—C3′	161.58 (11)	C6—C1—C2—C3	−2.4 (2)
O2′—C2′—C3′—O3′	−50.08 (15)	C1′—C1—C2—C3	176.40 (14)
C1′—C2′—C3′—O3′	73.24 (12)	F2—C2—C3—C4	178.85 (15)
O2′—C2′—C3′—C4′	−163.61 (11)	C1—C2—C3—C4	−0.6 (2)
C1′—C2′—C3′—C4′	−40.29 (12)	C2—C3—C4—F4	−178.05 (15)
C1′—O4′—C4′—C5′	119.36 (11)	C2—C3—C4—C5	2.1 (3)
C1′—O4′—C4′—C3′	−3.32 (14)	F4—C4—C5—C6	179.75 (15)
O3′—C3′—C4′—O4′	−88.31 (12)	C3—C4—C5—C6	−0.4 (3)
C2′—C3′—C4′—O4′	27.74 (13)	C4—C5—C6—F6	176.13 (14)
O3′—C3′—C4′—C5′	150.61 (11)	C4—C5—C6—C1	−3.0 (2)
C2′—C3′—C4′—C5′	−93.35 (12)	C2—C1—C6—F6	−174.87 (12)
O4′—C4′—C5′—O5′	65.94 (14)	C1′—C1—C6—F6	6.4 (2)
C3′—C4′—C5′—O5′	−175.56 (11)	C2—C1—C6—C5	4.3 (2)
O4′—C1′—C1—C6	46.99 (17)	C1′—C1—C6—C5	−174.46 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2′—H2*B*···O5′^{i}	0.78 (3)	2.05 (3)	2.8157 (17)	168 (2)
O3′—H3*B*···O4′^{i}	0.85 (3)	1.97 (3)	2.8151 (16)	175 (2)
O3′—H3*B*···F6^{i}	0.85 (3)	2.53 (2)	2.8865 (15)	106.1 (19)
O5′—H5*C*···O2′^{ii}	0.81 (3)	1.97 (3)	2.7719 (17)	175 (3)
C3′—H3*A*···F6^{iii}	1.00	2.48	3.4024 (17)	153
C4′—H4*A*···F4^{iv}	1.00	2.62	3.5489 (19)	154
C5′—H5*A*···F4^{v}	0.99	2.53	3.2788 (19)	132
C3—H3*C*···O5′^{vi}	0.95	2.55	3.448 (2)	159

Symmetry codes: (i) $x-1/2, -y+3/2, -z+2$; (ii) $x+1/2, -y+3/2, -z+1$; (iii) $x-1/2, -y+3/2, -z+1$; (iv) $-x+3/2, y-1/2, -z+2$; (v) $-x+3/2, y-1/2, -z+1$; (vi) $-x+3/2, y+1/2, -z+2$.

(III) 1′-(4-Chlorophenyl)-1′-deoxy-*β*-*D*-ribofuranose

Crystal data

$C_{11}H_{13}ClO_4$
$M_r = 244.66$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.7097\ (9)$ Å
$b = 6.8447\ (9)$ Å
$c = 23.948\ (4)$ Å
$V = 1099.9\ (3)$ Å^3
$Z = 4$

$F(000) = 512$
$D_x = 1.478$ Mg m^{-3}
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 167 reflections
$\theta = 3–23°$
$\mu = 0.34$ mm^{-1}
$T = 145$ K
Rod, colourless
0.50 × 0.20 × 0.14 mm

Data collection

Siemens SMART 1K CCD area-detector diffractometer
Radiation source: normal-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: numerical (*SHELXTL*; Sheldrick, 2008)
$T_{min} = 0.872$, $T_{max} = 0.955$

24334 measured reflections
4164 independent reflections
3470 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 33.5°$, $\theta_{min} = 1.7°$
$h = -10\rightarrow10$
$k = -10\rightarrow10$
$l = -35\rightarrow37$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.091$
$S = 1.04$
4164 reflections
157 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 0.36P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.46$ e Å^{-3}
Absolute structure: Flack (1983), with 1707 Friedel pairs
Absolute structure parameter: −0.01 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
Cl4	0.33378 (9)	1.10912 (7)	0.462908 (19)	0.05133 (16)
O2′	−0.06740 (14)	0.54119 (15)	0.67752 (4)	0.0202 (2)
H2B	−0.144 (4)	0.462 (4)	0.6741 (11)	0.065 (8)*
O3′	0.13673 (16)	0.38253 (16)	0.76749 (4)	0.0218 (2)
H3B	0.126 (3)	0.295 (3)	0.7843 (9)	0.035 (6)*
O4′	0.46384 (14)	0.53336 (14)	0.68725 (5)	0.0240 (2)
O5′	0.72931 (14)	0.19941 (15)	0.66821 (5)	0.0230 (2)
H5C	0.760 (3)	0.116 (3)	0.6873 (8)	0.031 (5)*
C1′	0.28076 (18)	0.62872 (18)	0.67316 (6)	0.0176 (2)
H1A	0.2426	0.7172	0.7046	0.021*
C2′	0.12592 (18)	0.46453 (18)	0.66913 (6)	0.0164 (2)
H2A	0.1336	0.4022	0.6314	0.020*
C3′	0.19912 (18)	0.32019 (19)	0.71343 (6)	0.0174 (2)
H3A	0.1523	0.1845	0.7053	0.021*
C4′	0.42579 (18)	0.33727 (18)	0.70708 (6)	0.0170 (2)
H4A	0.4888	0.3213	0.7446	0.020*
C5′	0.51630 (18)	0.1898 (2)	0.66741 (6)	0.0217 (3)
H5A	0.4730	0.0568	0.6782	0.026*
H5B	0.4679	0.2156	0.6291	0.026*
C1	0.3010 (2)	0.7494 (2)	0.62077 (6)	0.0204 (2)
C2	0.1382 (2)	0.8597 (2)	0.60277 (6)	0.0256 (3)
H2C	0.0184	0.8584	0.6239	0.031*
C3	0.1488 (3)	0.9712 (2)	0.55445 (7)	0.0316 (3)
H3C	0.0371	1.0448	0.5421	0.038*

C4	0.3260 (3)	0.9730 (2)	0.52456 (6)	0.0324 (4)
C5	0.4898 (3)	0.8700 (2)	0.54195 (7)	0.0350 (4)
H5D	0.6107	0.8759	0.5214	0.042*
C6	0.4770 (2)	0.7562 (2)	0.59032 (7)	0.0291 (3)
H6A	0.5894	0.6832	0.6024	0.035*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl4	0.0823 (4)	0.0445 (2)	0.02714 (19)	−0.0267 (3)	0.0062 (2)	0.00768 (19)
O2′	0.0119 (4)	0.0181 (4)	0.0307 (5)	−0.0001 (3)	−0.0003 (4)	−0.0017 (4)
O3′	0.0243 (5)	0.0194 (5)	0.0219 (4)	−0.0012 (4)	0.0055 (4)	0.0016 (4)
O4′	0.0125 (4)	0.0151 (4)	0.0445 (6)	−0.0013 (3)	−0.0030 (4)	0.0025 (4)
O5′	0.0137 (4)	0.0193 (5)	0.0359 (6)	0.0011 (3)	0.0003 (4)	0.0007 (4)
C1′	0.0144 (5)	0.0138 (5)	0.0245 (6)	0.0001 (4)	−0.0016 (5)	−0.0013 (5)
C2′	0.0121 (5)	0.0153 (5)	0.0217 (6)	−0.0005 (4)	−0.0003 (4)	−0.0012 (5)
C3′	0.0150 (5)	0.0153 (5)	0.0218 (6)	−0.0013 (4)	0.0014 (5)	−0.0009 (5)
C4′	0.0149 (5)	0.0140 (6)	0.0222 (6)	−0.0007 (4)	−0.0016 (4)	−0.0010 (4)
C5′	0.0149 (5)	0.0190 (6)	0.0311 (7)	−0.0007 (4)	−0.0009 (5)	−0.0060 (5)
C1	0.0217 (6)	0.0156 (5)	0.0238 (6)	−0.0025 (5)	0.0032 (5)	−0.0023 (5)
C2	0.0259 (6)	0.0242 (7)	0.0266 (6)	−0.0004 (5)	0.0041 (6)	0.0039 (5)
C3	0.0389 (8)	0.0266 (7)	0.0295 (7)	−0.0031 (7)	0.0004 (7)	0.0058 (6)
C4	0.0508 (10)	0.0247 (7)	0.0216 (6)	−0.0157 (7)	0.0073 (7)	−0.0009 (6)
C5	0.0405 (9)	0.0303 (8)	0.0341 (8)	−0.0092 (7)	0.0180 (7)	−0.0057 (7)
C6	0.0262 (7)	0.0233 (6)	0.0378 (8)	−0.0029 (6)	0.0105 (6)	−0.0047 (6)

Geometric parameters (Å, º)

Cl4—C4	1.7466 (16)	C3′—H3A	1.0000
O2′—C2′	1.4136 (15)	C4′—C5′	1.5134 (18)
O2′—H2B	0.75 (3)	C4′—H4A	1.0000
O3′—C3′	1.4259 (16)	C5′—H5A	0.9900
O3′—H3B	0.73 (2)	C5′—H5B	0.9900
O4′—C1′	1.4314 (16)	C1—C6	1.389 (2)
O4′—C4′	1.4465 (16)	C1—C2	1.396 (2)
O5′—C5′	1.4309 (15)	C2—C3	1.388 (2)
O5′—H5C	0.76 (2)	C2—H2C	0.9500
C1′—C1	1.5085 (19)	C3—C4	1.388 (2)
C1′—C2′	1.5336 (18)	C3—H3C	0.9500
C1′—H1A	1.0000	C4—C5	1.370 (3)
C2′—C3′	1.5308 (18)	C5—C6	1.399 (2)
C2′—H2A	1.0000	C5—H5D	0.9500
C3′—C4′	1.5329 (17)	C6—H6A	0.9500
C2′—O2′—H2B	110 (2)	C5′—C4′—H4A	108.8
C3′—O3′—H3B	106.5 (17)	C3′—C4′—H4A	108.8
C1′—O4′—C4′	110.43 (9)	O5′—C5′—C4′	111.21 (11)
C5′—O5′—H5C	103.9 (16)	O5′—C5′—H5A	109.4
O4′—C1′—C1	111.63 (11)	C4′—C5′—H5A	109.4
O4′—C1′—C2′	105.19 (10)	O5′—C5′—H5B	109.4

C1—C1′—C2′	114.20 (11)	C4′—C5′—H5B	109.4
O4′—C1′—H1A	108.5	H5A—C5′—H5B	108.0
C1—C1′—H1A	108.5	C6—C1—C2	119.02 (14)
C2′—C1′—H1A	108.5	C6—C1—C1′	122.12 (13)
O2′—C2′—C3′	115.81 (11)	C2—C1—C1′	118.86 (12)
O2′—C2′—C1′	109.91 (10)	C3—C2—C1	120.99 (14)
C3′—C2′—C1′	102.23 (10)	C3—C2—H2C	119.5
O2′—C2′—H2A	109.5	C1—C2—H2C	119.5
C3′—C2′—H2A	109.5	C2—C3—C4	118.57 (16)
C1′—C2′—H2A	109.5	C2—C3—H3C	120.7
O3′—C3′—C2′	110.00 (11)	C4—C3—H3C	120.7
O3′—C3′—C4′	111.01 (11)	C5—C4—C3	121.73 (15)
C2′—C3′—C4′	101.56 (10)	C5—C4—Cl4	120.49 (13)
O3′—C3′—H3A	111.3	C3—C4—Cl4	117.77 (14)
C2′—C3′—H3A	111.3	C4—C5—C6	119.27 (15)
C4′—C3′—H3A	111.3	C4—C5—H5D	120.4
O4′—C4′—C5′	109.99 (11)	C6—C5—H5D	120.4
O4′—C4′—C3′	106.17 (10)	C1—C6—C5	120.39 (15)
C5′—C4′—C3′	114.17 (11)	C1—C6—H6A	119.8
O4′—C4′—H4A	108.8	C5—C6—H6A	119.8

C4′—O4′—C1′—C1	−140.13 (11)	O4′—C4′—C5′—O5′	67.30 (14)
C4′—O4′—C1′—C2′	−15.76 (14)	C3′—C4′—C5′—O5′	−173.49 (12)
O4′—C1′—C2′—O2′	156.80 (11)	O4′—C1′—C1—C6	2.25 (18)
C1—C1′—C2′—O2′	−80.47 (14)	C2′—C1′—C1—C6	−116.91 (15)
O4′—C1′—C2′—C3′	33.27 (13)	O4′—C1′—C1—C2	−176.90 (12)
C1—C1′—C2′—C3′	156.00 (11)	C2′—C1′—C1—C2	63.94 (16)
O2′—C2′—C3′—O3′	−38.88 (14)	C6—C1—C2—C3	1.6 (2)
C1′—C2′—C3′—O3′	80.59 (12)	C1′—C1—C2—C3	−179.21 (14)
O2′—C2′—C3′—C4′	−156.52 (11)	C1—C2—C3—C4	−0.8 (2)
C1′—C2′—C3′—C4′	−37.04 (12)	C2—C3—C4—C5	−0.8 (2)
C1′—O4′—C4′—C5′	115.65 (12)	C2—C3—C4—Cl4	178.33 (12)
C1′—O4′—C4′—C3′	−8.35 (14)	C3—C4—C5—C6	1.6 (2)
O3′—C3′—C4′—O4′	−88.24 (13)	Cl4—C4—C5—C6	−177.54 (13)
C2′—C3′—C4′—O4′	28.66 (13)	C2—C1—C6—C5	−0.8 (2)
O3′—C3′—C4′—C5′	150.41 (11)	C1′—C1—C6—C5	−179.97 (14)
C2′—C3′—C4′—C5′	−92.69 (13)	C4—C5—C6—C1	−0.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2′—H2*B*···O5′^{i}	0.75 (3)	1.99 (3)	2.7172 (15)	162 (3)
O3′—H3*B*···O2′^{ii}	0.73 (2)	2.00 (2)	2.7220 (15)	172 (2)
O5′—H5*C*···O3′^{iii}	0.76 (2)	2.05 (2)	2.8077 (15)	174 (2)
C4′—H4*A*···O4′^{iii}	1.00	2.58	3.3584 (17)	135
C3—H3*C*···Cl4^{iv}	0.95	2.74	3.5902 (19)	150

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) −*x*, *y*−1/2, −*z*+3/2; (iii) −*x*+1, *y*−1/2, −*z*+3/2; (iv) *x*−1/2, −*y*+5/2, −*z*+1.

Ring-puckering parameters q (Å) and φ (°), as defined by Cremer & Pople (1975), and selected torsion angles (°) in (I)–(III) and in a number of related compounds (numbering scheme as in Figs. 1, 3 and 5)

	q	*φ*	O4′—C1′—C1—C6	H—C1′—C1—C2
(I)	0.3764 (16)	80.0 (2)	29.3 (2)	-32.8
(II)	0.4162 (14)	77.3 (2)	47.0 (2)	-13.3
(III)	0.3744 (14)	84.8 (2)	2.3 (2)	-57.3
(*a*), polymorph 1	0.3752	60.4	50.4	-4.1
(*a*), polymorph 2	0.3685	81.6	8.8	-50.0
(*a*), hemihydrate	0.4214	63.4	45.0	-15.9
(*b*)	0.3900	274.7	6.8	-57.0
(*c*)	0.3767	86.7	4.6	-53.5
(*d*)	0.3850	74.8	27.4	-35.4
(*e*)	0.3740	84.3	7.4	-52.0

Notes: (*a*) 1′-deoxy-1′-(4-fluorophenyl)-*β-D*-ribofuranose (Bats *et al.*, 2000); (*b*) 1′-deoxy-1′-(2,4-difluorophenyl)-*β-D*-ribofuranose (Bats *et al.*, 2000); (*c*) 1′-deoxy-1′-(3-fluorophenyl)-*β-D*-ribofuranose (Bats *et al.*, 1999*b*); (*d*) 1′-deoxy-1′-(2-fluorophenyl)-*β-D*-ribofuranose (Bats *et al.*, 1999*a*); (*e*) 1′-deoxy-1′-phenyl-*β-D*-ribofuranose (Štambaský *et al.*, 2011).