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Intermolecular C—F···H—C contacts in the molecular packing of three isostructural *N*-(fluorophenyl)manno-pyranosylamines

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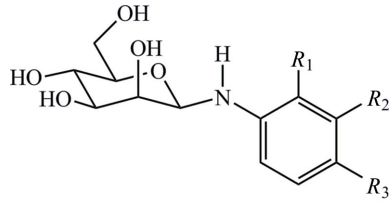
Among the three compounds reported here, namely *N*-(4-fluorophenyl)-β-D-mannopyranosylamine, (I), *N*-(3-fluorophenyl)-β-D-mannopyranosylamine, (II), and *N*-(2-fluorophenyl)-β-D-mannopyranosylamine, (III), all with chemical formula $C_{12}H_{16}FNO_5$, (I) and (II) are isostructural, whereas (III) assumes the same packing arrangement as the unfluorinated analogue *N*-phenyl-β-D-mannopyranosylamine, (IV), which has been reported previously. Similarities with respect to the intermolecular hydrogen-bonding patterns exist across the series (I)–(III). A packing motif that distinguishes the shared packing arrangement of (I) and (II) from that of (III) is a C—F···H—C chain of graph set *C*(4) that is preserved in the formal exchange of F and H atoms at the 4- and 3-positions on the aromatic ring of (I) and (II), but is replaced by a different chain of graph set *C*(5) when the F atom is located at the 2-position of the aromatic ring in (III). The steric role of the F atom in (I)–(III) is ambiguous but is examined here in detail.

Comment

The role played by covalently bonded fluorine in the solid-state packing of organic molecules is a topic of continuing active interest and is of direct relevance to the field of crystal engineering (Chopra & Guru Row, 2008; Zhu *et al.*, 2007; Reichenbächer *et al.*, 2005; Choudhury & Guru Row, 2004; Choudhury *et al.*, 2004, Brammer *et al.*, 2001), although whether solid-state interactions involving fluorine can actually be useful in the design and preparation of desired molecular packing motifs has been questioned in the literature. Previous studies have shown that the F atom of the C—F moiety is an exceptionally weak hydrogen-bond acceptor and that such interactions are significant only in particular cases, such as in those crystal structures from which stronger hydrogen-bonding groups are absent (Dunitz, 2004; Thalladi *et al.*, 1998; Dunitz & Taylor, 1997; Howard *et al.*, 1996). Solid-state halogen–halogen contacts between F atoms have been reported to be a consequence of molecular packing, rather than of attractive interactions that help determine that packing (Desiraju & Parthasarathy, 1989). Nevertheless, interactions of covalently bonded F atoms with neighboring H atoms, as well as with nearby π systems and with other halogen atoms, continue to be cited in the literature as factors that influence the packing of a variety of fluorine-bearing molecules, even in the presence of certain relatively strong hydrogen-bonding donors and acceptors (In *et al.*, 2003; Vangala *et al.*, 2002; Prasanna & Guru Row, 2000; Nangia, 2000).

Given this rather contradictory situation and the subtle nature of solid-state interactions involving C—F, we have been particularly interested in fluorine-substituted monosaccharide derivatives, which we have prepared and examined as part of our continuing study of the structures of the compounds formed upon reaction of monosaccharides with nitrogenous bases. We describe here the molecular and crystal structures of three fluorine-substituted glycosylamine derivatives of D-mannose, namely *N*-(4-fluorophenyl)-β-D-mannopyranosylamine, (I), *N*-(3-fluorophenyl)-β-D-mannopyranosylamine, (II), and *N*-(2-fluorophenyl)-β-D-mannopyranosylamine, (III).



(I) $R_1 = R_2 = H$, $R_3 = F$
(II) $R_1 = R_3 = H$, $R_2 = F$
(III) $R_2 = R_3 = H$, $R_1 = F$
(IV) $R_1 = R_2 = R_3 = H$

In general, the reaction of a monosaccharide with a nitrogenous base can yield as the crystalline product an open-chain Schiff base, as well as (or instead of) a cyclic glycosylamine. For example, we found in a previous study that D-mannose reacts with hydroxylamine to yield an open-chain oxime as the crystalline product (Ojala *et al.*, 2000). On the other hand, in related work we obtained glycosylamines, including the phenyl-substituted derivative, *N*-phenyl-β-D-mannopyranosylamine, (IV), rather than Schiff bases, from the reaction of D-mannose with aniline and its derivatives (Ojala *et al.*, 2000; Ojala, Ostman, Hanson & Ojala, 2001; Ojala, Ostman, Ojala & Hanson, 2001). We have now obtained the fluoro-substituted glycosylamines (I)–(III) by reaction of D-mannose with the corresponding fluoroanilines. In our previous studies, we found isostructuralism among certain glycosylamines, indicating that the solid-state hydrogen-bonding networks linking the monosaccharide moieties are sufficiently strong and extensive that even fairly drastic changes in the size, shape and even position of substitutents on the aryl ring may have little effect on them. For example, the *N*-4-bromophenyl, *N*-4-chlorophenyl, *N*-4-methylphenyl and *N*-3-chlorophenyl glycosylamines (Ojala *et al.*, 2000; Ojala, Ostman, Hanson &

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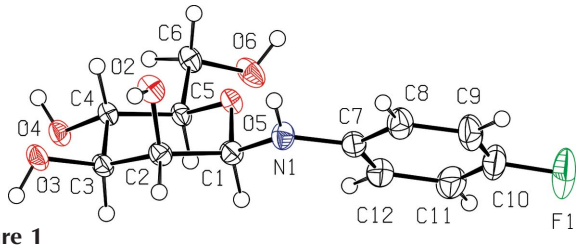

Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellispoids are drawn at the 50% probability level.

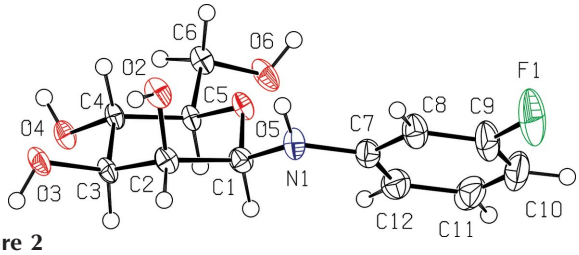

Figure 2
The molecular structure of (II), showing the atom-numbering scheme. Displacement ellispoids are drawn at the 50% probability level.

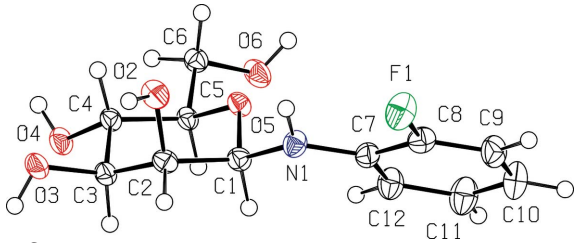

Figure 3
The molecular structure of (III), showing the atom-numbering scheme. Displacement ellispoids are drawn at the 50% probability level.

Ojala, 2001) formed upon reaction between D-mannose and 4-bromoaniline, 4-chloroaniline, 4-methylaniline and 3-chloroaniline, respectively, assume the same molecular packing arrangement. We thus consider it noteworthy that the crystal structures of (I), (II) and (III) are not all identical, given the rigidity expected of their hydrogen-bonding networks. If only the general size similarity between the F atom and the H atom were relevant to the packing, (I), (II) and (III) might all have been found to be isostructural with unfluorinated (IV). Although (I) and (II) are in fact isostructural with each other, they are not isostructural with (IV); only (III) is.

At the molecular level, little difference exists among (I), (II) and (III), except for the position of the F atom. The conformations of the three isomers are closely similar, as shown in Figs. 1–3 and by the corresponding torsion angles in Tables 1, 3 and 5. Similarities exist at the level of the molecular packing as well; details of the intermolecular hydrogen-bonding contacts are given in Tables 2, 4 and 6. A view of the hydrogen-bonding array in the molecular packing of (I) is shown in Fig. 4; the hydrogen bonding in (II) and (III) with respect to the monosaccharide moieties is similar. In all three structures, neighboring molecules are connected by an $R_2^2(10)$ hydrogen-bonding motif (Etter, 1990) between molecules related by translation along [100]. Neighboring molecules are also connected by an $R_2^2(11)$ interaction (in which the N—H

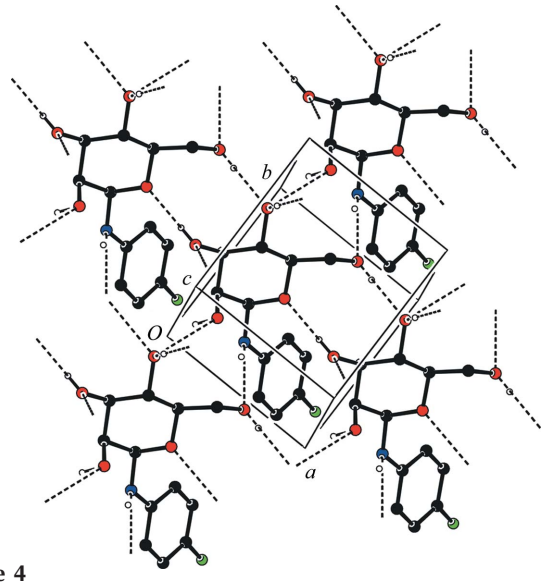

Figure 4
A view of part of the molecular packing arrangement in (I), showing the $R_2^2(10)$ and $R_2^2(11)$ hydrogen-bonding contacts. For clarity, only those H atoms involved in the hydrogen-bonding scheme are shown. At the centre of the figure is the molecule at (x, y, z); surrounding it are molecules at $(x, 1 + y, z)$ at top right, $(1 + x, y, z)$ at bottom right, $(x, -1 + y, z)$ at bottom left and $(-1 + x, y, z)$ at top left.

group participates as a hydrogen-bond donor) between molecules related by translation along [010]. Each molecule also participates, with two additional neighboring molecules, both related to the first by twofold screw-axial symmetry, in a 12-membered hydrogen-bonded ring interaction that builds up the structure along [001] and defines hydrophilic hydrogen-bonded regions extending parallel to (001) in all three structures (Figs. 5 and 6). With similar hydrogen-bonding contacts linking the monosaccharide moieties in all three structures, the differentiating factor that sets the overall crystal structure of (III) apart from that assumed by both (I) and (II) must lie elsewhere than in the conventional hydrogen-bonding system.

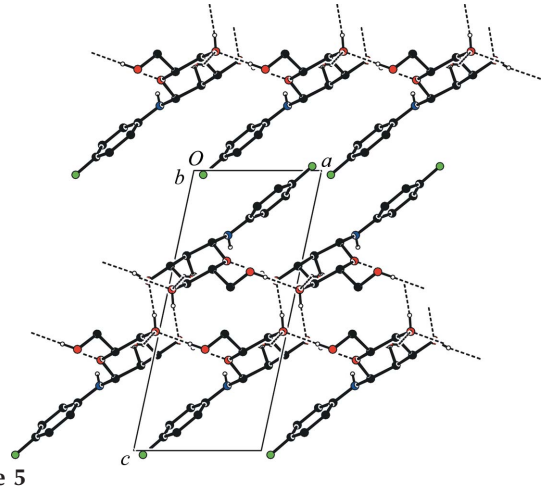

Figure 5
A view of the molecular packing arrangement in (I) along the *b* axis; the packing in (II) is identical. Hydrogen-bonded layers defined by the monosaccharide moieties alternate with non-hydrogen-bonded regions defined by the aryl groups along [001]. For clarity, only those H atoms involved in the hydrogen-bonding scheme are shown.

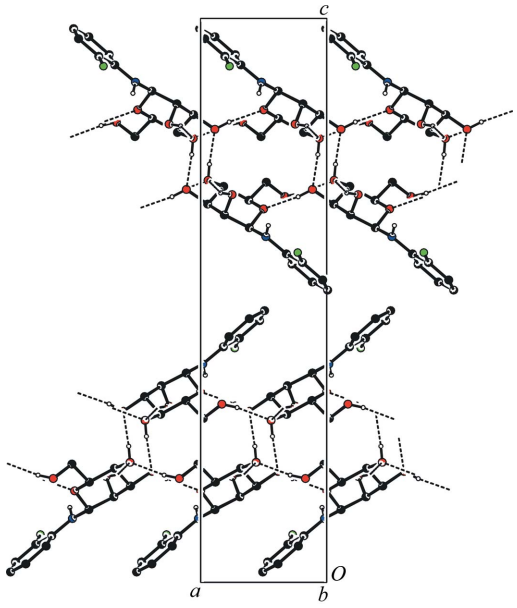

Figure 6
A view of the molecular packing arrangement in (III) along the *b* axis. As in (I) and (II), hydrogen-bonded layers defined by the monosaccharide moieties alternate with non-hydrogen-bonded regions defined by the aryl groups along [001]. For clarity, only those H atoms involved in the hydrogen-bonding scheme are shown.

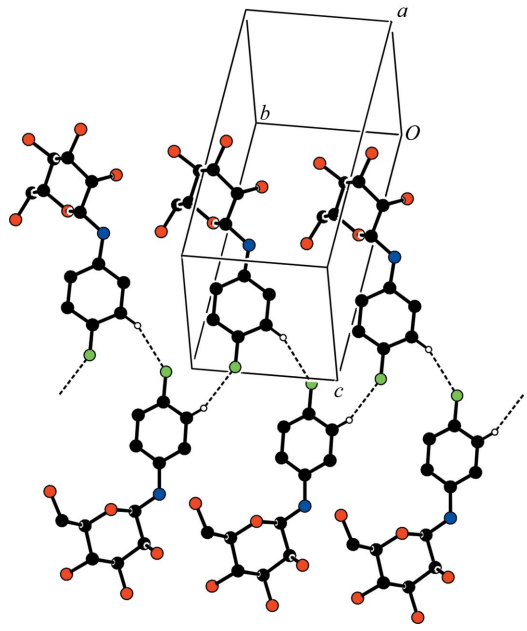

Figure 7
A view of the C—F···H—C *C*(4) chain motif in (I). Dashed lines represent approaches equal to or shorter than the van der Waals contact distance. Only the H atom participating in the contact with the F atom is shown. Exchanging the positions of the F and H atoms on each molecule gives the corresponding *C*(4) motif in (II).

That differentiating factor appears to be in the packing motifs involving C—F···H—C interactions. In all three structures, these interactions occur in the hydrophobic regions defined by the aryl groups, which are regions that lie between the hydrophilic regions defined by the hydrogen-bonded monosaccharide moieties (Figs. 5 and 6). In terms of graph-set notation, these C—F···H—C interactions define a *C*(4) chain motif in both (I) and (II), but a *C*(5) chain motif in (III). A view of the *C*(4) motif in (I) is shown in Fig. 7; exchanging the F and H atoms in the contact shown in Fig. 7 produces the corresponding *C*(4) chain motif in (II). In (I), the motif is defined by the C10—F1···H9—C9 interaction and in (II) the motif is defined by the C9—F1···H10—C10 interaction (see Table 7 for distances and angles). The only significant difference between (I) and (II) is the exchange of the F and H atoms at the 3- and 4-positions, an exchange that leaves the *C*(4) chain motif intact and does not yield a new crystal structure. When the F atom is located at the 2-position, this motif is replaced by the *C*(5) motif defined by the C8—F1···H12—C12 interaction (Fig. 8; see Table 7 for distances and angles) and (III) assumes a different packing arrangement, the higher-symmetry $P2_12_12_1$ structure, rather than the lower-symmetry $P2_1$ structure assumed by (I) and (II). Although a C—F···H—C contact is maintained in (III), the fact that unfluorinated (IV) assumes the same packing arrangement as (III) makes it unlikely (or at least unnecessary) that the F atom in (III) is doing anything special in terms of hydrogen bonding; the structural similarity between (III) and (IV) is consistent with the steric similarity between F and H atoms.

Behaviour parallel to that of the series (I)–(III) is shown by the 4-chlorophenyl-, 3-chlorophenyl- and 2-chlorophenyl-mannopyranosylamine analogues reported previously (Ojala *et al.*, 2000; Ojala, Ostman, Hanson & Ojala 2001) and listed in the Cambridge Structural Database (Allen, 2002) as QEDXEC, YIJJOQ and YIJDUQ, respectively. QEDXEC and YIJJOQ are isostructural with each other and with (I) and (II), and exhibit a chain motif defined by a C—Cl···H—C interaction, analogous to the C—F···H—C chain motif exhibited by (I) and (II). YIJDUQ, in contrast, assumes a

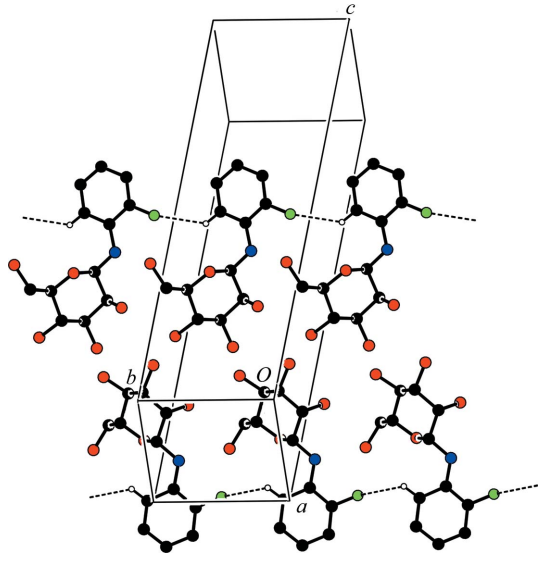

Figure 8
A view of the C—F···H—C *C*(5) chain motif in (III). Dashed lines represent approaches equal to or shorter than the van der Waals contact distance. Only the H atom participating in the contact with the F atom is shown.

packing arrangement different from those assumed by (I)–(III). In the 3- and 4-chlorinated structures, the halogen-for-hydrogen exchange between the 3- and 4-positions on the aromatic ring involves a more drastic change in space-filling requirements than in the corresponding fluorinated structures. Weak hydrogen-bonding character in the C—Cl···H—C interactions might help to stabilize this motif in the face of that change. With (I) and (II) assuming the same packing arrangement as their chlorinated analogues, some attractive character in the C—F···H—C interactions in (I) and (II) may be present as well. In both the fluorinated and chlorinated series, the *C*(4) chain motif is disrupted when the halogen atom is located at the 2-position on the aromatic ring. At this position, fluorine thus behaves more like an H atom than a halogen atom. When located at the 3- or 4-positions on the aromatic ring, fluorine engages in the C—F···H—C motif analogous to the C—Cl···H—C motif and behaves more like a halogen atom than an H atom.

Previous structural studies in which all three compounds in a 2/3/4-fluorophenyl series have been published together have yielded mixed results in terms of whether changing the F-atom position changes the packing arrangement (at least to the degree of changing the space-group symmetry). In some cases, all three compounds are isostructural (Donnelly *et al.*, 2008), in others, all three compounds assume unique packing arrangements, which appears to us to be the more common situation (Langley *et al.*, 1996; Klösener *et al.*, 2008; Wardell *et al.*, 2007; Chisholm *et al.*, 2002; Taira *et al.*, 1988; Larsen & Marthi, 1994). Our present series of fluorophenyl mannoglycosylamines represents the unusual situation in which a packing arrangement in the series is shared by more than one fluorine positional isomer. This situation may represent a compromise between a tendency toward isostructuralism arising from the rigidity of the hydrogen-bond network and a tendency toward non-isostructuralism arising from the disruption of the less rigid non-hydrogen-bonded part of the structure caused by the repositioning of the F atom from isomer to isomer. The loss of isostructuralism with the alteration of the C—F···H—C chain motif in (I)–(III) may be evidence for the structural significance of these contacts in this and other particular cases in which strong hydrogen-bond acceptors and donors are present but interact strongly only among themselves, leaving open the possibility that secondary interactions, even those as exceedingly weak as C—F···H—C interactions, might influence the overall packing arrangement, with the F atom acting as more than a simple size-and-shape mimic for the H atom.

Experimental

Compounds (I)–(III) were prepared by combining D-mannose (0.5 g) with equimolar amounts of 4-fluoroaniline, 3-fluoroaniline and 2-fluoroaniline, respectively, in ethanol, heating the solution to boiling for approximately 10 min and then allowing the solutions to cool and the products to crystallize. The synthesis of a series of *N*-arylglycosylamines from fluorinated anilines by a similar method has been reported elsewhere (Qian *et al.*, 2001), but none of the compounds we describe here was included in that study. All three compounds were obtained as colourless plates; m.p. 449–456 K for (I), 463–465 K for (II) and 466–474 K for (III). The synthesis and crystal structure of (IV) have been described previously (Ojala *et al.*, 2000; Metlitskikh *et al.*, 2005).

Compound (I)

Crystal data

$C_{12}H_{16}FNO_5$
M_r = 273.26
Monoclinic, $P2_1$
a = 6.4612 (12) Å
b = 6.7246 (13) Å
c = 14.501 (3) Å
β = 102.151 (3)°
V = 615.9 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 173 K
0.52 × 0.50 × 0.05 mm

Data collection

Siemens SMART Platform CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.937, T_{max} = 0.993
7296 measured reflections
1528 independent reflections
1393 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.075
S = 1.06
1528 reflections
181 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Compound (II)

Crystal data

$C_{12}H_{16}FNO_5$
M_r = 273.26
Monoclinic, $P2_1$
a = 6.4579 (8) Å
b = 6.7248 (9) Å
c = 14.5052 (19) Å
β = 102.364 (2)°
V = 615.32 (14) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 173 K
0.50 × 0.48 × 0.05 mm

Data collection

Bruker SMART Platform CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.941, T_{max} = 0.995
4666 measured reflections
1460 independent reflections
1261 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
S = 1.07
1460 reflections
180 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Compound (III)

Crystal data

$C_{12}H_{16}FNO_5$
M_r = 273.26
Orthorhombic, $P2_12_12_1$
a = 6.4133 (6) Å
b = 6.6924 (6) Å
c = 28.699 (3) Å
V = 1231.76 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 173 K
0.48 × 0.20 × 0.05 mm

Table 1
Selected torsion angles (°) for (I).

O5—C5—C6—O6	74.23 (19)	O5—C1—N1—C7	−73.0 (2)
C4—C5—C6—O6	−164.77 (15)	C2—C1—N1—C7	167.00 (17)
C8—C7—N1—C1	177.94 (18)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O6^{i}	0.84 (3)	2.26 (3)	3.063 (3)	160 (2)
O2—H2O···O4^{i}	0.84	2.13	2.908 (2)	154
O3—H3O···O5^{ii}	0.84	1.95	2.7800 (18)	171
O4—H4O···O3^{iii}	0.84	1.87	2.700 (2)	171
O6—H6O···O4^{iv}	0.84	1.91	2.7504 (19)	178

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

Table 3
Selected torsion angles (°) for (II).

O5—C5—C6—O6	74.2 (2)	O5—C1—N1—C7	−72.4 (3)
C4—C5—C6—O6	−164.46 (19)	C2—C1—N1—C7	167.4 (2)
C8—C7—N1—C1	176.4 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6O···O4^{i}	0.84	1.91	2.752 (2)	177
O4—H4O···O3^{ii}	0.84	1.86	2.696 (3)	172
O3—H3O···O5^{iii}	0.84	1.95	2.780 (2)	171
O2—H2O···O4^{iv}	0.84	2.14	2.911 (3)	152
N1—H1N···O6^{iv}	0.81 (4)	2.32 (5)	3.066 (3)	153 (4)

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

Data collection

Siemens SMART Platform CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.970, T_{max} = 0.993
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R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.072
S = 1.04
1672 reflections
181 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Given the absence from all three structures of significant anomalous scattering effects, equivalent reflections, including Friedel pairs, were merged. H atoms located on C atoms were placed in calculated positions and refined using a riding model, with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C) for aryl H atoms, C—H = 1.00 Å and U_{iso}(H) = 1.2U_{eq}(C) for methine H atoms, and C—H = 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene H atoms. H atoms located on O atoms were refined isotropically using a rotating-group model, with O—H distances constrained to 0.84 Å. H atoms located on N atoms were refined isotropically without constraints. Absolute configurations were assigned on the basis of the synthesis of each compound from D-mannose.

Table 5
Selected torsion angles (°) for (III).

O5—C5—C6—O6	77.93 (17)	O5—C1—N1—C7	−73.0 (2)
C4—C5—C6—O6	−161.32 (14)	C2—C1—N1—C7	165.61 (15)
C8—C7—N1—C1	−179.87 (17)		

Table 6
Hydrogen-bond geometry (Å, °) for (III).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O6^{i}	0.86 (2)	2.25 (2)	3.097 (2)	167 (2)
O2—H2O···O4^{i}	0.84	2.19	2.9584 (19)	152
O3—H3O···O5^{ii}	0.84	1.96	2.7914 (17)	172
O4—H4O···O3^{iii}	0.84	1.88	2.7139 (19)	174
O6—H6O···O4^{iv}	0.84	1.94	2.7774 (19)	172

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

Table 7
Fluorine contact geometries (Å, °) in (I)–(III).

	C—F···H—C	F···H	H···C	C—F···H	F···H—C
(I)	C10—F1···H9—C9^{i}	2.44	0.95	143	158
(II)	C9—F1···H10—C10^{ii}	2.46	0.95	145	154
(III)	C8—F1···H12—C12^{iii}	2.55	0.95	150	127

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

For all compounds, data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GZ3186). Services for accessing these data are described at the back of the journal.

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