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Carbohydrate Research 331 (2001) 319–325

CARBOHYDRATE RESEARCH

# Molecular and crystal structures of *N*-arylglycopyranosylamines formed by reaction between sulfanilamide and D-ribose, D-arabinose and D-mannose

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Received 21 August 2000; accepted 5 December 2000

**Abstract**

The X-ray crystal structures of three monosaccharide derivatives prepared by the reaction of sulfanilamide with D-ribose, D-arabinose, and D-mannose have been determined. The derivatives are *N*-(*p*-sulfamoylphenyl)-α-D-ribopyranosylamine (**1**), *N*-(*p*-sulfamoylphenyl)-α-D-arabinopyranosylamine (**2**), and *N*-(*p*-sulfamoylphenyl)-β-D-mannopyranosylamine monohydrate (**3**). The monosaccharide ring of **1** and **2** has the $^1C_4$ conformation, stabilized in **1** by an intramolecular hydrogen bond from O-2 to O-4. Compound **3** has the $^4C_1$ conformation at the monosaccharide ring and the *gt* conformation at the C-6–O-6 side chain. Occupancy of the water molecule in the crystal of **3** actually examined was 22%. The degree of interaction between sulfamoyl groups and monosaccharide moieties varies from structure to structure. The packing arrangement of **2** involves hydrogen bonding between sulfamoyl groups and monosaccharide hydroxyl groups, but interactions of this type are fewer in **1**, and in **3** the hydrogen bonds are either strictly between monosaccharide hydroxyl groups or strictly between sulfamoyl groups. Pairs of hydrogen bonds (two-point contacts) link neighboring molecules in all three structures, between screw-axially related molecules in **1** and **2** and between translationally related molecules in **3**. The contact in **3** defined by the O-3–H···O-5 and O-6–H···O-4 hydrogen bonds is found in several other *N*-aryl-β-D-mannopyranosylamine crystal structures and is apparently an especially favorable mode of intermolecular interaction in these compounds. 

*Keywords:* X-ray crystal structure; Glycosylamines; Hydrogen bonding; Monosaccharides, structure; Sulfonamides; Sulfanilamide derivatives

## 1. Introduction

We are conducting a systematic X-ray crystallographic study of the derivatives formed upon reaction of monosaccharides with nitrogenous bases. Depending on the monosaccharide, the base, and the reaction conditions, the resulting derivative could assume either acyclic (Schiff base) form or cyclic (glycosylamine) form in the solid state. For example, the derivatives formed when D-mannose reacts with hydroxylamine, with phenylhydrazine, or with *p*-bromophenylhydrazine crystallize as open-chain compounds, but the derivatives formed on reaction with semicarbazide, with aniline, or with *p*-chloroaniline crystallize in cyclic form[1–3] (Scheme 1). Which form monosaccharide anilides such as the latter two compounds should be expected to assume in

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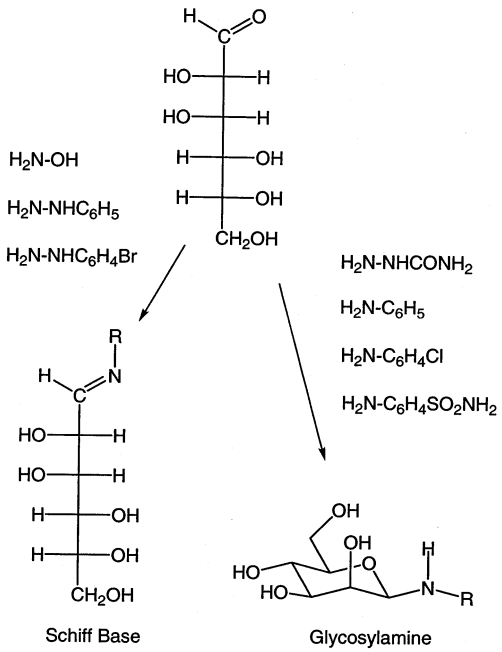
0008-6215/01/$ - see front matter 
PII: S0008-6215(01)00011-8

the solid state was the subject of some disagreement in the earlier literature,[4–7] but spectroscopic and X-ray studies have consistently supported the glycosylamine structure.[8–11]




Scheme 1. When D-mannose reacts with hydroxylamine, phenylhydrazine, or *p*-bromophenylhydrazine, the crystalline product is a Schiff base. When D-mannose reacts with semicarbazide, aniline, *p*-chloroaniline, or sulfanilamide, the crystalline product is a glycosylamine.

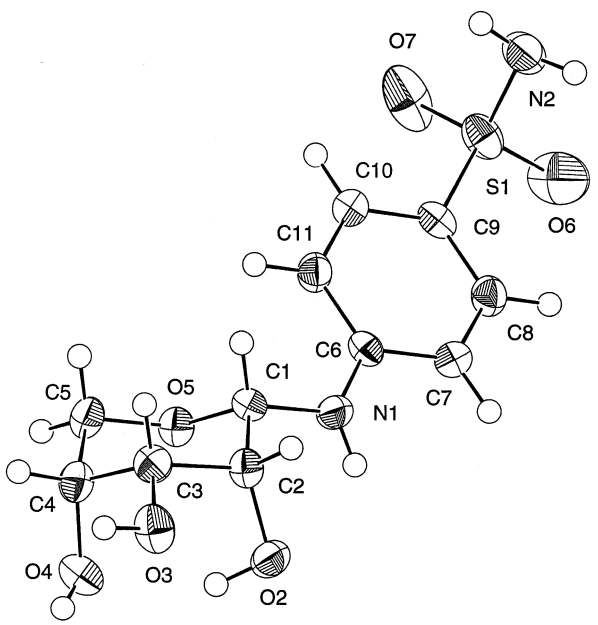



Fig. 1. ORTEPII[15] drawing of **1**, showing atom numbering. The ellipsoids are drawn at the 50% probability level.

More recently, the xylopyranosylamines in particular have been of interest as potential antitumor agents, their activity thought to arise from their ability to artificially initiate and modify the biosynthesis of glycosaminoglycans (GAGs) in melanoma cells.[12]

As part of our own structural study of monosaccharide anilides, we have begun to examine the compounds formed upon reaction of monosaccharides with those sulfonamides that are of biomedical significance as 'sulfa drugs.' Previous studies have indicated that glycosylation of these sulfonamides does not significantly enhance their effectiveness as drugs, but it does increase their solubility in the bloodstream and also renders them less toxic than the parent sulfonamides themselves. Also of interest is the possibility that one or more of several isomers might be isolated in solid form, the derivative potentially crystallizing as a furanosylamine or as a pyranosylamine if indeed the cyclic structure is the one assumed.[13,14] In this report we describe the molecular and crystal structures of the derivatives formed upon reaction of D-ribose, D-arabinose, and D-mannose with sulfanilamide. We have found all three derivatives to be *N*-arylglycopyranosylamines. The mannose derivative crystallizes as a monohydrate, as reported previously.[13]

## 2. Results and discussion

*Molecular geometry.*—ORTEPII[15] drawings showing the atom numbering schemes and molecular conformations of *N*-(*p*-sulfamoylphenyl)ribopyranosylamine (**1**), *N*-(*p*-sulfamoylphenyl)arabinopyranosylamine (**2**), and *N*-(*p*-sulfamoylphenyl)mannopyranosylamine (**3**) are presented as Figs. 1–3. Parameters related to the molecular conformations are listed in Table 1 (selected torsional angles) and Table 2 (Cremer–Pople puckering parameters[16–18] and ring asymmetry parameters[19] obtained using the program PLATON 94).[20]

The conformation of the pyranose ring of **1** and **2** is $^1C_4$; in **1** this conformation is stabilized by an intramolecular hydrogen bond, O-2–H···O-4 (see Table 3 for parameters for

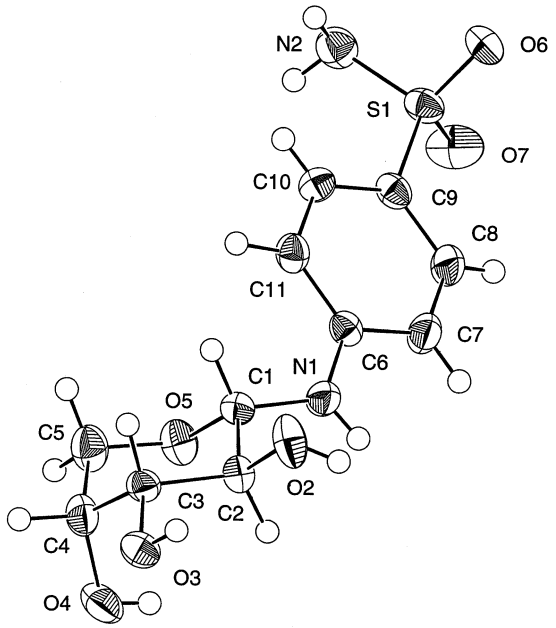



Fig. 2. ORTEPII[15] drawing of **2**, showing atom numbering. The ellipsoids are drawn at the 50% probability level.

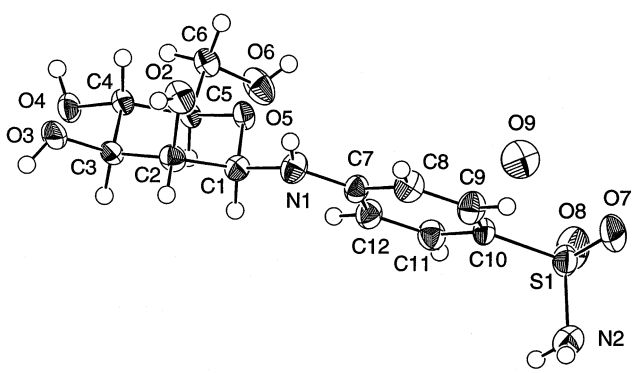



Fig. 3. ORTEPII[15] drawing of **3**, showing atom numbering. The ellipsoids are drawn at the 50% probability level.

this and other hydrogen bonds in **1**–**3**). In **3** the conformation of the monosaccharide ring is $^4C_1$. In **1** and **2** the configuration at C-1 is α; it is β in **3**. The conformation of the molecule about the C-1–N-1 bond, as defined by the C-2–C-1–N-1–C(aryl) and O-5–C-1–N-1–C(aryl) torsional angles, is essentially the same in all three structures, with the N-1–C(aryl) bond oriented *anti* with respect to the C-1–C-2 bond and *gauche* with respect to the C-1–O-5 bond. We have observed this same orientation about the C-1–N-1 bond in fourteen previous *N*-aryl-β-D-glycosylamine structures.[1] On the other hand, compounds **1**–**3** differ in conformation at the N-1–C(aryl) bond. In **1** and **3** the relevant linkage (C-1–N-1–C-6–C-7 in **1**, C-1–N-1–C-7–C-8 in **3**) is nearly planar, but in **2** this linkage (C-1–N-1–C-6–C-7) is twisted out of planarity about the N-1–C-6 bond by almost fourteen degrees. Compound **2** also differs from **1** and **3** in the orientation of the sulfamoyl group. In viewing both **1** and **3** down the S-1–C(aryl) bond, the S–N bond is oriented roughly perpendicular

Table 1
Torsional angles (°)

| | 1 | 2 | 3 |
|---|---|---|---|
| O-2–C-2–C-1–O-5 | 68.5(3) | −175.3(2) | −65.6(3) |
| O-2–C-2–C-1–N-1 | −52.5(3) | 65.4(3) | 55.3(3) |
| O-2–C-2–C-3–O-3 | 53.9(3) | −62.1(3) | −54.6(3) |
| O-2–C-2–C-3–C-4 | −69.6(3) | 176.3(3) | 67.6(3) |
| O-3–C-3–C-2–C-1 | 176.8(2) | 177.4(2) | −172.7(2) |
| O-3–C-3–C-4–O-4 | −53.2(3) | −55.2(3) | −67.9(3) |
| O-3–C-3–C-4–C-5 | −172.8(2) | −178.0(3) | 172.0(2) |
| O-4–C-4–C-3–C-2 | 66.5(3) | 67.5(3) | 170.7(2) |
| O-4–C-4–C-5–O-5 | −64.1(3) | −64.8(4) | −175.0(2) |
| O-4–C-4–C-5–C-6 | | | 65.4(3) |
| O-5–C-1–N-1–C-6 | 74.2(3) | 76.8(4) | |
| O-5–C-1–N-1–C-7 | | | −71.9(3) |
| O-5–C-1–C-2–C-3 | −56.0(3) | −57.5(3) | 54.3(3) |
| O-5–C-5–C-4–C-3 | 56.7(3) | 58.8(4) | −55.2(3) |
| O-5–C-5–C-6–O-6 | | | 74.4(3) |
| O-6–S-1–C-9–C-8 | 19.4(3) | −85.6(3) | |
| O-6–S-1–C-9–C-10 | −162.3(2) | 93.9(3) | |
| O-6–C-6–C-5–C-4 | | | −164.3(2) |
| O-7–S-1–C-9–C-8 | 149.1(2) | 43.0(3) | |
| O-7–S-1–C-9–C-10 | −32.7(3) | −137.5(3) | |
| O-7–S-1–C-10–C-9 | | | −20.4(3) |
| O-7–S-1–C-10–C-11 | | | 162.2(2) |
| O-8–S-1–C-10–C-9 | | | −150.4(3) |
| O-8–S-1–C-10–C-11 | | | 32.2(3) |
| N-1–C-1–O-5–C-5 | −178.3(2) | −176.0(3) | 176.1(2) |
| N-1–C-1–C-2–C-3 | −177.0(2) | −176.8(2) | 175.2(2) |
| N-2–S-1–C-9–C-8 | −96.8(2) | 159.2(3) | |
| N-2–S-1–C-9–C-10 | 81.5(2) | −21.3(3) | |
| N-2–S-1–C-10–C-9 | | | 95.0(3) |
| N-2–S-1–C-10–C-11 | | | −82.4(3) |
| C-1–O-5–C-5–C-4 | −61.2(3) | −64.6(4) | 62.1(3) |
| C-1–O-5–C-5–C-6 | | | −174.7(2) |
| C-1–N-1–C-6–C-7 | 176.0(2) | −165.6(3) | |
| C-1–N-1–C-6–C-11 | −1.5(4) | 13.5(5) | |
| C-1–N-1–C-7–C-8 | | | −179.9(3) |
| C-1–N-1–C-7–C-12 | | | −1.4(5) |
| C-1–C-2–C-3–C-4 | 53.3(3) | 55.8(3) | −50.5(3) |
| C-2–C-1–O-5–C-5 | 60.1(3) | 63.0(3) | −61.7(3) |
| C-2–C-1–N-1–C-6 | −164.4(2) | −163.8(3) | |
| C-2–C-1–N-1–C-7 | | | 166.7(3) |
| C-2–C-3–C-4–C-5 | −53.1(3) | −55.4(4) | 50.6(3) |
| C-3–C-4–C-5–C-6 | | | −174.9(2) |

Table 2
Cremer–pople puckering parameters and asymmetry parameters

| | 1 | 2 | 3 |
|---|---|---|---|
| $Q$ (Å) | 0.568(3) | 0.597(3) | 0.560(3) |
| $\Theta$ (°) | 177.5(3) | 177.3(3) | 5.4(3) |
| $\Phi$ (°) | 172.0(78) | 188.0(98) | 353.3(34) |
| $\Delta C_S$(C-1, C-4) (°) | 5.6(2) | 6.3(3) | 8.4(2) |
| $\Delta C_S$(C-2, C-5) (°) | 5.1(2) | 5.6(3) | 7.8(2) |
| $\Delta C_S$(C-3, O-5) (°) | 0.8(2) | 1.2(3) | 0.6(2) |

to the aryl ring, but in **2** it is one of the S–O bonds that occupies this position. In **3** the orientation of the C-6–O-6 side chain is *gt*.[21] This particular orientation of the side chain is commonly observed in *N*-aryl-β-D-mannopyranosylamines, being found in seven of the eight other compounds of this type we have examined recently.[1]

*Packing arrangements and intermolecular interactions.*—The packing arrangement assumed by **1** is shown in Fig. 4. In this structure the few hydrogen bonding interactions that do occur between the sulfamoyl group and the monosaccharide are not particularly close, involving donor–acceptor distances of approximately 3.0 Å. One of these interactions is a hydrogen bond between the glycosidic N–H group (N-1–H) and one of the sulfamoyl oxygen atoms, O-7; the other is a hydrogen bond between the sulfamoyl $-NH_2$ group (H–N-2–H) and a monosaccharide hydroxyl oxygen atom, O-2. No hydrogen bonding interactions are found in which monosaccharide hydroxyl groups serve as H-bond donors to sulfamoyl oxygen atoms. On the other hand, interactions strictly between monosaccharide hydroxyl groups are observed, and these are closer, involving donor–acceptor distances of approximately 2.8 Å. The closest interaction in the structure is the O-4–H···O-5 hydrogen bond, in which the donor strength of the O-4 hydroxyl group may result from an intramolecular cooperative

Table 3
Hydrogen bond parameters [a]

| Compound | D–H···A | D–H (Å) | H···A (Å) | D···A (Å) | D–H···A (°) |
|---|---|---|---|---|---|
| **1** | O-2–H-20···O-4$^{i}$ | 0.89(3) | 1.99(3) | 2.795(3) | 151(3) |
| | O-3–H-30···O-4$^{ii}$ | 0.88(3) | 1.92(3) | 2.781(3) | 166(3) |
| | O-4–H-40···O-5$^{ii}$ | 0.80(3) | 1.96(3) | 2.754(3) | 171(3) |
| | N-1–H-50···O-7$^{iii}$ | 0.83(3) | 2.25(3) | 3.054(3) | 163(3) |
| | N-2–H-21···O-6$^{iv}$ | 1.03(3) | 2.03(3) | 3.037(4) | 164(3) |
| | N-2–H-22···O-2$^{v}$ | 0.87(3) | 2.10(3) | 2.965(3) | 172(3) |
| **2** | O-2–H-20···O-4$^{iii}$ | 0.79(4) | 1.97(4) | 2.749(3) | 168(4) |
| | O-3–H-30···O-6$^{vi}$ | 0.87(4) | 2.01(4) | 2.813(3) | 152(4) |
| | O-4–H-40···O-7$^{v}$ | 0.73(4) | 2.32(4) | 2.928(3) | 141(4) |
| | N-1–H-50···O-3$^{iii}$ | 0.80(4) | 2.22(4) | 3.015(4) | 172(4) |
| | N-2–H-21···O-3$^{ii}$ | 0.86(4) | 2.43(4) | 3.224(4) | 156(3) |
| | N-2–H-22···O-2$^{vii}$ | 0.90(4) | 2.03(4) | 2.900(4) | 163(3) |
| **3** | O-2–H-20···O-4$^{viii}$ | 0.72(3) | 2.34(3) | 2.951(3) | 144(4) |
| | O-3–H-30···O-5$^{ix}$ | 0.81(3) | 2.00(3) | 2.798(3) | 170(3) |
| | O-4–H-40···O-3$^{x}$ | 0.84(3) | 1.87(3) | 2.706(3) | 175(3) |
| | O-6–H-60···O-4$^{xi}$ | 0.74(3) | 2.04(3) | 2.776(3) | 173(4) |
| | O-9–H [b]···O-7$^{xii}$ | | | 3.10(1) | |
| | O-9–H [b]···O-8$^{i}$ | | | 2.91(1) | |
| | N-1–H-70···O-6$^{viii}$ | 0.81(3) | 2.39(3) | 3.112(4) | 149(3) |
| | N-2–H-21···O-7$^{xiii}$ | 0.87(3) | 2.15(3) | 2.974(4) | 158(3) |
| | N-2–H-21···O-9$^{xiv}$ | 0.87(3) | 2.99(4) | 3.30(1) | 103(3) |
| | N-2–H-22···O-8$^{xiv}$ | 0.87(3) | 2.12(3) | 2.985(4) | 170(3) |

[a] Symmetry codes: (i) $xyz$; (ii) $3/2-x, 1-y, 1/2+z$; (iii) $1/2+x, 3/2-y, -z$; (iv) $3/2-x, 2-y, 1/2+z$; (v) $2-x, 1/2+y, 1/2-z$; (vi) $2-x, 1/2+y, -1/2-z$; (vii) $2-x, -1/2+y, -1/2-z$; (viii) $1+x, y, z$; (ix) $x, y, 1+z$; (x) $-1/2+x, 1/2-y, 3-z$; (xi) $x, y, -1+z$; (xii) $-1+x, y, z$; (xiii) $5/2-x, -y, 1/2+z$; (xiv) $3/2-x, -y, 1/2+z$.

[b] H-atom positions were not determined for the partially occupied water molecule.

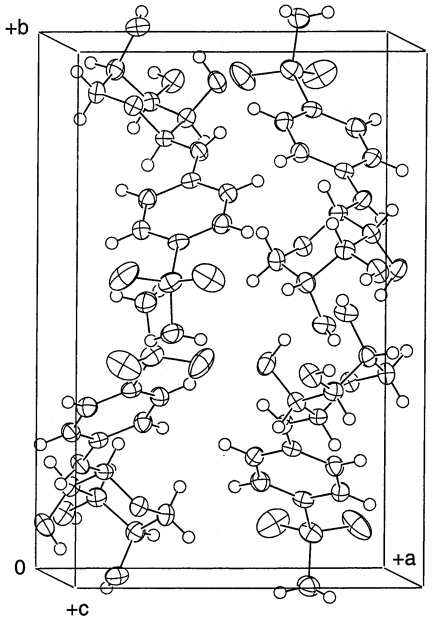



Fig. 4. Molecular packing in **1**.

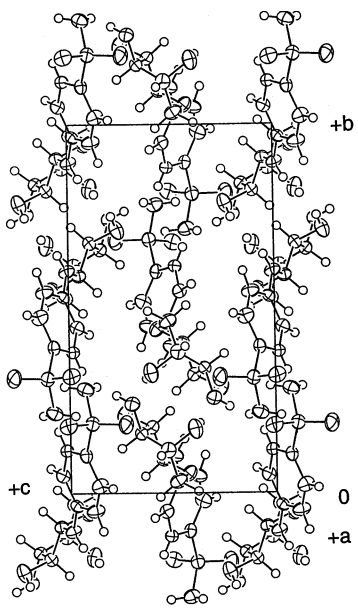



Fig. 5. Molecular packing in **2**.

effect,[22] O-4 also serving as the acceptor for the intramolecular hydrogen bond from O-2.

The packing arrangement assumed by **2** is shown in Fig. 5. The monosaccharide and sulfamoyl groups interact with each other to a greater extent in this structure than in **1** and **3**, with two of the three monosaccharide hydroxyl groups serving as hydrogen bond donors to sulfamoyl oxygen atoms. In addition, both hydrogen atoms of the sulfamoyl group are directed toward monosaccharide hydroxyl groups. The difference between **2** and the other two compounds in the orientation of the sulfamoyl group may be related to this higher degree of H-bonding interaction between sulfamoyl groups and monosaccharides, at least to the extent that the sulfamoyl conformation unique to **2** may facilitate these contacts. Similar to **1**, which makes a two-point contact with a neighboring screw-axially related molecule through O-3–H···O-4 and O-4–H···O-5 hydrogen bonds, **2** makes a two-point contact with a neighboring screw-axially related molecule through O-2–H···O-4 and N-1–H···O-3 hydrogen bonds.

The packing arrangement assumed by **3** is shown in Fig. 6. The compound crystallizes as a monohydrate, although the actual occupancy of the water oxygen atom was found to be only 22% in the crystal used for the X-ray analysis. The water molecule is located within a hydrogen bonding distance of both sulfamoyl oxygen atoms: O-9···O-7 $(-1+x,\ y,\ z) = 3.10(1)$ Å; O-9···O-8 $(xyz) = 2.91(1)$ Å. In this structure there are no hydrogen bonding interactions between the sulfamoyl groups and the monosaccharide hydroxyl groups. Thus left undisturbed by any such interactions, the hydrogen bonding pattern that links neighboring monosaccharides in **3** is identical to that found in several other mannopyranosylamines we have examined: *N*-phenylmannopyranosyl-

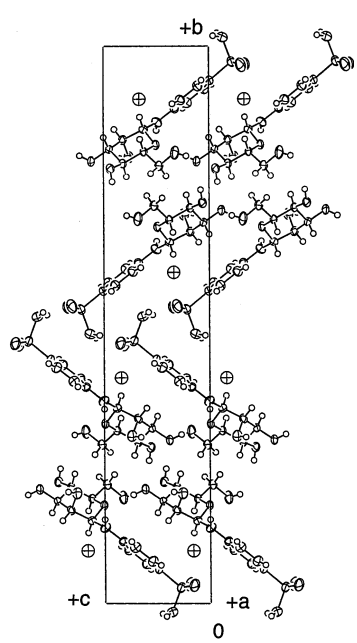



Fig. 6. Molecular packing in **3**.

amine and *N*-(*p*-chlorophenyl)-, *N*-(*p*-bromophenyl)-, *N*-(*p*-tolyl)-, and *N*-(*m*-chlorophenyl)mannopyranosylamine (the latter four compounds being isostructural).[1] This same pattern of H-bonds is preserved in **3** in spite of the presence of an aryl substituent that is very different, both sterically and electronically, from those in the previous structures, one that is capable of participating in multiple hydrogen bonds. This pattern is preserved also in spite of the presence of the water molecule, which is found to interact only with the sulfamoyl group and not at all with the monosaccharide hydroxyl groups. A portion of this pattern, the two-point contact in **3** defined by the O-3–H···O-5 and O-6–H···O-4 hydrogen bonds between translationally related neighboring molecules, is of special interest because it is found not only in **3** and in the structures listed above but also in three structures that have molecular packing patterns that are different overall from that found in **3**: *N*-(*p*-methoxyphenyl)-, *N*-(*o*-chlorophenyl)-, and *N*-(*o*-tolyl)mannopyranosylamine.[1] The recurrence of this pair of hydrogen bonds from structure to structure, in spite of overall differences in the packing arrangements, indicates that this two-point contact is an especially favorable mode of intermolecular interaction between mannopyranosylamines and may therefore play a role in their molecular recognition in biological systems.

## 3. Experimental

*Preparation of* N-*(*p-*sulfamoylphenyl)glycopyranosylamines*.—Compounds **1**–**3** were prepared by refluxing equimolar amounts (approximately 1 g each) of the monosaccharide and sulfanilamide in 95% EtOH for 15–30 min. The derivatives (**1**, mp 177–179 °C; **2**,

Table 4
Crystal data, data collection parameters, and refinement results for **1**–**3** [a]

| | **1** | **2** | **3** [b] |
|---|---|---|---|
| Empirical formula | $C_{11}H_{16}N_2O_6S$ | $C_{11}H_{16}N_2O_6S$ | $C_{12}H_{20}N_2O_8S$ |
| Formula weight | 304.32 | 304.32 | 352.36 |
| Crystal dimensions (mm) | $0.40\times0.14\times0.08$ | $0.56\times0.52\times0.44$ | $0.56\times0.46\times0.16$ |
| Crystal system | orthorhombic | orthorhombic | orthorhombic |
| Space group | $P2_12_12_1$ | $P2_12_12_1$ | $P2_12_12_1$ |
| $a$ (Å) | 10.195(3) | 9.992(2) | 6.744(1) |
| $b$ (Å) | 15.541(2) | 15.064(2) | 34.217(1) |
| $c$ (Å) | 8.283(2) | 8.473(2) | 6.455(1) |
| $V$ (Å$^3$) | 1312.4(4) | 1275.4(3) | 1489.5(3) |
| $Z$ | 4 | 4 | 4 |
| $D_{calcd}$ (g cm$^{-3}$) | 1.540 | 1.585 | 1.571 |
| $F(000)$ | 640 | 640 | 744 |
| $\mu$ (Cu K$_\alpha$) (cm$^{-1}$) | 24.83 | 25.55 | 23.76 |
| $2\theta_{max}$ (°) | 140.2 | 140.2 | 140.1 |
| Number of measured reflections | 2916 | 2500 | 3286 |
| Number of unique reflections | 1458 | 1250 | 1644 |
| $R_{int}$ | 0.033 | 0.029 | 0.020 |
| Number of observed reflections [$I>0.00\sigma(I)$] | 2471 | 2091 | 2711 |
| Number of variables | 200 | 230 | 225 |
| Transmission coefficients | 0.8305–1.0000 | 0.7943–1.0000 | 0.6189–1.0000 |
| Secondary extinction[23] | $5.27078\times10^{-6}$ | $9.75943\times10^{-6}$ | $9.07442\times10^{-6}$ |
| Min/max in final difference map (e Å$^{-3}$) | −0.33, 0.29 | −0.28, 0.23 | −0.93, 1.02 |
| $R$, $wR$ | 0.052, 0.032 | 0.038, 0.036 | 0.064, 0.037 |

[a] For all three structures: $T=298$ K; diffractometer: Rigaku AFC6S; radiation: Cu K$_\alpha$; $\lambda=1.54178$ Å; cell determination: 25 reflections, $46°<2\theta<50°$; data collection: MSC/AFC control software;[24] scan mode: $\omega/2\theta$; structure solution: SHELXS 86;[25] structure refinement: TEXSAN software package;[26] decay correction: none required; absorption corrections: psi scans;[27] figures: ORTEPII;[15] $R=\Sigma||F_o|-|F_c||/\Sigma|F_o|$; $wR=[(\Sigma w(|F_o|-|F_c|)^2/\Sigma wF_o^2]^{1/2}$; $w=4F_o^2/\sigma^2(F_o^2)$; $R_{int}=\Sigma\Sigma|<F_i^2>-F_{ij}^2|/\Sigma m<F_i^2>$.

[b] Values given are for full monohydrate; actual occupancy found for water molecule was 22%.

mp 182–189 °C (lit.[13] 191 °C for L-isomer); **3**, mp 188–190 °C (lit.[13] 194 °C) crystallized upon concentration and cooling of the solution.[13] Compound **3** was recrystallized from water for X-ray structure determination.

*X-ray crystal structure determinations.*—A summary of the crystal data, data collection parameters, and refinement results is given in Table 4. In **1** and **2**, all non-hydrogen atoms were refined anisotropically. In **3**, the water oxygen atom was refined isotropically and the other non-hydrogen atoms were refined anisotropically. The occupancy of the water oxygen atom was determined by decreasing its occupancy from 100% stepwise as long as both the *R* factor for the structure and the isotropic *B* value for this atom continued to decrease. The minimum value of *R* was reached at occupancy of 22%. In **1** and **3**, attempts to refine all of the hydrogen atoms led to unreasonable bond lengths, so hydrogen atoms bonded to carbon atoms were placed in calculated positions and only those hydrogen atoms bonded to oxygen or nitrogen were refined (positional parameters only). In **2**, the positional parameters of all the hydrogen atoms were refined.

## 4. Supplementary material

Full crystallographic details (excluding structure features) has been deposited with the Cambridge Crystallographic Data Centre, for compounds 1–3. Copies of this data may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

## Acknowledgements

The authors thank Dr William B. Gleason of the Department of Laboratory Medicine and Pathology of the University of Minnesota for making the X-ray crystallographic facilities of the department available to us. The support of the Minnesota Medical Foundation in establishing these facilities is also gratefully acknowledged.

## References

[1] (a) Ojala, W. H.; Ostman, J. M.; Ojala, C.R. *Carbohydr. Res.* **2000**, *326*, 104–112. (b) Ojala, C. R.; Ostman, J. M.; Hanson, S. E.; Ojala, W. H. *Carbohydr. Res.* submitted. (c) Ojala, W. H.; Hanson, S. E.; Ojala, C. R.; Ostman, J. M. *Carbohydr. Res.* submitted.
[2] Ojala, W. H.; Gleason, W. B. *Acta Crystallogr., Sect. C* **1996**, *52*, 3188–3190.
[3] Furberg, S.; Solbakk, J. *Acta Chem. Scand.* **1969**, *23*, 3248–3256.
[4] Smith, F.; Butler, K.; Overend, W. G.; Stacey, M. *Chem. Ind. (London)* **1949**, 551.
[5] Butler, K.; Laland, S.; Overend, W. G.; Stacey, M. *J. Chem. Soc.* **1950**, 1433–1439.
[6] Ellis, G. P.; Honeyman, J. *Nature (London)* **1951**, *167*, 239–240.
[7] Ellis, G. P.; Honeyman, J. In *Advances in Carbohydrate Chemistry*; Wolfrom, M. L., Ed.; Academic Press: New York, 1955; Vol. 10, pp. 95–120.
[8] Ellis, G. P. *J. Chem. Soc. B* **1966**, 572–576.
[9] Dokurno, P.; Lubkowski, J.; Myszka, H.; Smiatacz, Z. *Z. Kristallogr.* **1994**, *209*, 808–812.
[10] Sanghvi, Y. S.; Larson, S. B.; Robins, R. K.; Revankar, G. R.; Gupta, P. K.; George, R. D.; Dalley, N. K. *J. Heterocycl. Chem.* **1988**, *25*, 623–633.
[11] Dideberg, P. O.; Lamotte, J.; Dupont, L. *Acta Crystallogr., Sect. B* **1980**, *36*, 1500–1503.
[12] Wang, L.; Maniglia, C. A.; Mella, S. L.; Sartorelli, A. C. *J. Med. Chem.* **1983**, *26*, 629–632.
[13] Bognár, R.; Nánási, P. *J. Chem. Soc.* **1953**, 1703–1708.
[14] Northey, E. H. In *The Sulfonamides and Allied Compounds, American Chemical Society Monograph Series*; Hamor, W. A., Ed.; Reinhold Publishing: New York, 1948; pp. 46–47.
[15] Johnson, C. K. *ORTEPII, Report ORNL-5138*; Oak Ridge National Laboratory: Oak Ridge TN, USA, **1976**.
[16] Cremer, D.; Pople, J. A. *J. Am. Chem. Soc.* **1975**, *97*, 1354–1358.
[17] Boeyens, J. C. A. *J. Cryst. Mol. Struct.* **1978**, *8*, 317–320.
[18] Köll, P.; Saak, W.; Pohl, S.; Steiner, B.; Koós, M. *Carbohydr. Res.* **1994**, *265*, 237–248.
[19] Duax, W. L.; Weeks, C. M.; Rohrer, D. C. *Top. Stereochem.* **1976**, *9*, 271–383.
[20] Spek, A. L. *Acta Crystallogr., Sect. A* **1990**, *46*, C34.
[21] Jeffrey, G. A. *Acta Crystallogr., Sect. B* **1990**, *46*, 89–103.
[22] Jeffrey, G. A.; Mitra, J. *Acta Crystallogr., Sect. B* **1983**, *39*, 469–480.
[23] Zachariasen, W. H. *Acta Crystallogr.* **1963**, *16*, 1139–1144.
[24] *Molecular Structure Corporation, MSC/AFC Diffractometer Control Software*; MSC: 9009 New Trails, The Woodlands TX, USA, **1988**.
[25] Sheldrick, G. M. *SHELXS 86, Program for the Solution of Crystal Structures*; University of Göttingen: Germany, **1985**.
[26] *Molecular Structure Corporation, TEXSAN, Single Crystal Structure Analysis Software, Version 1.7-1*; MSC: 9009 New Trails, The Woodlands TX, USA, **1995**.
[27] North, A. C. T.; Phillips, D. C.; Mathews, F. S. *Acta Crystallogr., Sect. A* **1968**, *24*, 351–359.