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# THE CRYSTAL STRUCTURE OF ISOPROPYL 1-THIO-β-D-GALACTOPYRANOSIDE MONOHYDRATE AT 123 K

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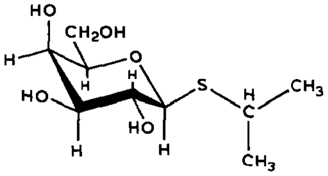
## ABSTRACT

The crystal structure of isopropyl 1-thio-β-D-galactopyranoside monohydrate is orthorhombic, $P2_12_12_1$, $Z = 4$, with cell dimensions at 123 K [293 K] of $a = 7.983(1)$ [8.037(1)], $b = 24.574(5)$ [24.709(4)], $c = 6.329(1)$ [6.3736(8)] Å, $V = 1241.84$ [1265.71] Å$^3$. The calculated and measured density is $D_x = 1.371$ [1.345] g cm$^{-3}$, $D_m =$ [1.340] g cm$^{-3}$. Diffraction data were obtained with CuK$\alpha$ radiation and a Nonius CAD-4 diffractometer. The structure was solved by using MULTAN, and refined to $R(F^2) = 0.051$, $R_w(F^2) = 0.078$, $R(F) = 0.029$, $S = 1.16$ for 1502 reflections. The molecule has the $^4C_1$(D) conformation. The orientation of the primary alcohol group is *gauche/trans*, and that about the glycosidic C–S bond is (−)*synclinal* relative to the ring C–O bond. Although this compound does not form thermotropic liquid crystals, it has two crystal-to-crystal phase-transitions, at 70 and 104°, prior to melting at 126°. The crystal structure has a characteristic, amphiphilic, head-to-head bilayer molecular packing, with intercalated alkyl groups. The water molecule is included in the hydrogen-bond structure that links the galactoside moieties.

## INTRODUCTION

Isopropyl 1-thio-β-D-galactopyranoside (**1**) is an amphiphilic molecule, as it combines an unblocked sugar moiety with a hydrocarbon moiety. It undergoes no thermotropic phase-transition leading to liquid-crystal formation as do the alkyl glycosides having acyclic chains exceeding six carbon atoms[1,2].

Because we are interested in the relationship, if any, between the molecular packing and hydrogen bonding in the crystalline state and the formation of liquid



**1**

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crystals, the question asked is, do carbohydrates that are mesogens have crystal structural packing different from that of those which are not?

## EXPERIMENTAL

Crystals of isopropyl 1-thio-β-D-galactopyranoside monohydrate were obtained by recrystallization from methanol of a sample obtained from Calbiochem (San Diego, CA 92112, USA). The crystal and intensity measurement data were obtained by using a Nonius CAD-4 diffractometer at 123 K. The structure was solved by using MULTAN (ref. 3) with 150 E-values greater than 1.53. The E-map for the best solution showed 14 of the 16 non-hydrogen atoms, and the remaining non-hydrogen and all hydrogen atoms were located in subsequent difference-Fourier syntheses. The atomic parameters were refined by a full-matrix, least-squares procedure, with anisotropic thermal parameters for the non-hydrogen atoms. In the final stages of the refinement, the measured intensities were corrected for absorption, and an isotropic extinction parameter (type I, Becker–Coppens formalism[4,5]) was included. Several low-angle reflections were seen to be severely affected by extinction, (0 4 1) with $F_o^2 = 0.465, F_c^2$ being the most affected. The crystal density was measured by using a Westphal balance with a benzene–carbon tetrachloride flotation-mixture at 293 K.

The crystal and refinement data are given in Table I. The atomic notation

TABLE I

CRYSTAL STRUCTURE AND REFINEMENT DATA FOR ISOPROPYL 1-THIO-β-D-GALACTOPYRANOSIDE MONOHYDRATE

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$C_9H_{18}O_5S \cdot H_2O$; m.w. = 256.32; $P2_12_12_1$; $Z = 4$

Cell dimensions at 123 K [293 K]: $a = 7.983(1)$ [8.037(1)], $b = 24.579(5)$ [24.709(4)], $c = 6.329(1)$ [6.3736(8)] Å

$V = 1241.84$ [1265.71] Å$^3$; $D_x = 1.371$ [1.345], $D_m =$ [1.340] g cm$^{-3}$

Crystal dimensions: 0.28 × 0.31 × 0.47 mm

Radiation CuK$_\alpha$ ($\lambda = 1.5418$ Å), Ni filter, $\mu_{\mathrm{CuK}_\alpha} = 23.89$ cm$^{-1}$

Cell dimensions based on 40 reflections with $35° < \theta < 44°$

1505 unique intensities measured by $\omega/2\theta$ scan, 3 considered unobserved with $F_o < \sigma(F_o)$; $\theta_{max} = 75°$; range of hkl: h, 0 → 10; k, 0 → 30; l, 0 → 7

Function minimized, R = $\Sigma[w(|kF_o^2| - |F_c^2|)]$, using program UPALS (ref. 6), where w = $1/[C.\sigma(F_o^2)]$, with $\sigma(F_o^2)$ based on counting statistics; initially, $C = 1$, but in the final stages of the refinement, $C = 2.63$, taken from the slope of a normal probability plot of the residuals in a previous run with $C = 1$

Absorption corrections applied prior to final refinement by using program ABL (ref. 7): transmission coefficients in the range 0.4428 to 0.6201

Isotropic type I extinction coefficient included in the final refinement converged to a value corresponding to a r.m.s. mosaic spread of 0.61(6) × $10^{-4}$ rad

Final agreement factors: $R(F_o^2) = 0.051$, $R_w(F_o^2) = 0.078$, $R(F_o) = 0.029$, $S = 1.16$, based on 1502 observed reflections

Maximum shift/e.s.d. ratio in final refinement cycle < 0.09

Highest peak in final difference-Fourier synthesis < 0.38 $e$/Å$^3$

Atomic scattering factors from International Tables for X-ray Crystallography (ref. 8)

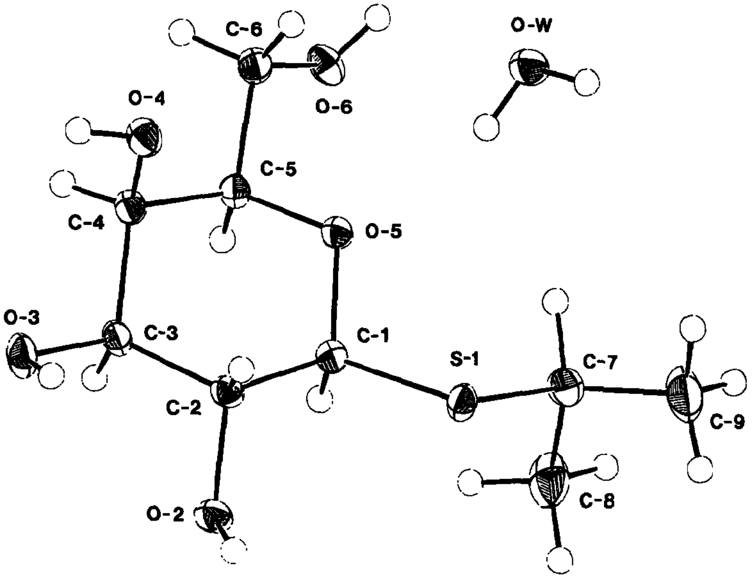

Fig. 1. Atomic notation and thermal ellipsoids (50% probability) for isopropyl 1-thio-β-D-galactopyranoside monohydrate at 123 K.

and thermal ellipsoids are shown in Fig. 1. Atomic positional and thermal parameters* are given in Table II and the molecular dimensions in Table III.

## DISCUSSION

*The molecular structure.* — The galactopyranoside has the $^4C_1$(D) conformation, with Cremer–Pople puckering parameters[9] $Q$ = 0.570(7) Å, $\theta$ = 9(4)°, and $\varphi$ = 319(29)°, which are within the range normally observed in the crystal structures of monosaccharides. The orientation of the primary alcohol group is *gauche/trans,* with O-6–C-6–C-5–O-5 = 78.7(2)°; this is one of the two orientations that avoid a *peri* interaction with the galacto hydroxyl group on C-4. The thioglycoside linkage is (−)*synclinal,* with O-5–C-5–S-1–C-7 = −72.1(1)°, consistent with the exo-anomeric effect. The ring C–C bond-lengths lie in the normal range, 1.516 to 1.540 Å. The exocyclic C–O bond-lengths lie in the range 1.425 to 1.434 Å, with a mean value of 1.428 Å and a r.m.s. dispersion of 0.004 Å, and are not significantly different. However, the two ring C–O bond-lengths are significantly different, with C-1–O-5 = 1.422(2) Å, and C-5–O-5 = 1.446(2) Å. This difference is considerably larger than expected from observations and theoretical calculations relating to β-D-aldopyranosides[10]. The two C–S bond-lengths are also significantly different, with

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*Lists of structure factors and anisotropic thermal parameters are deposited with, and can be obtained from, Elsevier Scientific Publishing Company, BBA Data Deposition, P.O. Box 1527, Amsterdam, The Netherlands. Reference should be made to No. BBA/DD/342/*Carbohydr. Res.*, 153 (1986) 217–226.

## TABLE II

ATOMIC PARAMETERS[a] FOR THE CRYSTAL STRUCTURE OF ISOPROPYL 1-THIO-$\beta$-D-GALACTOPYRANOSIDE MONOHYDRATE

| *Atom* | *Fractional coordinates*[b] | | | $B_{eq}$ *or* $B_{iso}$[c] |
|---|---|---|---|---|
| | $x$ | $y$ | $z$ | |
| S-1 | 62220(6) | 41331(2) | 73155(8) | 135(1) |
| C-1 | 6169(2) | 3829(1) | 4727(3) | 122(4) |
| C-2 | 7639(2) | 3428(1) | 4506(3) | 123(4) |
| C-3 | 7482(2) | 3097(1) | 2475(3) | 123(4) |
| C-4 | 5717(3) | 2869(1) | 2156(3) | 124(4) |
| C-5 | 4467(2) | 3330(1) | 2376(3) | 118(4) |
| C-6 | 2678(3) | 3155(1) | 2022(3) | 138(4) |
| C-7 | 4767(3) | 4705(1) | 6964(4) | 184(5) |
| C-8 | 5499(4) | 5147(1) | 5570(5) | 256(6) |
| C-9 | 4353(4) | 4916(1) | 9164(5) | 286(6) |
| O-2 | 9158(2) | 3730(1) | 4355(3) | 151(3) |
| O-3 | 8694(2) | 2672(1) | 2371(3) | 177(3) |
| O-4 | 5334(2) | 2456(1) | 3661(3) | 158(3) |
| O-5 | 4596(2) | 3566(1) | 4461(2) | 121(3) |
| O-6 | 1698(2) | 3624(1) | 1498(3) | 159(3) |
| O-W | 1614(2) | 3571(1) | 7240(3) | 170(3) |
| H-1 | 635(4) | 412(1) | 353(5) | 18(5) |
| H-2 | 767(3) | 321(1) | 569(4) | 10(4) |
| H-3 | 770(3) | 335(1) | 117(5) | 15(5) |
| H-4 | 566(3) | 273(1) | 73(4) | 11(4) |
| H-5 | 476(3) | 361(1) | 130(4) | 12(5) |
| H-61 | 265(3) | 292(1) | 82(5) | 16(5) |
| H-62 | 226(4) | 299(1) | 325(5) | 16(5) |
| H-7 | 367(4) | 454(1) | 633(5) | 21(5) |
| H-81 | 467(4) | 544(1) | 543(5) | 31(7) |
| H-82 | 654(4) | 530(1) | 622(6) | 31(7) |
| H-83 | 589(4) | 501(1) | 420(5) | 25(6) |
| H-91 | 382(6) | 462(2) | 998(7) | 55(10) |
| H-92 | 358(5) | 519(1) | 891(6) | 35(7) |
| H-93 | 534(5) | 505(2) | 997(6) | 46(9) |
| H-O-2 | 975(4) | 371(1) | 548(5) | 27(6) |
| H-O-3 | 910(4) | 262(1) | 372(5) | 20(5) |
| H-O-4 | 580(4) | 219(1) | 318(5) | 20(5) |
| H-O-6 | 87(4) | 362(1) | 225(5) | 21(6) |
| H1-W | 261(4) | 360(1) | 680(5) | 24(6) |
| H2-W | 167(4) | 364(1) | 863(6) | 33(7) |

[a] E.s.d. values, given in parentheses, refer to the least significant digit. [b] Fractional coordinates × $10^5$ for sulfur, × $10^4$ for carbon and oxygen, × $10^3$ for hydrogen atoms. [c] Values for non-hydrogen atoms are $B_{eq} = 4/3(\Sigma_{ij}\beta_{ij}\,\vec{a}_i \cdot \vec{a}_j)$, calculated in Å$^2$ from the refined anisotropic thermal parameters, and multiplied by $10^2$. Values for hydrogen atoms are $B_{iso} \times 10$, in Å$^2$, from the refined isotropic thermal parameters.

TABLE III

MOLECULAR DIMENSIONS OF ISOPROPYL 1-THIO-β-D-GALACTOPYRANOSIDE[a]

| | | | |
|---|---|---|---|
| *Bond lengths* | | | |
| C-1–C-2 | 1.540(3) | C-3–O-3 | 1.426(3) |
| C-2–C-3 | 1.526(3) | C-4–O-4 | 1.426(3) |
| C-3–C-4 | 1.530(3) | C-5–C-6 | 1.508(3) |
| C-4–C-5 | 1.516(3) | S-1–C-7 | 1.837(2) |
| C-5–O-5 | 1.446(2) | C-6–O-6 | 1.434(3) |
| O-5–C-1 | 1.422(2) | C-7–C-8 | 1.517(4) |
| C-1–S-1 | 1.801(2) | C-7–C-9 | 1.522(4) |
| C-2–O-2 | 1.425(3) | | |
| *Valence angles* | | | |
| C-1–C-2–C-3 | 110.8(2) | C-2–C-3–O-3 | 111.9(2) |
| C-2–C-3–C-4 | 112.5(2) | C-4–C-3–O-3 | 110.5(2) |
| C-3–C-4–C-5 | 108.7(2) | C-3–C-4–O-4 | 111.8(2) |
| C-4–C-5–O-5 | 109.7(2) | C-5–C-4–O-4 | 109.2(2) |
| C-5–O-5–C-1 | 110.7(2) | C-4–C-5–C-6 | 113.3(2) |
| O-5–C-1–C-2 | 111.8(2) | O-5–C-5–C-6 | 108.5(2) |
| O-5–C-1–S-1 | 108.4(2) | C-1–S-1–C-7 | 101.1(1) |
| C-2–C-1–S-1 | 109.3(1) | C-5–C-6–O-6 | 108.7(2) |
| C-1–C-2–O-2 | 108.7(2) | S-1–C-7–C-8 | 112.0(2) |
| C-3–C-2–O-2 | 106.9(2) | S-1–C-7–C-9 | 106.7(2) |
| | | C-8–C-7–C-9 | 111.8(2) |
| *Torsion angles* | | | |
| C-1–C-2–C-3–C-4 | −46.8(2) | C-5–C-4–C-3–O-3 | 177.5(2) |
| C-2–C-3–C-4–C-5 | 51.6(2) | C-4–C-3–C-2–O-2 | −165.1(2) |
| C-3–C-4–C-5–O-5 | −60.3(2) | C-3–C-2–C-1–S-1 | 171.1(1) |
| C-4–C-5–O-5–C-1 | 66.5(2) | S-1–C-1–C-2–O-2 | −71.7(2) |
| C-5–O-5–C-1–C-2 | −61.2(2) | O-2–C-2–C-3–O-3 | 69.8(2) |
| O-5–C-1–C-2–C-3 | 51.0(2) | O-3–C-3–C-4–O-4 | 56.8(2) |
| C-1–C-2–C-3–O-3 | −171.9(2) | O-4–C-4–C-5–C-6 | −59.6(2) |
| C-2–C-3–C-4–O-4 | −69.0(2) | C-4–C-5–C-6–O-6 | −159.1(2) |
| C-3–C-4–C-5–C-6 | 178.2(2) | O-5–C-1–S-1–C-7 | −72.1(1) |
| C-5–O-5–C-1–S-1 | 178.2(1) | O-5–C-5–C-6–O-6 | 78.7(2) |
| O-5–C-1–C-2–O-2 | 168.2(2) | C-2–C-1–S-1–C-7 | 165.8(1) |
| C-1–O-5–C-5–C-6 | −169.1(2) | C-1–S-1–C-7–C-8 | −71.1(2) |
| O-5–C-5–C-4–O-4 | 61.9(2) | C-1–S-1–C-7–C-9 | 166.3(2) |

[a] Bond lengths in Å, valence and torsion angles in degrees. E.s.d. values, given in parentheses, refer to the least significant digit.

C-1–S-1 = 1.801(2) Å and S-1–C-7 = 1.827(2) Å. As shown in Table IV, however, there are no apparent systematics in the differences (some of which are significant) in the C–S bond-lengths observed in the crystal structures of various 1-thiopyranosides.

*The molecular packing and hydrogen bonding.* — As shown in Fig. 2, the molecular packing is typical head-to-head bilayer, with intercalating alkyl groups. Except that the isopropyl groups are bulky, rather than fully extended chains, the crystal structure is analogous to those of heptyl 1-thio-α-D-mannopyranoside[15],

TABLE IV

STRUCTURAL AND CONFORMATIONAL DATA FOR THE THIOGLYCOSIDE LINKAGE IN SOME 1-THIOPYRANOSIDES

| *Compound* | *Conformation* | *Bond lengths*[a] <br> $\left\{\begin{matrix}O\text{-}5\\S\text{-}5\end{matrix}\right\}$—C-1 | C-1—S-1 ($\varphi$) | S-1—$\left\{\begin{matrix}C\text{-}6\\C\text{-}7\end{matrix}\right\}$ | $\varphi$ |
|---|---|---|---|---|---|
| Compound **1** | $^4C_1$ D | 1.422(2) | 1.801(2) | 1.837(2) | −72.1(2) |
| Heptyl 1-thio-α-D-mannopyranoside[b] | $^4C_1$ D | 1.433(2) | 1.819(2) | 1.824(2) | +67.7(2) |
| Pyridyl 1-thio-β-D-glucopyranoside[c] | $^4C_1$ D | 1.421(4) | 1.793(3) | 1.759(3) | −74.9(2) |
| Methyl 1-thio-β-D-galactopyranoside[d] | $^4C_1$ D | 1.429(2) | 1.806(1) | 1.811(3) | −99.4(2) |
| Methyl 1-thio-α-D-ribopyranoside[e] | $^1C_4$ D | 1.435(4) | 1.796(3) | 1.809(6) | +75.2(2) |
| Methyl 1,5-dithio-α-D-ribopyranoside · ¼ $H_2O$[f,g] | $^1C_4$ D | 1.807(5) | 1.777(6) | 1.756(8) | +159.0(3) |
| | | 1.846(6) | 1.803(6) | 1.848(10) | +81.5(4) |
| Methyl 1,5-dithio-β-D-ribopyranoside[f,g] | $^4C_1$ D | 1.83(1) | 1.81(1) | 1.78(2) | −66.7(6) |
| | | 1.79(1) | 1.80(1) | 1.78(2) | −66.3(7) |

[a] Bond lengths in Å, angles in degrees. E.s.d. values, given in parentheses, refer to the least significant digit. [b] Ref. 1. [c] Ref. 11. [d] Ref. 12. [e] Ref. 13. [f] Ref. 14. [g] There are two molecules per asymmetric unit.

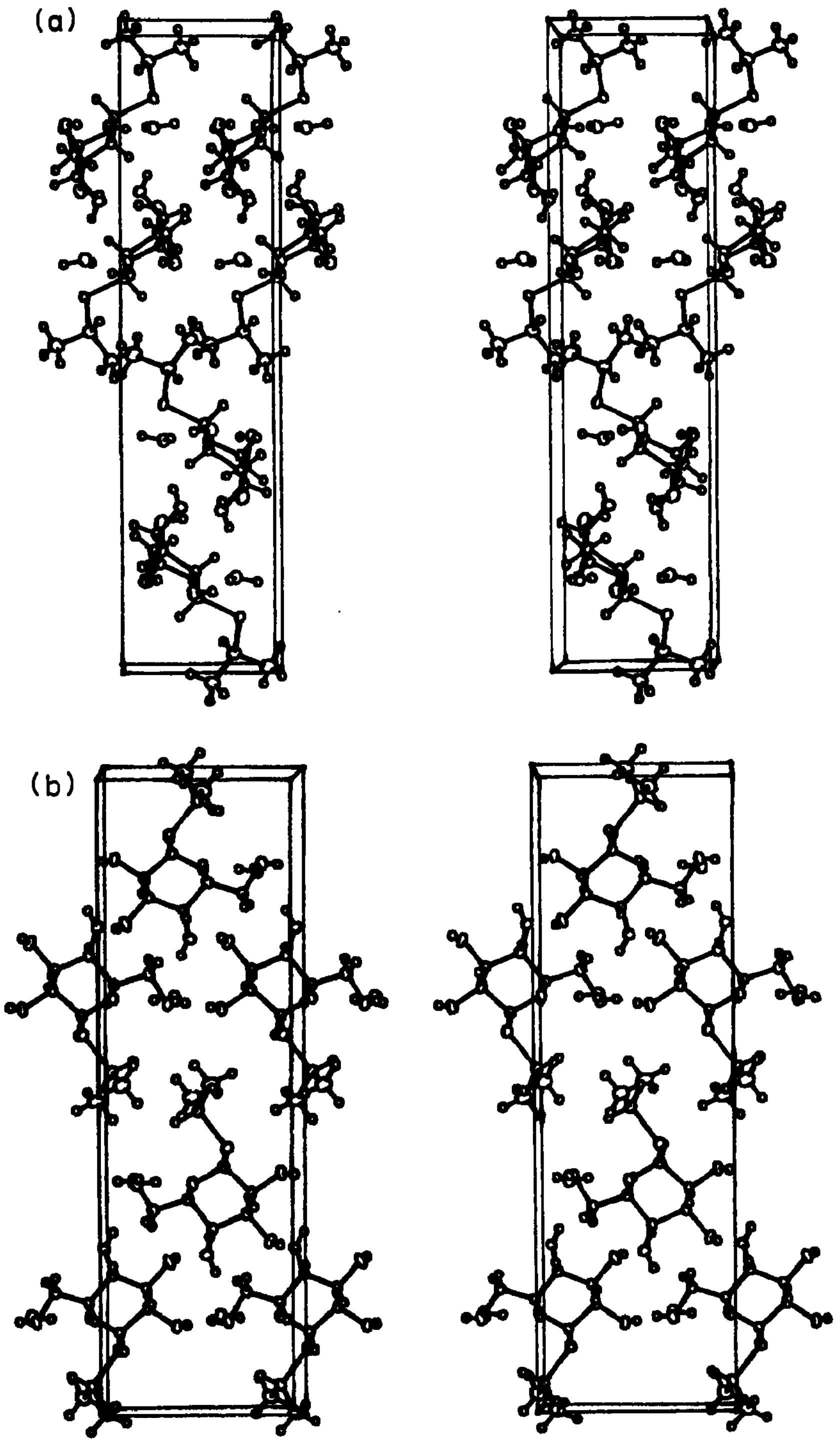

Fig. 2. Stereoviews of the molecular packing in the crystal structure of isopropyl 1-thio-β-D-galactopyranoside monohydrate at 123 K. In (a), the view is down the *a* axis, and the *c* axis points to the left. In (b), the view is down the *c* axis. In both views the *b* axis is vertical and points up.

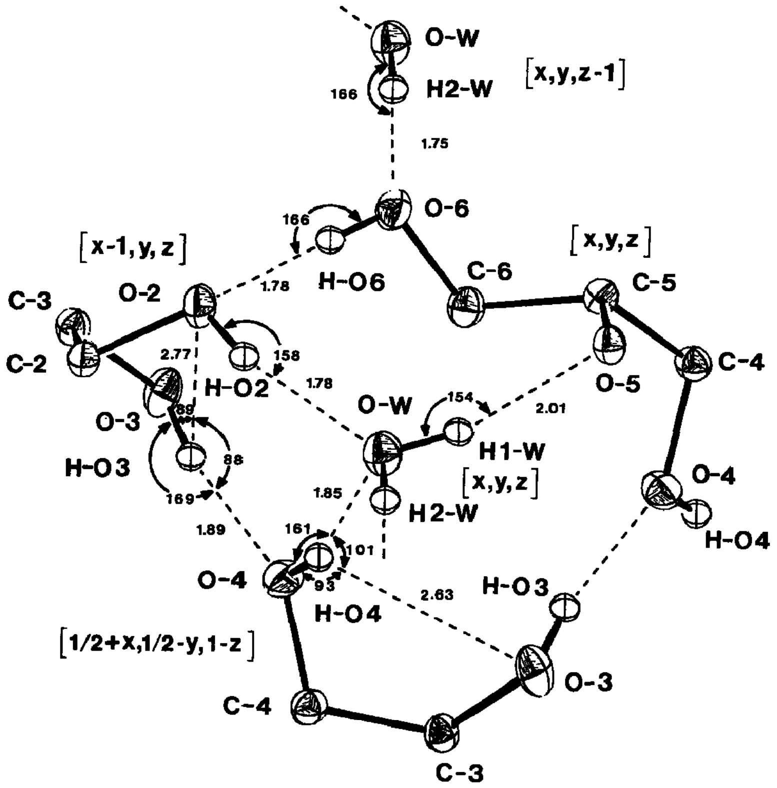

Fig. 3. Hydrogen bonding in the crystal structure of isopropyl 1-thio-β-D-galactopyranoside monohydrate. The H · · · O distances and O–H · · ·O angles were obtained by normalizing the covalent O–H distances to the standard value of 0.97 Å (ref. 18).

TABLE V

GEOMETRICAL DATA FOR THE HYDROGEN-BONDED STRUCTURE OF THE GALACTOPYRANOSIDE MONOHYDRATE MOIETY[a]

| *Contact* | *O–H* | *H · · · O* | *O–H · · · O* |
|---|---|---|---|
| O-2–H · · · O-W [*x* + 1, *y*, *z*] | 0.85(3) | 1.89(3) | 158(3) |
| O-3–H · · · O-4 [½ + *x*, ½ − *y*, 1 − *z*] | 0.92(3) | 1.94(3) | 170(3) |
| · · · O-2 [*x*, *y*, *z*][b] | | 2.77(3) | 90(2) |
| O-4–H · · · O-W [½ + *x*, ½ − *y*, 1 − *z*] | 0.81(3) | 2.00(3) | 162(3) |
| · · · O-3 [*x*, *y*, *z*][c] | | 2.65(3) | 97(2) |
| O-6–H · · · O-2 [*x* − 1, *y*, *z*] | 0.82(3) | 1.93(3) | 167(3) |
| O-W–H-1 · · · O-5 [*x*, *y*, *z*] | 0.85(3) | 2.17(3) | 155(3) |
| O-W–H-2 · · · O-6 [*x*, *y*, *z* + 1] | 0.90(4) | 1.81(3) | 167(3) |

[a] Distances in Å, angles in degrees. E.s.d. values, given in parentheses, refer to the least significant digit. [b] Atom H-O-3 is at 0.11(3) Å from the plane defined by atoms O-2 [*x*, *y*, *z*], O-3 [*x*, *y*, *z*] and O-4 [½ + *x*, ½ − *y*, 1 − *z*]. [c] Atom H-O-4 is at 0.12(3) Å from the plane defined by atoms O-3 [*x*, *y*, *z*], O-4 [*x*, *y*, *z*], and O-W [½ + *x*, ½ − *y*, 1 − *z*].

octyl 1-thio-α-D-xylopyranoside[16], and decyl α-D-glucopyranoside[17], which form thermotropic liquid crystals.

The galactopyranose residues are hydrogen-bonded with the water molecule, as shown in Fig. 3, with the geometry given in Table V. The hydrogen-bond structure consists of an infinite chain, →O-6-H→O-2-H→O-W-H→O-6-H→, which intersects with a finite chain at the water molecule. The finite chain, O-3-H→O-4-H→OW-H→O-5, terminates at the ring-oxygen atom. There are two weak intramolecular components of three-centered bonds, the first from O-3-H→O-2 and the second from O-4-H→O-3, which connect the finite chains with the infinite chains and one another, respectively*. Similar hydrogen-bonding patterns have been observed in other pyranose and pyranoside crystal structures[20].

The isopropyl groups interlock, with the shortest-contact distance being H-7 $[x, y, z]$ · · ·H-92 $[2 - x, -y + 1, \frac{1}{2} + z]$ = 2.45(5) Å.

Although this structure resembles that of the mesogenic alkyl glycosides in having two crystal-to-crystal phase-transitions prior to melting, it does not form a thermomesophase. Clearly, this requires more than an amphiphile molecule *per se*; the conformation of the hydrophobic aglycon is important.

ACKNOWLEDGMENTS

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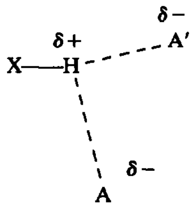
*We use the descriptor *three-centered H-bond* for an orientation where there is evidence of an attractive force between a deshielded hydrogen atom and two electronegative acceptor-atoms, as in



where the H atom is in, or close to, the plane of X, A, and A′ (see ref. 19). At these distances, the primary attractive component of the hydrogen bond is electrostatic, and attenuates as $r^{-1}$.

The use of a van der Waals criterion for hydrogen bonding dates back to 1960, at which time hydrogen atoms were rarely located in crystal-structure analyses. When the hydrogen atoms *are* located, it becomes irrelevant, as the attractive component of the van der Waals forces attenuates as $\sim r^{-6}$.

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