Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1043). Services for accessing these data are described at the back of the journal.

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Lactosylurea Dihydrate

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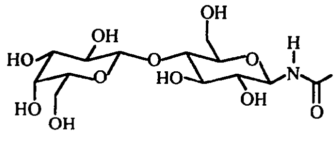
Abstract

Lactosylurea dihydrate [(4-*O*-β-D-galactopyranosyl-1-*N*-β-D-glucopyranosyl)urea], $C_{13}H_{24}N_2O_{11}.2H_2O$, crystallizes as a dihydrate in the space group $P1$. There are twelve distinct hydrogen bonds, including one intramolecular hydrogen bond joining the glucose and galactose rings. This structure determination establishes that the reaction of urea with lactose does not lead to rearrangement or to an acyclic form of lactose and that the urea forms a β-*N*-glycoside linkage.

Comment

As the major carbohydrate component of whey (Gouda, Larm & Larsson, 1980), lactose is a surplus carbohydrate which is of interest as a reusable resource. Reaction with urea to produce a lactosylurea makes it more useful as an animal feed (Merry, Smith & McAllan, 1982). Reduction of lactosylurea may also yield a cheap starting material for the manufacture of polyurethane. The structure of the urea-modified saccharide, (I), has been inferred from spectroscopy and chemical means (Benn & Jones, 1960; Segal, O'Connor & Eggerton, 1960; Shkantova, Dudkin & Grinshpun, 1967; Cerbulis, Pfeffer & Farrell, 1978), but there has been disagreement on whether or not rearrangement or ring opening occurs.



(I)

This structure determination shows that the lactosylurea has a β-*N*-glycoside linkage and that the ring structure is otherwise unchanged. There is an intramolecular hydrogen bond joining the O3′ H atom of glucose to O5 of galactose, as is also found in the structures of β-lactose (Hirotsu & Shimada, 1974) and in α-lactose monohydrate (Noordik, Beurskens, Visser & Gould, 1984). The comparable O···O distances are 2.744, 2.707 and 2.813 Å, respectively. There are eleven other relatively strong hydrogen bonds, including those to the two water molecules of hydration.

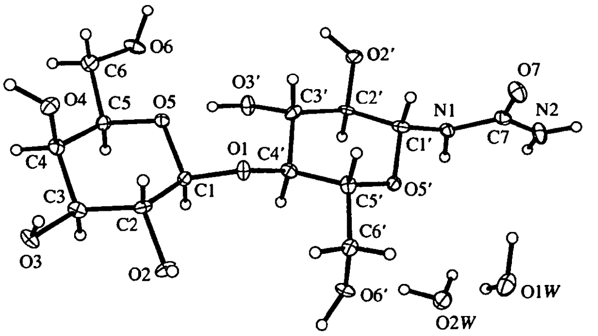


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms are shown as small circles of arbitrary radii.

Experimental

The title compound was prepared following the literature procedure of Merry, Smith & McAllan (1982).

Crystal data

$C_{13}H_{24}N_2O_{11}.2H_2O$	Cu $K\alpha$ radiation
$M_r = 420.37$	$\lambda = 1.54178$ Å

Triclinic
$P1$
$a = 4.7378\,(3)$ Å
$b = 7.5284\,(6)$ Å
$c = 13.1036\,(11)$ Å
$\alpha = 87.836\,(7)^\circ$
$\beta = 86.108\,(6)^\circ$
$\gamma = 77.183\,(6)^\circ$
$V = 454.54\,(4)$ Å^3
$Z = 1$
$D_x = 1.536$ Mg m^{-3}
D_m not measured

Cell parameters from 35 reflections
$\theta = 10$–30°
$\mu = 1.207$ mm^{-1}
$T = 130\,(2)$ K
Parallelepiped
$0.38 \times 0.15 \times 0.12$ mm
Colorless

Data collection

Syntex $P2_1$ diffractometer
2θ–ω scans
Absorption correction:
*XABS*2 (Parkin, Moezzi & Hope, 1995)
$T_{\min} = 0.83$, $T_{\max} = 0.89$
1590 measured reflections
1590 independent reflections

1573 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 56.99^\circ$
$h = -1 \rightarrow 5$
$k = -7 \rightarrow 8$
$l = -14 \rightarrow 14$
2 standard reflections every 198 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.069$
$S = 1.072$
1590 reflections
254 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.1374P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.001$

$\Delta\rho_{\max} = 0.190$ e Å^{-3}
$\Delta\rho_{\min} = -0.193$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.010 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = 0.0 (2)

Table 1. *Selected geometric parameters (Å, °)*

O1—C1	1.390 (4)	O3′—C3′	1.426 (4)
O1—C4′	1.435 (4)	O5′—C5′	1.421 (4)
O2—C2	1.438 (4)	O5′—C1′	1.435 (3)
O3—C3	1.435 (3)	O6′—C6′	1.429 (4)
O4—C4	1.423 (4)	C1′—N1	1.430 (4)
O5—C1	1.429 (4)	C1′—C2′	1.519 (4)
O5—C5	1.442 (3)	C2′—C3′	1.521 (4)
O6—C6	1.434 (4)	C3′—C4′	1.534 (4)
C1—C2	1.523 (4)	C4′—C5′	1.518 (4)
C2—C3	1.523 (4)	C5′—C6′	1.514 (4)
C3—C4	1.512 (4)	O7—C7	1.242 (4)
C4—C5	1.528 (4)	N1—C7	1.380 (4)
C5—C6	1.506 (4)	N2—C7	1.329 (4)
O2′—C2′	1.427 (4)		
C1—O1—C4′	117.2 (2)	N1—C1′—C2′	111.0 (2)
C1—O5—C5	112.9 (2)	O5′—C1′—C2′	109.1 (2)
O1—C1—O5	106.9 (2)	O2′—C2′—C1′	108.3 (2)
O1—C1—C2	109.7 (2)	O2′—C2′—C3′	111.1 (2)
O5—C1—C2	109.3 (2)	C1′—C2′—C3′	112.4 (2)
O2—C2—C1	108.9 (2)	O3′—C3′—C2′	106.3 (2)
O2—C2—C3	110.3 (2)	O3′—C3′—C4′	112.7 (3)
C1—C2—C3	108.7 (2)	C2′—C3′—C4′	111.4 (2)
O3—C3—C4	110.8 (2)	O1—C4′—C5′	105.4 (2)
O3—C3—C2	113.0 (2)	O1—C4′—C3′	112.4 (2)
C4—C3—C2	110.4 (2)	C5′—C4′—C3′	110.1 (2)
O4—C4—C3	108.4 (3)	O5′—C5′—C6′	107.2 (2)
O4—C4—C5	110.3 (3)	O5′—C5′—C4′	110.6 (2)
C3—C4—C5	109.1 (2)	C6′—C5′—C4′	112.9 (2)
O5—C5—C6	106.4 (2)	O6′—C6′—C5′	109.4 (3)
O5—C5—C4	109.0 (2)	C7—N1—C1′	121.3 (3)
C6—C5—C4	113.1 (2)	O7—C7—N2	123.1 (3)
O6—C6—C5	109.4 (2)	O7—C7—N1	122.0 (3)
C5′—O5′—C1′	112.0 (2)	N2—C7—N1	114.8 (3)
N1—C1′—O5′	107.7 (2)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2—H2$\cdots$O3′$^{\rm i}$	0.940	1.830	2.759	169.34
O3—H3$\cdots$O6$^{\rm i}$	0.940	1.798	2.690	157.37
O4—H4$\cdots$O1$W^{\rm ii}$	0.940	1.712	2.644	170.91
O6—H6$\cdots$O3$^{\rm iii}$	0.940	1.742	2.676	172.20
O2′—H2′$\cdots$O2$^{\rm iii}$	0.940	1.845	2.757	162.83
O3′—H3′$\cdots$O5	0.940	1.873	2.744	153.06
O6′—H6′$\cdots$O2′$^{\rm iv}$	0.940	1.872	2.711	147.34
N1—H1N$\cdots$O7$^{\rm v}$	0.946	2.006	2.917	161.07
O1W—H1$W\cdots$O2W	1.044	1.784	2.817	169.57
O1W—H2$W\cdots$O2$W^{\rm vi}$	1.033	1.766	2.740	155.62
O2W—H3$W\cdots$O7$^{\rm v}$	0.878	1.981	2.829	162.03
O2W—H4$W\cdots$O6′	0.989	1.798	2.727	155.24

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

The handedness of the crystal was indicated by the Flack parameter (Flack, 1983) and also by the measurement of selected Bivjoet differences.

Data collection: *P3/P4-PC Diffractometer Program* (Siemens, 1991*a*). Cell refinement: *P3/P4-PC Diffractometer Program*. Data reduction: *XDISK* (Siemens, 1991*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1015). Services for accessing these data are described at the back of the journal.

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