

traction in all three unit-cell directions and a very even contraction of the four hydrogen bonds by nearly the same magnitude, as shown by the data in Table 5. However, in (I), the larger 2.3%, volume contraction corresponds to a 7% contraction in the weak hydrogen bonds and a less than 1% contraction in the other three bonds. [The apparently large contraction in O(1)—H(D) is due to the riding-motion foreshortening of the O(1)—H covalent bond in the room-temperature study.] Two of the unit-cell dimensions contract by 1.2 and 1.3%, but the third shows a slight expansion.

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## 1,2,4,6-Tetra-O-acetyl-3-O-(2,3,4,6-tetra-O-acetyl- $\beta$ -D-galactopyranosyl)- $\alpha$ -D-galactopyranose\*

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### Abstract

$C_{28}H_{38}O_{19}$  crystallizes in the space group  $P2_12_12_1$ , with  $a = 19.194$  (1),  $b = 14.719$  (1),  $c = 12.415$  (1) Å,  $Z = 4$ ,  $D_x = 1.285$  Mg m<sup>-3</sup>. Two sets of data (Mo and Cu radiations) and the derived results are analysed by normal probability plots. The structure was refined to  $R = 0.061$  ( $R_w = 0.074$ ). The anomeric parts have bond lengths of C(5')—O(5') = 1.439, O(5')—C(1') = 1.390, C(1')—O(1') = 1.435 and O(1')—C(11') = 1.331 Å for the  $\alpha$  moiety and, in the same order, 1.437, 1.409, 1.407 and 1.424 Å for the  $\beta$  moiety. The glycosidic link has an angle of 114.5°. The distortions of the usual

<sup>4</sup>C<sub>1</sub> chair conformation of both rings are analysed. The rings can be considered to be twisted by 75° from each other. The two galactoside substituents are attached to the rings with a difference in twist of 105°. The signs of angles which describe the relative configuration at every asymmetric C atom are defined.

### Introduction

The title compound is an intermediate in the synthesis of D-galactopyranosyl-D-galactose compounds (Chacón-Fuertes & Martín-Lomas, 1975). These compounds are being studied as part of a project on con-

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\* Crystal Structures of Analogues of D-Galactose. III.

formations of sugar materials with biological interest. Once the configuration of the present compound was clear from NMR spectroscopy, a detailed structural study was considered of interest.

### Structure solution

The crystal, experimental data and structure-refinement parameters are given in Table 1. The structure was solved from the Mo set, but the low ratio of observations to variables made the anisotropic refinement difficult, so a new set of data with Cu radiation

Table 1. *Crystal analysis parameters for C<sub>28</sub>H<sub>38</sub>O<sub>19</sub> at room temperature*

(a) Crystal data

Crystal size and habit: 0.20 × 0.43 × 0.17 mm; transparent prismatic

Symmetry: orthorhombic *mmm*, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>

Unit-cell determination: least-squares fit from 30 reflections; settings with  $\theta < 20^\circ$

Unit-cell dimensions:  $a = 19.194(1)$ ,  $b = 14.719(1)$ ,  $c = 12.415(1)$  Å (Mo  $K\alpha$ )

Packing:  $V = 3507.4(4)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_c = 1.285$  Mg m<sup>-3</sup>,  $M_r = 678.6$ ,  $F(000) = 1432$

(b) Experimental data collected with Mo and Cu radiations

Technique: four-circle Philips PW 1100 diffractometer; bisecting geometry

Orientation: (001) with  $\chi \sim -25^\circ$ ,  $\varphi \sim 95^\circ$ ; (*h**h*0) with  $\chi \sim -2^\circ$ ,  $\varphi \sim 185^\circ$

Monochromator: graphite, oriented; Mo  $K\alpha$  ( $\lambda = 0.71069$  Å) and Cu  $K\alpha$  ( $\lambda = 1.5418$  Å)

Collection mode:  $\omega$ - $2\theta$  up to  $\sin \theta/\lambda = 0.573$  Å<sup>-1</sup>;  $24.03^\circ$  (Mo) and  $62.06^\circ$  (Cu); scan width  $1.3^\circ$ ;

detector windows  $1^\circ \times 1^\circ$ ; 1 min/reflection

Number of independent data: equalized for both radiations to 3096 data

Number of observed data:  $I > 2\sigma(I)$ : 1524 (Mo) and 2626 (Cu)

Stability: two standards monitored every 90 min; no variation detected

Absorption  $\mu$ (mm<sup>-1</sup>): 0.103 (Mo), 0.907 (Cu); not corrected

(c) Solution and refinement

Solution mode: Mo data; *MULTAN 77* system with eight reflections per non-H atom

Least-squares refinement mode: on *F*'s; only observed used; eight blocks; mixed thermal mode with H atoms isotropic; final  $\langle \text{shift/error} \rangle = 0.73$  (Cu), 0.94 (Mo); *XRAY 70*

Parameters: number of variables = 586; degrees of freedom: 948 (Mo) and 2050 (Cu); ratio of freedom = 2.6 (Mo) and 4.6 (Cu)

Weighting scheme: empirical fit to give no trends in  $\langle w\Delta^2 \rangle$  vs  $\langle F_o \rangle$  or  $\langle \sin \theta/\lambda \rangle$

Thermal values: had to be fixed for six methyl H atoms; maximum values for the extremes of substituents:  $U_{11}[\text{O}(10)] = 0.307$ ,  $U_{22}[\text{C}(8')] = 0.202$ ,  $U_{33}[\text{C}(14')] = 0.155$  Å<sup>2</sup>

$\Delta F$  synthesis: to give all H atoms; final with residues less than  $0.45$  e Å<sup>-3</sup> (Cu) and  $0.26$  e Å<sup>-3</sup> (Mo)

$R$  and  $R_w$  (final): 0.067 and 0.085 (Mo), 0.061 and 0.074 (Cu)

Absolute configuration: not determined; the one displayed was already known from chemical synthesis; no anomalous-dispersion correction applied

Atomic scattering factors: from *International Tables for X-ray Crystallography* (1974)

was collected and the refinement continued. Two systems of programs were used: *MULTAN 77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977) and *XRAY 70* (Stewart, Kundell & Baldwin, 1970).\*

\* Lists of structure factors, fractional coordinates and thermal parameters for the Mo and Cu data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34830 (49 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Mo–Cu *weighted atomic coordinates* ( $\times 10^5$ ) *with their pooled e.s.d.'s multiplied by 1.375*

$$r = \left( \frac{1}{\sigma_{\text{Cu}}^2} r_{\text{Cu}} + \frac{1}{\sigma_{\text{Mo}}^2} r_{\text{Mo}} \right) \left/ \left( \frac{1}{\sigma_{\text{Cu}}^2} + \frac{1}{\sigma_{\text{Mo}}^2} \right) \right.;$$

$$\sigma(r) = 1.375 / (1/\sigma_{\text{Cu}}^2 + 1/\sigma_{\text{Mo}}^2)^{1/2}.$$

	x	y	z
C(1)	68535 (22)	30934 (31)	12683 (31)
C(2)	74791 (26)	25683 (32)	8452 (37)
O(2)	80813 (16)	30485 (27)	12252 (27)
C(3)	74895 (27)	26006 (34)	-3771 (37)
O(3)	80122 (20)	19612 (26)	-7316 (27)
C(4)	67907 (27)	23981 (30)	-8745 (33)
O(4)	66172 (21)	14559 (21)	-6955 (26)
C(5)	62372 (25)	29939 (32)	-3540 (33)
O(5)	62265 (14)	28106 (19)	7831 (23)
C(6)	55235 (27)	28139 (37)	-7903 (44)
O(6)	50378 (17)	35120 (27)	-4750 (27)
C(7)	49651 (30)	42153 (43)	-11528 (50)
O(7)	52896 (28)	42655 (38)	-19767 (43)
C(8)	44296 (48)	48590 (65)	-7748 (89)
O(8)	84917 (43)	18667 (57)	21265 (65)
C(9)	85418 (34)	26400 (61)	18923 (47)
O(9)	83068 (29)	28893 (39)	-20662 (42)
C(10)	90837 (38)	32899 (84)	22489 (59)
O(10)	63147 (59)	13033 (46)	-24132 (48)
C(11)	83838 (33)	21995 (46)	-16062 (43)
C(12)	89036 (69)	14664 (95)	-18884 (95)
C(13)	63754 (52)	9756 (46)	-15390 (61)
C(14)	61469 (74)	348 (52)	-11961 (104)
O(1)	68127 (14)	28815 (19)	23713 (20)
C(1')	62120 (23)	47353 (33)	42266 (33)
O(1')	56936 (14)	51131 (23)	35244 (25)
C(2')	67075 (22)	42125 (33)	35163 (33)
O(2')	70068 (14)	47789 (20)	26890 (23)
C(3')	63403 (22)	34329 (30)	29671 (33)
C(4')	60086 (21)	28383 (32)	38278 (33)
O(4')	65516 (14)	24541 (21)	44896 (26)
C(5')	55391 (23)	34008 (37)	45447 (37)
O(5')	59035 (15)	41700 (25)	49865 (23)
C(6')	52480 (32)	29034 (52)	55030 (44)
O(6')	48769 (19)	21075 (29)	51723 (30)
C(7')	42144 (40)	22362 (69)	48610 (50)
O(7')	39645 (27)	29802 (63)	48000 (56)
C(8')	38868 (57)	13599 (99)	45679 (82)
O(8')	78679 (21)	51643 (31)	38084 (35)
C(9')	76231 (23)	51844 (30)	29303 (43)
O(9')	54849 (32)	61967 (41)	47158 (44)
C(10')	79189 (46)	56420 (56)	19717 (65)
O(10')	63589 (30)	10722 (31)	38023 (52)
C(11')	53557 (34)	58464 (45)	38713 (48)
C(12')	48263 (67)	61519 (91)	31025 (82)
C(13')	66831 (32)	15489 (45)	43908 (54)
C(14')	72511 (42)	12839 (72)	51349 (104)

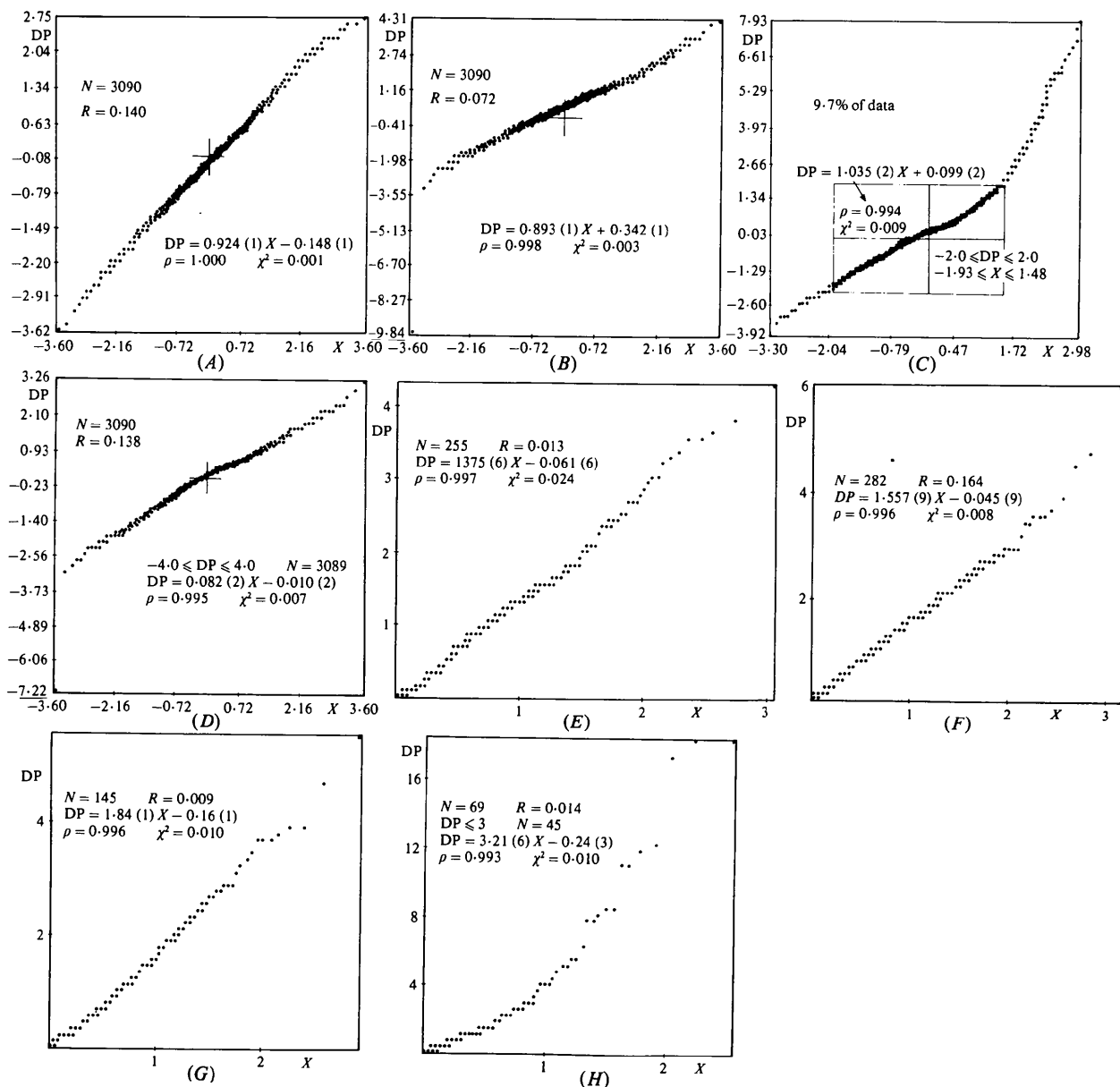


Fig. 1. Normal probability plots for the comparison of both data and results sets: (A)  $R(\text{Mo})$  plot:  $\text{DP} = (F_o - F_c)/(w)^{-1/2}$ ; (B)  $R(\text{Cu})$  plot:  $\text{DP} = (F_o - F_c)/(w)^{-1/2}$ ; (C)  $\text{Cu vs Mo}$  plot:  $\text{DP} = (F_{o1} - F_{o2})/(\sigma_1^2 + \sigma_2^2)^{1/2}$ ; (D)  $\text{Cu vs Mo}$  plot:  $\text{DP} = (F_{o1} - F_{o2})/(1/w_1 + 1/w_2)^{1/2}$ ; (E)  $\text{Cu vs Mo}$  plot:  $\text{DP} = |r_1 - r_2|/(\sigma_1^2 + \sigma_2^2)^{1/2}$  (H atoms included); (F)  $\text{Cu vs Mo}$  plot:  $\text{DP} = |U_1 - U_2|/(\sigma_1^2 + \sigma_2^2)^{1/2}$  (H atoms not included); (G)  $\text{Cu vs Mo}$  plot:  $\text{DP} = |d_1 - d_2|/(\sigma_1^2 + \sigma_2^2)^{1/2}$  (H atoms not included) ( $d$  up to 3.5 Å); (H)  $\alpha$  vs  $\beta$  plot:  $\text{DP} = |d_1 - d_2|/(\sigma_1^2 + \sigma_2^2)^{1/2}$  (H atoms not included) ( $d$  up to 3.5 Å) (weighted values and  $\sigma$ 's  $\times 1.375$ ).

## Results

Fig. 1 shows comparisons made from both sets of data by normal probability methods (Abrahams & Keve, 1971; De Camp, 1973). The full-normal probability comparisons of all data (A to D) show that the agreement obtained with the weighting schemes used is better for these than when the e.s.d.'s of the  $F_o$  are used. Only the final least-squares scale factors have been

introduced, and a non-zero intercept is present. From plot E the underestimation of the least-squares e.s.d.'s in fractional coordinates is taken as the slope and was used for the weighted averaging of them (Tables 2 and 3). Thermal factors and interatomic distances compare well for both data sets, while differences between the  $\alpha$  and  $\beta$  moieties, with weighted coordinates, emerge from the non-linear plot H, which is significant even with non-independent data.

Table 3. Mo—Cu weighted atomic coordinates ( $\times 10^4$ ) with their pooled e.s.d.'s multiplied by 1.375 for the hydrogen atoms with bond distances in Å

$$r = \left( \frac{1}{\sigma_{\text{Cu}}^2} r_{\text{Cu}} + \frac{1}{\sigma_{\text{Mo}}^2} r_{\text{Mo}} \right) \left( \frac{1}{\sigma_{\text{Cu}}^2} + \frac{1}{\sigma_{\text{Mo}}^2} \right)^{-1/2}$$

$$\sigma(r) = 1.375 / (1/\sigma_{\text{Cu}}^2 + 1/\sigma_{\text{Mo}}^2)^{1/2}$$

	<i>x</i>	<i>y</i>	<i>z</i>	C—H
H(1)	6925 (32)	3748 (50)	1164 (51)	0.98 (7)
H(2)	7479 (33)	1999 (47)	1151 (51)	0.92 (7)
H(3)	7666 (33)	3123 (50)	-588 (55)	0.88 (7)
H(4)	6796 (32)	2518 (43)	-1629 (52)	0.95 (7)
H(5)	6360 (34)	3597 (49)	-402 (52)	0.92 (7)
H(6a)	5375 (35)	2199 (50)	-584 (57)	0.98 (7)
H(6b)	5521 (35)	2734 (47)	-1577 (69)	0.98 (8)
H(8a)	4558 (62)	5219 (81)	-274 (104)	0.85 (13)
H(8b)	4030 (63)	4954 (73)	-991 (90)	0.82 (12)
H(8c)	4136 (58)	4700 (76)	-171 (100)	0.97 (12)
H(10a)	8941 (56)	3940 (76)	2569 (93)	1.07 (11)
H(10b)	9322 (55)	3442 (73)	1609 (102)	0.94 (12)
H(10c)	9441 (60)	3057 (71)	2572 (97)	0.87 (12)
H(12a)	9186 (76)	1028 (109)	-1620 (119)	0.91 (15)
H(12b)	8561 (73)	919 (109)	-2136 (127)	1.08 (15)
H(12c)	8892 (69)	1450 (99)	-2542 (133)	0.81 (17)
H(14a)	6613 (74)	-99 (94)	-572 (118)	1.20 (15)
H(14b)	6073 (72)	-71 (91)	-330 (123)	1.10 (15)
H(14c)	6388 (69)	-441 (101)	-1646 (113)	1.01 (14)
H(1')	6469 (36)	5279 (48)	4559 (57)	1.03 (7)
H(2')	7060 (39)	4005 (47)	3952 (60)	0.92 (7)
H(3')	5973 (37)	3648 (44)	2564 (56)	0.92 (7)
H(4')	5741 (33)	2363 (45)	3584 (54)	0.92 (7)
H(5')	5165 (38)	3816 (46)	4033 (59)	1.01 (9)
H(6'a)	4980 (43)	3294 (57)	5910 (73)	0.92 (9)
H(6'b)	5586 (46)	2726 (56)	5967 (73)	0.91 (9)
H(8'a)	3835 (64)	865 (83)	5074 (109)	0.97 (13)
H(8'b)	3841 (73)	1086 (93)	4106 (127)	0.71 (15)
H(8'c)	3469 (73)	1529 (108)	4727 (106)	0.86 (14)
H(10'a)	8389 (52)	5814 (59)	2060 (70)	0.94 (10)
H(10'b)	7724 (51)	5834 (65)	1461 (86)	0.79 (10)
H(10'c)	7946 (45)	5274 (63)	1547 (82)	0.76 (10)
H(12'a)	4580 (71)	5640 (95)	2609 (126)	1.08 (15)
H(12'b)	4465 (79)	6045 (99)	3417 (123)	0.81 (15)
H(12'c)	4680 (71)	6744 (98)	3104 (107)	0.92 (14)
H(14'a)	7574 (64)	1645 (79)	5634 (101)	1.03 (12)
H(14'b)	7529 (63)	893 (81)	4873 (94)	0.85 (12)
H(14'c)	7228 (83)	993 (116)	5952 (147)	1.10 (12)

With the weighted averaged coordinates (Tables 2 and 3), the bond distances (Table 4), the bond angles (Table 5) and the torsion angles (Table 6) have been calculated. In Tables 4 and 5 the points which differentiate the two moieties, in plot *H*, have been marked with an asterisk. The molecule with the atomic numbering appears in Fig. 2.

## Discussion

### A. Molecular constitution

#### A.1. The anomeric moieties and the glycosidic link.

The anomeric effect gives different values for the most favourable geometry in the  $\alpha$  and  $\beta$  anomers. From the

Table 4. Bond distances (Å) from the weighted coordinates, with the corresponding e.s.d.'s in parentheses

For completeness some values have been repeated. Asterisks mark the values flagged as different by the half-normal probability plot between moieties.

	$\alpha$ Moiety	$\beta$ Moiety	
Non-anomeric moiety			
C(1')—C(2')	1.508 (6)	C(1)—C(2)	1.522 (7)
C(2')—C(3')	1.509 (7)	C(2)—C(3)	1.518 (7)
C(3')—C(4')	1.521 (6)	C(3)—C(4)	1.506 (7)
C(4')—C(5')	1.513 (7)	C(4)—C(5)	1.522 (7)
C(2')—O(2')	1.442 (5)	C(2)—O(2)	1.435 (6)
*C(3')—O(1)	1.424 (5)	*C(3)—O(3)	1.444 (6)
C(4')—O(4')	1.443 (5)	C(4)—O(4)	1.444 (6)
C(5) substituents			
C(5')—C(6')	1.505 (8)	C(5)—C(6)	1.497 (7)
C(6')—O(6')	1.431 (8)	C(6)—O(6)	1.442 (7)
Acetal sequence			
C(5')—O(5')	1.439 (6)	C(5)—O(5)	1.437 (5)
O(5')—C(1')	1.390 (6)	O(5)—C(1)	1.409 (5)
*C(1')—O(1')	1.435 (6)	*C(1)—O(1)	1.407 (5)
*O(1')—C(11')	1.331 (8)	*O(1)—C(3')	1.424 (5)
Glycosidic link			
C(3')—O(1)	1.424 (5)	C(1)—O(1)	1.407 (5)
O(1)—C(1)	1.407 (5)	O(1)—C(3')	1.424 (5)
Acetyl groups			
O(2')—C(9')	1.358 (6)	O(2)—C(9)	1.352 (8)
*O(1')—C(11')	1.331 (8)	*O(3)—C(11)	1.346 (7)
O(4')—C(13')	1.362 (8)	O(4)—C(13)	1.346 (8)
O(6')—C(7')	1.343 (9)	O(6)—C(7)	1.341 (7)
C(9')—O(8')	1.188 (7)	C(9)—O(8)	1.179 (12)
C(11')—O(9')	1.194 (9)	C(11)—O(9)	1.174 (9)
C(13')—O(10')	1.189 (9)	C(13)—O(10)	1.193 (10)
C(7')—O(7')	1.198 (13)	C(7)—O(7)	1.200 (8)
C(9')—C(10')	1.481 (9)	C(9)—C(10)	1.481 (13)
C(11')—C(12')	1.465 (14)	C(11)—C(12)	1.511 (15)
C(13')—C(14')	1.481 (12)	C(13)—C(14)	1.514 (12)
C(7')—C(8')	1.480 (16)	C(7)—C(8)	1.475 (12)

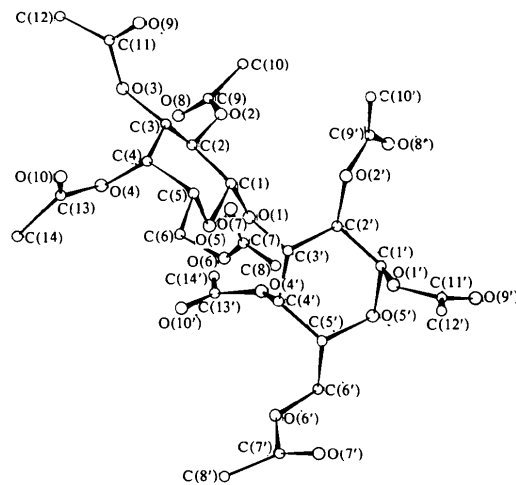


Fig. 2. A perspective view of the molecule with the atomic numbering.

Table 5. Bond angles ( $^{\circ}$ ) from the weighted coordinates, with the corresponding e.s.d.'s in parentheses

For completeness some values have been repeated. Asterisks mark the values flagged as different by the half-normal probability plot between moieties.

$\alpha$ Moiety		$\beta$ Moiety	
Non-anomeric moiety			
C(1')-C(2')-C(3')	111.0 (4)	C(1)-C(2)-C(3)	109.9 (4)
*C(2')-C(3')-C(4')	108.4 (4)	*C(2)-C(3)-C(4)	113.1 (4)
C(3')-C(4')-C(5')	110.4 (4)	C(3)-C(4)-C(5)	109.5 (4)
*C(1')-C(2')-O(2')	111.9 (4)	*C(1)-C(2)-O(2)	105.8 (4)
C(3')-C(2')-O(2')	107.7 (4)	C(3)-C(2)-O(2)	107.6 (4)
*C(2')-C(3')-O(1)	111.8 (4)	*C(2)-C(3)-O(3)	107.1 (4)
*C(4')-C(3')-O(1)	107.7 (3)	*C(4)-C(3)-O(3)	111.4 (4)
C(3')-C(4')-O(4')	108.9 (4)	C(3)-C(4)-O(4)	109.4 (4)
C(5')-C(4')-O(4')	108.0 (4)	C(5)-C(4)-O(4)	109.1 (4)
C(5) substituents			
*C(4')-C(5')-C(6')	114.8 (4)	*C(4)-C(5)-C(6)	112.5 (4)
C(6')-O(6')-C(7')	116.0 (5)	C(6)-O(6)-C(7)	116.6 (4)
*C(6')-C(5')-O(5')	105.2 (4)	*C(6)-C(5)-O(5)	108.0 (4)
C(5')-C(6')-O(6')	110.9 (5)	C(5)-C(6)-O(6)	111.6 (4)
*C(4')-C(5')-O(5')	111.4 (4)	*C(4)-C(5)-O(5)	108.6 (4)
Acetal sequence			
*C(5')-O(5')-C(1')	114.8 (4)	*C(5)-O(5)-C(1)	110.6 (3)
*O(5')-C(1')-O(1')	110.4 (4)	*O(5)-C(1)-O(1)	107.7 (3)
C(1')-O(1')-C(11')	117.1 (4)	C(1)-O(1)-C(3')	114.5 (3)
*C(2')-C(1')-O(5')	111.1 (4)	*C(2)-C(1)-O(5)	112.1 (4)
C(2')-C(1')-O(1')	106.3 (4)	C(2)-C(1)-O(1)	105.5 (4)
Glycosidic link			
C(1)-O(1)-C(3')	114.5 (3)	C(1)-O(1)-C(3')	114.5 (3)
C(2')-C(3')-C(4')	108.4 (4)	C(2)-C(1)-O(5)	112.1 (4)
C(2')-C(3')-O(1)	111.8 (4)	C(2)-C(1)-O(1)	105.5 (4)
C(4')-C(3')-O(1)	107.7 (3)	O(5)-C(1)-O(1)	107.7 (3)
Acetyl groups			
O(2')-C(9')-C(10')	110.9 (5)	O(2)-C(9)-C(10)	110.8 (6)
O(1')-C(11')-C(12')	112.1 (7)	O(3)-C(11)-C(12)	110.6 (7)
O(4')-C(13')-C(14')	109.8 (6)	O(4)-C(13)-C(14)	111.2 (7)
O(6')-C(7')-C(8')	110.5 (7)	O(6)-C(7)-C(8)	111.6 (6)
O(2')-C(9')-O(8')	122.4 (5)	O(2)-C(9)-O(8)	121.8 (7)
O(1')-C(11')-O(9')	122.2 (6)	O(3)-C(11)-O(9)	123.4 (6)
O(4')-C(13')-O(10')	122.4 (6)	O(4)-C(13)-O(10)	121.9 (8)
O(6')-C(7')-O(7')	121.8 (8)	O(6)-C(7)-O(7)	121.9 (6)
C(10')-C(9')-C(8')	126.7 (6)	C(10)-C(9)-O(8)	127.4 (8)
C(12')-C(11')-O(9')	125.7 (8)	C(12)-C(11)-O(9)	126.0 (8)
C(14')-C(13')-O(10')	127.8 (7)	C(14)-C(13)-O(10)	126.7 (9)
C(8')-C(7')-O(7')	127.7 (9)	C(8)-C(7)-O(7)	126.4 (7)
*C(2')-O(2')-C(9')	116.4 (3)	*C(2)-O(2)-C(9)	120.6 (4)
C(1')-O(1')-C(11')	117.1 (4)	C(3)-O(3)-C(11)	116.4 (4)
C(4')-O(4')-C(13')	117.8 (4)	C(4)-O(4)-C(13)	117.7 (5)
C(6')-O(6')-C(7')	116.0 (5)	C(6)-O(6)-C(7)	116.6 (4)

values found in the present compound we can differentiate the two moieties, but they also present some differences from the characteristic rules tabulated from the literature (Table 7):

-  $\beta$  Moiety: C(5)-O(5) and O(1)-C(3') are alike, but different from O(5)-C(1) which is equal to C(1)-O(1) and lies between the values for the C-O ring bond and for the acetyl groups. The bond angles follow the rule as well as the torsion angles, and are the values considered as preferred for  $\beta$ -pyranoses.

-  $\alpha$  Moiety: The two inner bonds are not shorter than the outer ones, nor are they alike, although the torsion angle is  $83^{\circ}$ . The bond angles again follow the rule, but

Table 6. Some torsion angles ( $^{\circ}$ ) from the weighted coordinates

The corresponding e.s.d.'s are given in parentheses.

$\alpha$ Moiety		$\beta$ Moiety	
Pyranose groups			
O(5')-C(1')-C(2')-C(3')	56.9 (5)	O(5)-C(1)-C(2)-C(3)	52.2 (5)
O(5')-C(1')-C(2')-O(2')	177.2 (3)	O(5)-C(1)-C(2)-O(2)	168.0 (3)
H(1')-C(1')-C(2')-H(2')	62 (6)	H(1)-C(1)-C(2)-H(2)	161 (6)
C(1')-C(2')-C(3')-C(4')	-56.1 (5)	C(1)-C(2)-C(3)-C(4)	-46.7 (5)
C(1')-C(2')-C(3')-O(1)	-174.6 (3)	C(1)-C(2)-C(3)-O(3)	-169.7 (4)
H(2')-C(2')-C(3')-H(3')	177 (6)	H(2)-C(2)-C(3)-H(3)	-156 (6)
C(2')-C(3')-C(4')-C(5')	54.8 (5)	C(2)-C(3)-C(4)-C(5)	51.0 (5)
C(2')-C(3')-C(4')-O(4')	-63.7 (4)	C(2)-C(3)-C(4)-O(4)	-68.6 (5)
H(3')-C(3')-C(4')-H(4')	58 (6)	H(3)-C(3)-C(4)-H(4)	46 (6)
C(3')-C(4')-C(5')-O(5')	53.8 (5)	C(3)-C(4)-C(5)-O(5)	-59.1 (5)
C(3')-C(4')-C(5')-C(6')	-173.3 (4)	C(3)-C(4)-C(5)-C(6)	-178.6 (4)
H(4')-C(4')-C(5')-H(5')	-63 (6)	H(4)-C(4)-C(5)-H(5)	-66 (6)
C(4')-C(5')-O(5')-C(1')	55.1 (5)	C(4)-C(5)-O(5)-C(1)	66.3 (4)
C(5')-O(5')-C(1')-C(2')	-56.4 (5)	C(5)-O(5)-C(1)-C(2)	-63.4 (4)
C(5')-O(5')-C(1')-O(1')	61.2 (5)	C(5)-O(5)-C(1)-O(1)	-179.1 (3)
C(5')-O(5')-C(1')-H(1')	180 (4)	C(5)-O(5)-C(1)-H(1)	60 (4)
Glycosidic link			
C(1)-O(1)-C(3')-C(4')	144.9 (3)	O(5)-C(1)-O(1)-C(3')	-71.2 (4)
C(1)-O(1)-C(3')-C(2')	-96.2 (4)	C(2)-C(1)-O(1)-C(3')	168.9 (3)
C(1)-O(1)-C(3')-H(3')	29 (5)	H(1)-C(1)-O(1)-C(3')	50 (4)
Substituents			
C(3')-C(2')-O(2')-C(9')	-145.7 (4)	C(3)-C(2)-O(2)-C(9)	-124.4 (5)
O(5')-C(1')-O(1')-C(11')	83.1 (5)	C(4)-C(3)-O(3)-C(11)	92.1 (5)
C(5')-C(4')-O(4')-C(13')	131.2 (5)	C(5)-C(4)-O(4)-C(13)	106.7 (5)
O(5')-C(5')-C(6')-O(6')	-179.2 (4)	O(5)-C(5)-C(6)-O(6)	73.2 (5)
C(5')-C(6')-O(6')-C(7')	-83.1 (7)	C(5)-C(6)-O(6)-C(7)	91.5 (5)

here we can distinguish two halves, the first following the anomeric character and the second C(1')-O(1')-C(11') behaving like the acetyl substituents.

- The glycosidic link could modify the  $\beta$  anomeric moiety, its angle at O(1) being slightly less than for 1 $\rightarrow$ X linkages (Kanters, Roelofsen, Doesburg & Koops, 1976).

A.2. The non-anomeric parts. Ranges of values characterizing the molecule are presented in Table 8. The C-C bonds for both galactoside substituents, although shorter than those in the rings, can be included in their range. The smallest is C(5)-C(6) for the  $\beta$  ring. The main differences in CCC angles between rings are in C(2)-C(3)-C(4) vs C(2')-C(3')-C(4'), which is excluded from the first range, and C(4)-C(5)-C(6) vs C(4')-C(5')-C(6'). The total spread of  $11.3\sigma_p$  is quite high.

The C-O bonds have their smallest distances at C(6')-O(6'), C(1)-O(5), C(1')-O(5'), C(1)-O(1) and C(3')-O(1). All are included in the anomeric parts, but have to be left out of the range to give an acceptable spread for it. That gives another difference between  $\alpha$  and  $\beta$  moieties, as C(1')-O(1') is within the current C-O range while C(1)-O(1) is not. The usual differences (Kanters, Roelofsen, Doesburg & Koops, 1976) between the exocyclic CCO angles at each ring atom and the spread of values is usually attributed to steric interactions between substituents. Only at C(4) and C(4') are the angles equal, and the differences reverse the sense when going from the  $\alpha$  to the  $\beta$  ring. In

Table 7. *The anomeric parts as described in the literature (Å and degrees)*

Bond or angle	Methyl- $\alpha$ -pyranoses <sup>(a)</sup>	Theory <sup>(a)</sup>	Other $\alpha$ -pyranoses <sup>(b)</sup>	$\beta$ -Linkages <sup>(a)</sup>	Theory <sup>(a)</sup>	Other $\beta$ -pyranoses <sup>(b)</sup>
C(5)—O(5)	1.420–1.450	1.444	1.426–1.443	1.436–1.448	1.434	1.427–1.439
O(5)—C(1)	1.410–1.420	1.423	1.412–1.445	1.425–1.427	1.425	1.413–1.437
C(1)—O(1)	1.390–1.410	1.423	1.396–1.400	1.384–1.397	1.400	1.374–1.396
O(1)—C	1.420–1.440	1.444	1.431–1.442	1.436–1.446	1.443	1.416–1.442
C(5)—O(5)—C(1)	112.0–114.0	115.9	112.4–114.2	111.2–112.2	115.8	111.2–112.6
O(5)—C(1)—O(1)	111.0–113.0	113.9	110.9–112.2	106.9–107.7	110.9	107.3–108.6
C(1)—O(1)—C	113.0–113.5	115.9	111.5–113.6	115.8–117.3	116.1	113.2–115.8
O(5)—C(1)—O(1)—C	61–71	60	64	–71, –94	60	–70.8, –82.3

References: (a) Jeffrey, Pople, Binkley & Vishveshwara (1978); (b) Kanters, Roelofsen, Doesburg & Koops (1976), Ohanessian & Gillier-Pandraud (1976), Takagi & Jeffrey (1977, 1979), Sheldrick (1976), Lindberg (1976).

Table 8. *Ranges of values found in the present work and in the literature*

For each kind of bond (Å) or angle (°) the sequence is: type of bond length or angle [minimum value (e.s.d.), maximum value (e.s.d.)]; number of data; range in terms of their pooled e.s.d.'s ( $\sigma_p^2 = \sigma_1^2 + \sigma_2^2$ ); average of the data, when considered possible; dispersion, measured as the range/(number of data)<sup>1/2</sup>. For the literature values the last three are substituted by a reference letter.

Bond or angle	This work range	Literature range
C—C (rings)	[1.506 (7), 1.522 (7)]; 8; 1.6 $\sigma_p$ ; (1.515, 0.006)	[1.494 (9), 1.538 (15)]; 36(b)
C—C (all)	[1.497 (7), 1.522 (7)]; 10; 2.5 $\sigma_p$ ; (1.512, 0.008)	[1.494 (9), 1.538 (15)]; 45(b)
C—C	[1.435 (6), 1.444 (6)]; 5; 1.1 $\sigma_p$ ; (1.442, 0.004)	[1.414 (5), 1.494 (7)]; 27(b)
C—O [and C(6)—O(6)]	[1.431 (8), 1.444 (6)]; 10; 1.3 $\sigma_p$ ; (1.439, 0.004)	[1.415 (3), 1.449 (7)]; 36(b)
C—C—C	[109.5 (4), 110.0 (4)]; 4; 2.7 $\sigma_p$ ; (110.2, 0.8)	[107.8 (8), 112.6 (2)]; 27(b)
C—C—C [and at C(5)]	[108.4 (4), 114.8 (4)]; 8; 11.3 $\sigma_p$ ;	[107.8 (8), 114.7 (4)]; 36(b)
C—C—O (no acetal)	[105.8 (4), 111.9 (4)]; 12; 10.7 $\sigma_p$ ;	[106.4 (3), 114.6 (3)]; 54(b)
C—C—O (no acetal- $\alpha$ )	[107.7 (4), 111.9 (4)]; 6; 7.6 $\sigma_p$ ;	[106.4 (3), 114.6 (3)]; 30(b)
C—C—O (no acetal- $\beta$ )	[105.8 (4), 109.1 (4)]; 6; 5.8 $\sigma_p$ ;	[107.7 (7), 112.1 (2)]; 24(b)
O—C (acetyl)	[1.331 (8), 1.362 (8)]; 8; 2.7 $\sigma_p$ ; (1.348, 0.011)	[1.323 (4), 1.364 (7)]; 18(c)
O=C (acetyl)	[1.174 (9), 1.200 (8)]; 8; 2.0 $\sigma_p$ ; (1.190, 0.009)	[1.135 (21), 1.225 (7)]; 18(c)
C—C (acetyl)	[1.465 (14), 1.514 (12)]; 8; 2.7 $\sigma_p$ ; (1.485, 0.017)	[1.434 (11), 1.509 (11)]; 18(c)
O—C—C (acetyl)	[109.8 (6), 112.1 (7)]; 8; 2.5 $\sigma_p$ ; (110.9, 1.0)	[110.1 (5), 113.1 (6)]; 18(c)
O—C—O (acetyl)	[121.8 (8), 123.4 (6)]; 8; 1.6 $\sigma_p$ ; (122.3, 0.6)	[121.3 (6), 125.1 (13)]; 18(c)
C—C=O (acetyl)	[125.7 (8), 127.8 (7)]; 8; 2.0 $\sigma_p$ ; (126.8, 0.7)	[124.5 (5), 128.0 (16)]; 18(c)
C—O—C (acetyl)	[116.0 (5), 117.8 (4)]; 8; 2.8 $\sigma_p$ ; (116.8, 0.7)	[115.4 (4), 119.6 (9)]; 18(c)
C—H (rings)	[0.88 (7), 1.03 (7)]; 14; 1.4 $\sigma_p$ ; (0.94, 0.04)	—
C—H (methyl)	[0.71 (15), 1.19 (15)]; 24; 2.4 $\sigma_p$ ; (0.93, 0.10)	—
C—C—H (rings)	[109. (4), 116. (4)]; 22; 1.2 $\sigma_p$ ; (105.6, 1.5)	—
O—C—H (rings)	[104. (4), 114. (4)]; 16; 1.8 $\sigma_p$ ; (109.3, 2.5)	—
C—C—H (methyl)	[95. (10), 133. (7)]; 24; 3.1 $\sigma_p$ ; (115., 8.)	—
H—C—H (methyl)	[60. (12), 122. (12)]; 26; 2.8 $\sigma_p$ ; (92., 12.)	—

References: (b) Kanters, Roelofsen, Doesburg & Koops (1976), Ohanessian & Gillier-Pandraud (1976), Takagi & Jeffrey (1977, 1979), Sheldrick (1976), Lindberg (1976). (c) Heitmann, Richards & Schroeder (1974), Foces-Foces, Cano & Garcia-Blanco (1976a,b,c), Foces-Foces, Alemany, Bernabe & Martin-Lomas (1979).

the  $\alpha$  ring C(6')—C(5')—O(5') is smaller than the ranged values.

**A.3. The acetyl substituents.** The only bond distance different from the half-normal probability comparison between rings is O(3)—C(11) vs O(1')—C(11'), but both can be included in the range presented in Table 8. The O—C and C—C bonds are different from the corresponding ring bonds, and it seems that O—C and C—C are slightly shorter at C(6) and at C(6'). The C(2)—O(2)—C(9) angle departs from the ranged values.

**A.4. The hydrogen atoms.** Those attached to the ring are better defined than the rest. The ranges defining their geometry are also given in Table 8.

## B. Molecular conformation

**B.1. The rings.** Both rings have the typical <sup>4</sup>C<sub>1</sub> chair conformation usual for *D* series, and the dispersion of values in the  $\beta$  ring (27.6 $\sigma_p$ ) is higher than for the  $\alpha$  ring (4.5 $\sigma_p$ ). The  $\alpha$  ring is flattened at O(5')—C(1')—C(2')—C(3') and more puckered at C(3')—C(4')—C(5')—

O(5'), while for the  $\beta$  ring these characteristics are at C(1)–C(2)–C(3)–C(4) and C(4)–C(5)–O(5)–C(1) respectively.

For these, if we apply the Cano, Foces-Foces & García-Blanco (1979) description, we have (for the torsion angles, in degrees):  $\varphi_{j\alpha}(-1)^j = 55.6 + 1.6 \cos(10.6 + 60j)$  and  $\varphi_{j\beta}(-1)^j = 56.5 + 9.9 \cos(110.5 + 60j)$ . The agreements, measured by  $[\sum(\varphi_o - \varphi_c)^2/6]^{1/2}$ , are 0.25 and 0.64 respectively. For some galactoside compounds [references (b) in Table 8]  $\tau m$  ranges from 53 to 59° (average 57°),  $q$  ranges from

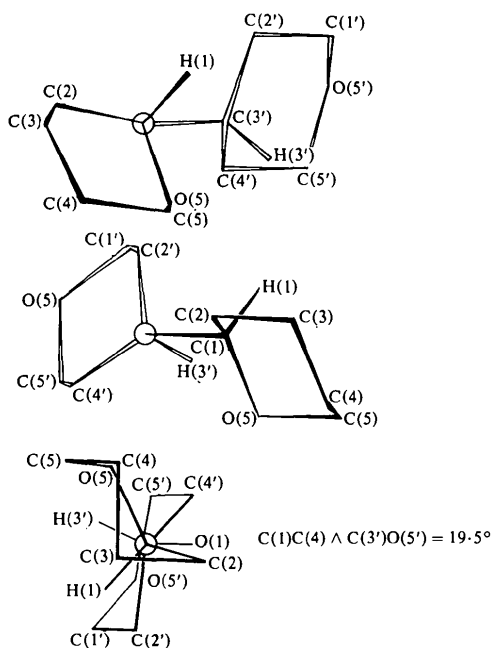


Fig. 3. The relative twist between both rings through the glycosidic link.

1 to 12° (average 6°) and  $\sum$  is grouped around either 108 or 236°. From these values, several facts can be noticed:

– The analogous chair character for both rings (55.6 vs 56.5°).

– The higher amount of boat–twist puckering for the  $\beta$  ring (1.6 vs 9.9°).

– The value of  $\sum$  for the  $\alpha$  ring, different from the values in the literature.

– The different type of quasi-symmetry present. This is given for the common elements to the  $3m$  group, in the perfect chair, and the 222 group at  $\sum = 0^\circ$  for  $\alpha$  and 222 at  $\sum = 240^\circ$  for  $\beta$ , leaving a quasi-axis through C(1')–C(2'), C(4')–C(5') and another through C(2)–C(3), C(5)–O(5).

**B.2. The glycosidic link.** When going from the  $\beta$  to the  $\alpha$  ring in the C(1)–O(1)–C(3') sense the twist around C(1)–O(1) is found (Cano, Foces-Foces & García-Blanco, 1979) to be 49.2°, while around O(1)–C(3') it is 25.9°. For estimating the relative twist of one ring with respect to the other, several parameters are used in the literature (Kanters, Gaykema & Roelofsen, 1978; Mo & Jensen, 1978). If we take as reference the conformation with H(1)–C(1)–O(1)–C(3')–H(3') coplanar, we can use as a measure of that relative twist the sum of the above-mentioned two partial twists (Fig. 3) to give 75.1°. This way of measuring the relative twist gives values near to those given by the second-mentioned authors.

**B.3. The substituents.** The substituent chains are characterized in Table 9. For the acetyl groups the torsion angles give conformations with COCO  $\sim 0^\circ$  for all groups. Around the C(5')–C(6') and C(5)–C(6) galactoside bonds the twists are 60.2 and  $-45.4^\circ$  respectively. So there is a relative intrinsic rotation of about  $\pm 105^\circ$  one from the other.

Table 9. Conformational characteristics and configurational angles for the ring substituents

(a) Conformational characteristics of the ring substituents, with respect to the previous/next ring atom; within parentheses are values near 90°

$\alpha$ Angle	Conformation	$\beta$ Angle	Conformation
C(1')/C(3')–C(2')–O(2')–C(9')	(+ac), –ac	C(1)/C(3)–C(2)–O(2)–C(9)	+ac, –ac
C(2')/C(4')–C(3')–O(1)–C(1)	(–ac), +ac	C(2)/C(4)–C(3)–O(3)–C(11)	–ac, (+ac)
C(3')/C(5')–C(4')–O(4')–C(13')	(–ac), +ac	C(3)/C(5)–C(4)–O(4)–C(13)	–ac, (+ac)
O(5')/C(2')–C(1')–O(1')–C(11')	(+sc), –ap	O(5)/C(2)–C(1)–O(1)–C(3')	–sc, +ap
C(4')/O(5')–C(5')–C(6')–O(6')	–gauche, –trans	C(4)/O(5)–C(5)–C(6)–O(6)	–trans, +gauche

(b) Configurational angles (°) for the ring substituents

Configuration angle	Substituent angle	Ring angle	$\beta$ Value	$\alpha$ Value
$\rho[C(5)–O(5)–C(1)–O(1)] = \tau[C(5)–O(5)–C(1)–O(1)]$	$\tau[C(5)–O(5)–C(1)–O(1)]$	$\tau[C(5)–O(5)–C(1)–C(2)]$	$-115.7 = +117.6$	
$\rho[O(5)–C(1)–C(2)–O(2)] = \tau[O(5)–C(1)–C(2)–O(2)]$	$\tau[O(5)–C(1)–C(2)–O(2)]$	$\tau[O(5)–C(1)–C(2)–C(3)]$	$+115.8 = +120.3$	
$\rho[C(1)–C(2)–C(3)–O(3)] = \tau[C(1)–C(2)–C(3)–O(3)]$	$\tau[C(1)–C(2)–C(3)–O(3)]$	$\tau[C(1)–C(2)–C(3)–C(4)]$	$-123.0 = -118.5$	
$\rho[C(2)–C(3)–C(4)–O(4)] = \tau[C(2)–C(3)–C(4)–O(4)]$	$\tau[C(2)–C(3)–C(4)–O(4)]$	$\tau[C(2)–C(3)–C(4)–C(5)]$	$-119.6 = -118.5$	
$\rho[C(3)–C(4)–C(5)–C(6)] = \tau[C(3)–C(4)–C(5)–C(6)]$	$\tau[C(3)–C(4)–C(5)–C(6)]$	$\tau[C(3)–C(4)–C(5)–O(5)]$	$-119.5 = -119.9$	

### C. Molecular configuration

The absolute molecular configuration has not been determined, but is known from chemical synthesis to be the one displayed. We can define the configurational angles by the exocyclic substitutional torsion angles reduced by the corresponding endocyclic one, to give values around  $\pm 120^\circ$  (Table 9) and going around both isomers in the same sense. The sign of these configurational angles gives the relative configuration, not only between rings, but also between atoms within the same ring. Whenever a different sign appears a different relative configuration is present.

### D. Molecular packing

The packing is due to van der Waals forces. The packing ratio, volume/atoms in the unit cell, is  $18.7 \text{ \AA}^3$ . We have examined some crystal structures with and without hydrogen bonds and have found some differences: with hydrogen bonds the ratio ranges from 15.4 to  $17.8 \text{ \AA}^3$  (five compounds, average  $16.4 \text{ \AA}^3$ ) while when hydrogen bonds are absent the ratio ranges from 17.5 to  $19.2 \text{ \AA}^3$  (fifteen compounds, average  $18.2 \text{ \AA}^3$ ).

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## 1,3,4,6-Tetra-O-acetyl-2-(N-acetylacetamido)-2-deoxy- $\beta$ -D-galactopyranose

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#### Abstract

C<sub>18</sub>H<sub>25</sub>NO<sub>11</sub>,  $M_r = 431.44$ , orthorhombic,  $P2_12_12_1$ ,  $a = 18.720$  (6),  $b = 14.070$  (4),  $c = 8.248$  (2) Å,  $Z = 4$ ,

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$U = 2172.44 \text{ \AA}^3$ ,  $D_m = 1.310$ ,  $D_c = 1.319 \text{ Mg m}^{-3}$ , Cu  $K\alpha$  radiation ( $\mu = 2.323 \text{ mm}^{-1}$ ). The structure was refined to  $R = 0.069$ . The sugar ring exhibits a  ${}^4C_1$  chair conformation. Bond lengths and angles are

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