$$C_{28}H_{38}O_{19}$$

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 $\pm 120°$ (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 Å^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 Å^3 (five compounds, average 16.4 Å^3) while when hydrogen bonds are absent the ratio ranges from 17.5 to 19.2 Å^3 (fifteen compounds, average 18.2 Å^3).

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1,3,4,6-Tetra-O-acetyl-2-(N-acetylacetamido)-2-deoxy-β-D-galactopyranose

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Abstract

$C_{18}H_{25}NO_{11}$, $M_r = 431.44$, orthorhombic, $P2_12_12_1$, $a = 18.720$ (6), $b = 14.070$ (4), $c = 8.248$ (2) Å, $Z = 4$, $U = 2172.44 \text{ Å}^3$, $D_m = 1.310$, $D_c = 1.319 \text{ Mg m}^{-3}$, 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

comparable to those in other hexoses. Owing to the absence of free hydroxyl groups, O—H$\cdots$O hydrogen bonds are not possible and the molecules are joined by van der Waals interactions only.

Introduction

An X-ray analysis of a series of peracetylated unsaturated (1,2- and 2,3-) amino sugars has been carried out. The crystal structures of 1,4,6-tri-O-acet-yl-2-(N-acetylacetamido)-2,3-dideoxy-α-D-*threo*-hex-2-enopyranose (Kojić-Prodić, Rogić & Ružić-Toroš, 1976) and its *erythro* isomer (Ružić-Toroš, Rogić & Kojić-Prodić, 1980) confirmed the configuration previously assigned on the basis of chemical reactions and ^{1}H NMR data (Pravdić, Židovec & Fletcher, 1973). Two isomeric compounds from the same series, earlier described as 1,4,6-tri-O-acetyl-2-(N-acetylacetamido)-2,3-dideoxy-β-D-*threo*-hex-2-enopyranose and its *erythro* isomer (Pravdić, Židovec & Fletcher, 1973), were, on the basis of X-ray structure determination, found to be 3,4,6-tri-O-acetyl-2-(N-acetylacetamido)-1,2-dideoxy-D-*xylo*-hex-1-enopyranose and its *ribo* isomer (Rogić, Ružić-Toroš, Kojić-Prodić & Pravdić, 1977). In order to complete the structure investigations on this class of sugars the crystal structures of 3,4,6-tri-O-acetyl-2-(N-acetylacetamido)-1,2-dideoxy-D-*arabino*-hex-1-enopyranose and its *lyxo* isomer have been studied. The configuration of the D-*arabino* isomer has already been confirmed (Kojić-Prodić & Rogić, 1978) but the other compound is found to be 1,3,4,6-tetra-O-acetyl-2-(N-acetylacetamido)-2-deoxy-β-D-galacto-pyranose and its structure determination is described in this paper.

Intensities were collected on a Philips PW 1100 computer-controlled diffractometer in the ω-scan mode [scan width = 1·2° (θ), scan speed = 0·04° (θ) s^{-1}] with graphite-monochromated Cu $K\alpha$ radiation. The intensities of 2104 observed reflexions [$I > 2\sigma(I)$] were measured and used in the calculations. The data were corrected for Lorentz and polarization effects. Overall temperature (B = 4·01 Å^2) and scale factors were determined (Wilson, 1942) and used to compute normalized amplitudes by the *NORMAL* routine included in *MULTAN*. The structure was solved with *MULTAN* 77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977) on the basis of 300 reflexions with |E| > 1·38. The E map corresponding to the solution with the best figure of merit revealed the positions of all non-hydrogen atoms. Refinement was by full-matrix least squares minimizing $\sum w||F_o| - |F_c||^2$. A weighting scheme of type 3 from XRAY (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) was used. The weights were assigned as: $w = w_1 w_2$, where $w_1 = 1$ for $|F_o| \leq 25$ and $w_1 = 25/|F_o|$ for $|F_o| > 25$; $w_2 = 1$ for $\sin \theta \geq 0·5$ and $w_2 = (\sin \theta)/0·5$ for $\sin \theta < 0·5$. Aniso-

Table 1. *Final atomic coordinates for non-hydrogen atoms* ($\times 10^4$)

	x	y	z
O(0)	1249 (2)	2637 (2)	10881 (4)
O(1)	1561 (2)	1290 (3)	9619 (5)
O(2)	2289 (4)	1587 (7)	7553 (11)
O(3)	790 (2)	2655 (2)	6027 (5)
O(4)	−360 (2)	344 (3)	6165 (5)
O(5)	−769 (1)	2460 (2)	8974 (5)
O(6)	−1164 (2)	3861 (3)	8083 (6)
O(7)	−205 (2)	2851 (2)	11961 (4)
O(8)	−855 (4)	4092 (4)	12713 (9)
O(9)	1804 (2)	4389 (3)	12121 (7)
O(10)	1609 (4)	5658 (4)	3544 (11)
N	350 (2)	1416 (3)	7463 (5)
C(1)	1197 (2)	2156 (3)	9384 (6)
C(2)	430 (2)	1945 (3)	8991 (6)
C(3)	−50 (2)	2830 (3)	9073 (6)
C(4)	79 (2)	3383 (3)	10609 (6)
C(5)	882 (2)	3526 (3)	10829 (6)
C(6)	1071 (3)	4078 (4)	12331 (9)
C(7)	2120 (4)	1069 (5)	8578 (10)
C(8)	2427 (4)	121 (5)	9038 (12)
C(9)	626 (3)	1826 (3)	6020 (6)
C(10)	762 (4)	1189 (4)	4572 (8)
C(11)	−91 (3)	600 (3)	7435 (7)
C(12)	−215 (3)	78 (4)	8985 (8)
C(13)	−1282 (3)	3043 (4)	8433 (7)
C(14)	−1992 (3)	2550 (5)	8362 (11)
C(15)	−716 (3)	3272 (4)	12868 (7)
C(16)	−1034 (3)	2629 (6)	14089 (8)
C(17)	1996 (4)	5194 (5)	2724 (10)
C(18)	2757 (4)	5453 (8)	2366 (13)

Table 2. *Final positional parameters* ($\times 10^3$) *for hydrogen atoms*

	x	y	z		x	y	z
H(1)	145	275	850	H(12,1)	30	0	960
H(2)	27	151	992	H(12,2)	−59	48	976
H(3)	9	328	820	H(12,3)	−44	−63	873
H(4)	−18	406	1060	H(14,1)	−200	206	760
H(5)	104	387	992	H(14,2)	−211	225	956
H(6,1)	73	460	1240	H(14,3)	−240	307	804
H(6,2)	100	366	1330	H(16,1)	−127	200	1360
H(8,1)	273	14	1010	H(16,2)	−70	216	1483
H(8,2)	198	−38	921	H(16,3)	−151	285	1480
H(8,3)	277	−13	804	H(18,1)	312	520	340
H(10,1)	80	51	480	H(18,2)	307	492	170
H(10,2)	27	128	400	H(18,3)	284	616	182
H(10,3)	118	143	408				

tropic refinement and a subsequent weighted difference synthesis located the H atoms. On the terminal methyl groups at C(8), C(12), C(14), C(16) and C(18) only one H atom for each group could be located. Positions of other H atoms attached to them were calculated on stereochemical grounds. The H atoms were included in the structure factor calculations only. For the H atoms the isotropic thermal parameters were those of the bonded atoms. A scale factor, heavy-atom coordinates and anisotropic thermal parameters (271 variables in

all) were refined. Anisotropic thermal parameters (of the non-hydrogen atoms) are in the usual range: maximum values of U_{22}, 0·218 (8), and U_{33}, 0·202 (8) Å^2, were obtained for O(2). The final $R = 0·069$ and $R_w = 0·065$ for 2104 reflexions having $I > 2\sigma(I)$.

Scattering factors given by Cromer & Mann (1968) and (for H) Stewart, Davidson & Simpson (1965) were used.

The calculations were carried out on the Univac 1100 computer at the University Computing Centre in Zagreb with XRAY (Stewart, Kruger, Ammon, Dickinson & Hall, 1972).

Positional parameters are listed in Tables 1 and 2.*

Discussion

The structural formula and bond lengths are given in Fig. 1 and bond angles in Table 3. The molecular packing is illustrated in Fig. 2. The conformation of the sugar ring is shown in Fig. 3 and Tables 4, 5.

In the preparation of peracetylated amino sugars, D compounds were used as the starting materials (Pravdić & Fletcher, 1969). There is no reason to expect conversion from the D to the L enantiomer. Therefore the D enantiomer was selected and the configuration and conformation were defined in accordance with its torsion angles (Table 5).

The sugar ring exhibits the $^4C_1$ chair conformation (Tables 4, 5, Fig. 3), common for D-aldohexopyranoses with gluco, manno and galacto configurations (Stoddart, 1971). Endocyclic torsion angles

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34843 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
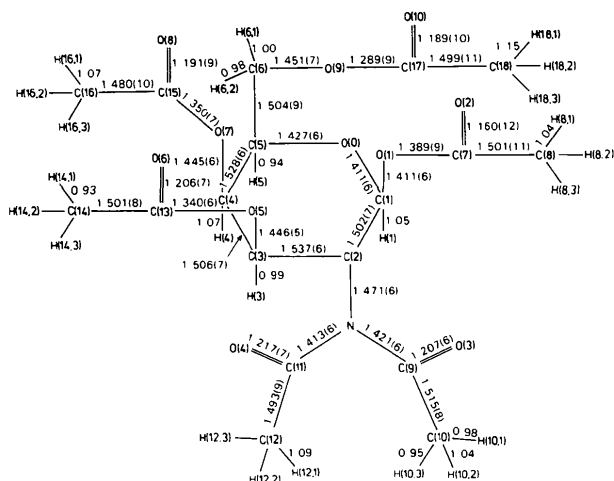

Fig. 1. The structural formula and intramolecular distances (Å).

Table 3. Bond angles (°)

Pyranose ring		N-acetyl groups	
O(0)—C(1)—C(2)	110·5 (4)	C(2)—N—C(9)	118·4 (4)
C(1)—C(2)—C(3)	112·8 (4)	C(2)—N—C(11)	119·0 (4)
C(2)—C(3)—C(4)	111·2 (4)	C(9)—N—C(11)	121·9 (4)
C(3)—C(4)—C(5)	109·0 (4)	O(3)—C(9)—N	118·8 (5)
C(4)—C(5)—O(0)	111·2 (4)	O(3)—C(9)—C(10)	122·2 (5)
C(1)—O(0)—C(5)	111·1 (4)	N—C(9)—C(10)	118·8 (4)
O(0)—C(1)—O(1)	105·1 (4)	O(4)—C(11)—N	119·8 (5)
O(1)—C(1)—C(2)	108·7 (4)	O(4)—C(11)—C(12)	121·8 (5)
O(0)—C(1)—H(1)	108	N—C(11)—C(12)	118·5 (5)
O(1)—C(1)—H(1)	111		
C(2)—C(1)—H(1)	113	O(5)-acetyl group	
C(1)—C(2)—N	112·5 (4)	C(3)—O(5)—C(13)	117·7 (4)
C(1)—C(2)—H(2)	104	O(5)—C(13)—O(6)	122·3 (5)
C(3)—C(2)—N	112·9 (4)	O(5)—C(13)—C(14)	111·3 (5)
C(3)—C(2)—H(2)	106	O(6)—C(13)—C(14)	126·4 (5)
N—C(2)—H(2)	108		
O(5)—C(3)—C(2)	104·4 (3)	O(7)-acetyl group	
O(5)—C(3)—C(4)	112·5 (4)	C(4)—O(7)—C(15)	117·5 (4)
O(5)—C(3)—H(3)	116	O(7)—C(15)—O(8)	121·4 (6)
C(2)—C(3)—H(3)	109	O(7)—C(15)—C(16)	113·2 (5)
C(4)—C(3)—H(3)	104	O(8)—C(15)—C(16)	125·3 (6)
O(7)—C(4)—C(3)	108·8 (3)		
O(7)—C(4)—C(5)	109·8 (4)	C(6)—O(9)-acetyl group	
O(7)—C(4)—H(4)	107	O(9)—C(6)—C(5)	106·2 (5)
C(3)—C(4)—H(4)	112	O(9)—C(6)—H(6,1)	114
C(5)—C(4)—H(4)	109	O(9)—C(6)—H(6,2)	113
O(0)—C(5)—C(6)	108·4 (4)	C(5)—C(6)—H(6,1)	106
O(0)—C(5)—H(5)	109	C(5)—C(6)—H(6,2)	109
C(4)—C(5)—C(6)	113·4 (4)	H(6,1)—C(6)—H(6,2)	108
C(4)—C(5)—H(5)	106	C(6)—O(9)—C(17)	118·9 (5)
C(6)—C(5)—H(5)	108	O(9)—C(17)—O(10)	122·2 (7)
		O(9)—C(17)—C(18)	113·8 (7)
O(1)-acetyl group		O(10)—C(17)—C(18)	124·0 (8)
C(1)—O(1)—C(7)	118·2 (5)		
O(1)—C(7)—O(2)	121·0 (7)		
O(1)—C(7)—C(8)	109·3 (6)		
O(2)—C(7)—C(8)	129·7 (8)		

Table 4. Displacements from least-squares plane (Å)

Atoms included in the calculation of the plane are denoted by an asterisk.

O(0)*	0·052 (6)	C(5)*	−0·050 (8)
C(2)*	−0·048 (8)	C(1)	−0·638 (8)
C(3)*	0·047 (6)	C(4)	0·653 (8)

(Table 5) are close to those found in β-D-galactose (Longchambon, Ohannessian, Avenel & Neuman, 1975).

Bond lengths and angles (Fig. 1 and Table 3) are similar to the values found in β-D-galactose (Longchambon, Ohannessian, Avenel & Neuman, 1975) with the exception of two rather short distances C(7)—O(2), 1·160 (12), and O(9)—C(17), 1·289 (9) Å. These deviations from the normal values can be attributed to poor crystal quality and thus a rather high value of R. The endocyclic and exocyclic C(1)—O(0) and C(1)—O(1) bonds have the same length, 1·411 (6) Å. The asymmetry of the endocyclic C(1)—O(0) [1·411 (6) Å] and C(5)—O(0) [1·427 (6) Å] bonds is not well pronounced. The tendency toward equal C(1)—O(0) and C(5)—O(0) distances and shortening of the

Table 5. *Torsion angles* (°)

In pyranose ring

O(0)—C(1)—C(2)—C(3)	52·7 (5)	C(2)—C(3)—C(4)—C(5)	48·7 (5)	C(4)—C(5)—O(0)—C(1)	65·2 (5)
C(1)—C(2)—C(3)—C(4)	−47·7 (5)	C(3)—C(4)—C(5)—O(0)	−57·9 (5)	C(5)—O(0)—C(1)—C(2)	−61·3 (4)

On pyranose ring

O(0)—C(1)—C(2)—N	−178·2 (3)	H(2)—C(2)—C(3)—H(3)	179	C(3)—C(4)—C(5)—H(5)	61
O(1)—C(1)—C(2)—N	−63·3 (6)	C(2)—C(3)—C(4)—O(7)	−71·0 (4)	O(7)—C(4)—C(5)—C(6)	−61·2 (5)
N—C(1)—C(2)—H(1)	60	C(2)—C(3)—C(4)—H(4)	170	O(5)—C(4)—C(5)—O(0)	−45·6 (7)
H(1)—C(1)—C(2)—H(2)	177	O(5)—C(3)—C(4)—O(7)	45·8 (4)	H(4)—C(4)—C(5)—H(5)	−63
C(1)—C(2)—C(3)—O(5)	−169·3 (4)	O(5)—C(3)—C(4)—C(5)	165·6 (3)	C(6)—C(5)—O(0)—C(1)	−169·6 (4)
C(1)—C(2)—C(3)—H(3)	66	H(3)—C(3)—C(4)—H(4)	53	H(5)—C(5)—O(0)—C(1)	−52
N—C(2)—C(3)—H(3)	−63	C(3)—C(4)—C(5)—C(6)	179·7 (4)	C(5)—O(0)—C(1)—O(1)	−178·4 (4)

Acetyl groups

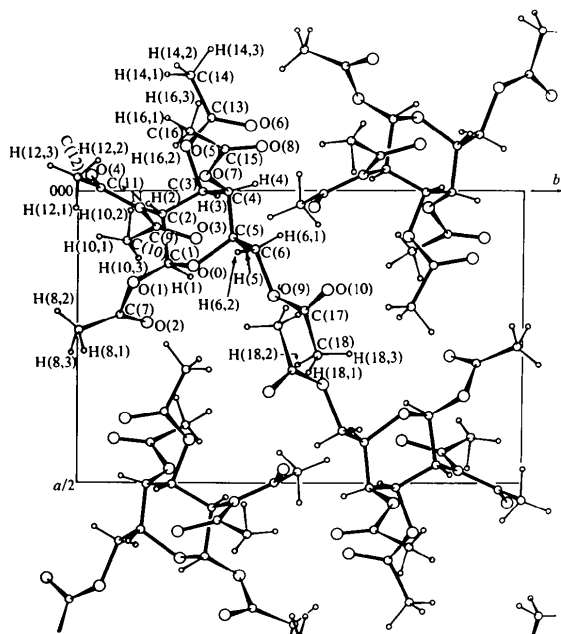|					
O(0)—C(1)—O(1)—C(7)	−123·8 (7)	C(2)—N—C(9)—C(10)	160·9 (5)	C(4)—O(7)—C(15)—O(8)	−10·6 (8)
H(1)—C(1)—O(1)—C(7)	−7	C(2)—N—C(11)—O(4)	155·1 (5)	C(4)—O(7)—C(15)—C(16)	172·8 (5)
C(1)—O(1)—C(7)—C(8)	−179·7 (7)	C(2)—N—C(11)—C(12)	−24·6 (7)	C(4)—C(5)—C(6)—O(9)	−163·6 (4)
C(1)—O(1)—C(7)—O(2)	1 (1)	C(2)—C(3)—O(5)—C(13)	−156·2 (4)	C(5)—C(6)—O(9)—C(17)	33·2 (4)
C(1)—C(2)—N—C(9)	−58·2 (5)	C(3)—O(5)—C(13)—O(6)	−2·4 (8)	C(6)—O(9)—C(17)—O(10)	63·5 (6)
C(1)—C(2)—N—C(11)	131·6 (4)	C(3)—O(5)—C(13)—C(14)	178·6 (5)	C(6)—O(9)—C(17)—C(18)	−176·0 (6)
C(2)—N—C(9)—O(3)	−13·9 (7)	C(3)—C(4)—O(7)—C(15)	−120·8 (4)		

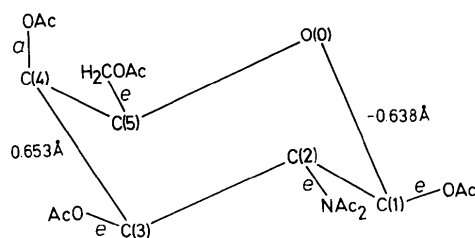Fig. 2. A view of crystal structure along c.

Fig. 3. Schematic drawing of the $^4C_1$ half-chair conformation illustrating the displacements of C(4) above and C(1) below the plane defined by O(0), C(2), C(3) and C(5).

anomeric bond implies the presence of an anomeric effect (Sundaralingam, 1968) appearing rather often in the equatorial anomers. The endocyclic C(1)—O(0)—C(5) angle is 111·1 (4)°, and the exocyclic O(0)—C(1)—O(1) is 105·1 (4)°. The endocyclic C—C distances are in the range 1·502 (7) to 1·537 (6) Å. In the acetyl groups the mean C—C length of 1·498 (9) Å is reasonable for a single bond next to a C=O bond. There are two categories of C to O bonds: the C—O bonds ranging from 1·411 (6) to 1·451 (7) Å and the O—C=O bonds from 1·289 (9) to 1·389 (9) Å. The

carbonyl bonds are in the range 1·160 (12) to 1·217 (7) Å.

The mean value of the endocyclic valence angles at the C(sp^3) atoms is 111·0 (4)°. The C valence angles exterior to the ring are in a wider range [104·4 (3) to 113·4 (4)°] than the interior angles.

The peracetylated sugar has no free hydroxyl groups and thus is not able to form hydrogen bonds. The inter-molecular distances do not give any evidence of short C—H···O contacts. In the crystal structure the molecules are connected by van der Waals forces only

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$C_{18}H_{25}NO_{11}$

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Structure of (10RS,5RS,9SR)-10β-Ethyl-5,6,7,8,9,10-hexahydro-5α,9α-methanobenzocyclooctene-10α-carboxamide, $C_{16}H_{21}NO$

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Abstract

The title compound crystallizes in $P2_1/c$ with $a = 6.314$ (2), $b = 29.815$ (5), $c = 15.158$ (3) Å, $\beta = 109.55$ (2)°, $Z = 8$. The structure was refined to an R of 0.055 for 1935 observed reflexions [$I > 2\sigma(I)$]. The conformations of both symmetrically independent molecules are the same: the cyclohexene ring exhibits a half-chair and the cyclohexane moiety a chair conformation. The interatomic distances and angles are in agreement with the given atom type and hybridization. The symmetrically independent molecules are hydrogen bonded by N—H···O contacts of 2.870 and 2.857 Å between carboxamide groups, forming dimers.

Introduction

α,α-Disubstituted-1-benzylcyclohexenes as well as their intramolecular cycloalkylation products were synthesized (Mutak, 1979). The main products, 10,10-disubstituted ethyl-5,6,7,8,9,10-hexahydro-5,9-methanocyclooctenes, contain a basic skeleton similar to that of benzomorphan, typical of strong analgesics. Some derivatives of the title compound, e.g. 10α-, 10β-amines, acids and lactones, possess marked analgesic

properties; amines also show antihistaminic, antiserotoninic and anticholinergic properties (Mutak, 1979).

The crystal structure investigation of these compounds has been undertaken to establish their configuration and conformation and also to correlate their biological activities with some structural parameters.

Experimental

The space group was determined from Weissenberg photographs recorded with Cu $K\alpha$ radiation. Table 1 lists crystallographic and physical data.

Table 1. Crystallographic and physical data

Numbers in parentheses here and throughout this paper are the e.s.d.'s in the least significant digits.

$C_{16}H_{21}NO$

M_r	243.35	U	2689.02 Å³
Space group	$P2_1/c$	Z	8
a	6.314 (2) Å	D_c	1.202 Mg m⁻³
b	29.815 (5)	μ(Mo $K\alpha$)	0.168 mm⁻¹
c	15.158 (3)	Crystal shape	Prismatic
β	109.55 (2)°		