surrounding O atoms. For the three bonds involving O(1')—H, O(3')—H and O(6')—H the distances of the H atoms from the plane of the O atoms are 0·13, 0·16 and 0·02 Å respectively.

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References

ARNOTT, S. & SCOTT, W. E. (1972). J. Chem. Soc. Perkin Trans. 2, pp. 324–335.

BROWN, G. M. & LEVY, H. A. (1973). Acta Cryst. B29, 790–797.

CHU, S. S. C. & JEFFREY, G. A. (1967). Acta Cryst. 23, 1038–1049.

CHU, S. S. C. & JEFFREY, G. A. (1968). Acta Cryst. B24, 830–838.

COPPENS, P. (1970). Crystallographic Computing, edited by F. R. AHMED, pp. 225–270. Copenhagen: Munksgaard.

CREMER, D. & POPLE, J. A. (1975). J. Am. Chem. Soc. 97, 1354–1358.

CROMER, D. T. & MANN, J. B. (1968). Acta Cryst. A24, 321–324.

FRIES, D. C., RAO, S. T. & SUNDARALINGAM, M. (1971). Acta Cryst. B27, 994–1005.

GRESS, M. E. & JEFFREY, G. A. (1977). Acta Cryst. B33, 2490–2495.

HIROTSU, K. & SHIMADA, A. (1974). Bull. Chem. Soc. Jpn, 47, 1872–1879.

JEFFREY, G. A. & KIM, H. S. (1970). Carbohydr. Res. 14, 207–216.

JEFFREY, G. A., McMULLAN, R. K. & TAKAGI, S. (1977). Acta Cryst. B33, 728–737.

JEFFREY, G. A., POPLE, J. A., BINKLEY, J. S. & VISHVESHWARA, S. (1978). J. Am. Chem. Soc. 100, 373–379.

JEFFREY, G. A. & TAKAGI, S. (1977). Acta Cryst. B33, 738–742.

KANTERS, J. A., GAYKEMA, W. P. J. & ROELOFSEN, G. (1978). Acta Cryst. B34, 1873–1881.

KANTERS, J. A., ROELOFSEN, G. & SMITS, D. (1977). Acta Cryst. B33, 3635–3640.

KIM, H. S., JEFFREY, G. A. & ROSENSTEIN, R. D. (1971). Acta Cryst. B27, 307–314.

KIM, S. H. & ROSENSTEIN, R. D. (1967). Acta Cryst. 22, 648–656.

KLYNE, W. & PRELOG, V. (1960). Experientia, 16, 521–523.

LARSON, A. C. (1967). Acta Cryst. 23, 664–666.

MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1974). MULTAN 74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data. Univs. of York, England, and Louvain-la-Neuve, Belgium.

PARK, Y. J., JEFFREY, G. A. & HAMILTON, W. C. (1971). Acta Cryst. B27, 2393–2401.

PÉREZ, S. & MARCHESSAULT, R. H. (1978). Carbohydr. Res. 65, 114–120.

POPPLETON, B. J., JEFFREY, G. A. & WILLIAMS, G. J. B. (1975). Acta Cryst. B31, 2400–2404.

STEWART, J. M. (1976). The XRAY system. Tech. Rep. TR-466. Computer Science Center, Univ. of Maryland, College Park, Maryland.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). J. Chem. Phys. 42, 3175–3187.

SUNDARALINGAM, M. (1968). Biopolymers, 6, 189–213.

TAKAGI, S. & JEFFREY, G. A. (1977a). Acta Cryst. B33, 2377–2380.

TAKAGI, S. & JEFFREY, G. A. (1977b). Acta Cryst. B33, 3033–3040.

TAKAGI, S. & JEFFREY, G. A. (1977c). Acta Cryst. B33, 3510–3515.

TAKUGASAWA, F. & JACOBSON, R. A. (1978). Acta Cryst. B34, 213–218.

TANAKA, I., TANAKA, N., ASHIDA, T. & KAKUDO, M. (1976). Acta Cryst. B32, 155–160.

The Crystal and Molecular Structure of 7-(Methyl 2-acetamido-6-O-acetyl-2,3,4-trideoxy-α-D-threo-hex-2-enopyranosid-4-yl)theophylline, $C_{18}H_{23}N_5O_7$

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Abstract

The title compound, $C_{18}H_{23}N_5O_7$, crystallizes in the orthorhombic space group $P2_12_12_1$ with $a = 13\cdot1444$ (4), $b = 14\cdot9593$ (4), $c = 10\cdot5007$ (3) Å, $Z = 4$. The structure was refined to an R of 0·037. The orientation of the base relative to the sugar ring, defined in terms of rotation about the C(4')—N(7) glycosyl bond, is anti (−81·0°). The base conformation can be described by the mean values of the torsion angles of 1·8 and 0·0° for the six- and five-membered rings respectively. The sugar moiety exhibits a half-chair $^0H_5$

conformation. Molecules are connected by N(1')—H···O(4'), 2·917 (3) Å, hydrogen bonds between sugar moieties. There is no base stacking.

Introduction

Interest in the synthesis and crystal structures of unsaturated nucleosides is related to their biochemical significance (Bloch, 1975). The use of nucleosides and their analogues as therapeutic agents would be facilitated if correlations could be established between a given structural modification and biological activity. Thus X-ray structure analysis of the new type of theophylline nucleosides containing an unsaturated amino sugar has been undertaken.

The method most frequently employed for the preparation of unsaturated nucleosides is acid-catalysed fusion giving a mixture of reaction products. Two general types were isolated: 2',3'-unsaturated nucleosides with the base linked at C(1'), and 1',2'-unsaturated isomers having the base attached at C(3') (Kojić-Prodić, Danilov & Pravdić, 1976). Besides 2',3'-unsaturated nucleosides with the base linked at C(1') some new products having the base attached at C(4') have been isolated and identified (Kojić-Prodić, Danilov & Pravdić, 1976; Pravdić, 1979). The title compound was designated at that time as the α-D-erythro isomer due to the lack of a strict definition of the quasi-axial or quasi-equatorial positions dealing with the magnitude of a particular projection angle. The X-ray crystal structure data of the title compound and its isomers were correlated with the ^{1}H NMR data and thus some stereochemical ambiguities were avoided.

Experimental

The space group was determined as $P2_12_12_1$ from Weissenberg photographs recorded with Cu $K\alpha$ radiation. Cell parameters were determined by least-squares procedures from the θ values for 84 reflexions in the interval $73·82 < 2\theta < 89·90°$ at 291 K (Cu $K\alpha$ radiation). Integrated intensities for 2405 independent reflexions with $\theta < 75°$ were measured on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation.

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.

7-(Methyl 2-acetamido-6-O-acetyl-2,3,4-trideoxy-α-D-threo-hex-2-enopyranosid-4-yl) theophylline, $C_{18}H_{23}N_5O_7$

FW	437·42	Z	4
Space group	$P2_12_12_1$	D_c	1·407 Mg m^{-3}
a	13·1444 (4) Å	μ(Cu $K\alpha$)	1·137 mm^{-1}
b	14·9593 (4)	Crystal shape	Prismatic
c	10·5007 (3)	Crystal size	0·24 × 0·42 × 0·60 mm
U	2064·76 Å^3		

Crystal data are given in Table 1. The data were corrected for background, Lorentz and polarization effects.

Structure determination and refinement

The structure amplitudes were normalized by the K-curve method (Hauptman & Karle, 1953). The structure was solved by *MULTAN* (Germain, Main & Woolfson, 1971). The solution was based on 357 reflexions with $|E| > 1·40$. The E map corresponding to the solution with the best figure of merit revealed the positions of 28 non-hydrogen atoms. The remaining two atoms belonging to the methyl groups were located from the resulting Fourier synthesis.

Refinement was by the full-matrix least-squares method. The weights were $1/\sigma^2_{F_o}$ where σ_{F_o} is as defined by Stout & Jensen (1968) (equation H14) and the instability correction was 0·06 rather than 0·01. Anisotropic refinement and a subsequent difference synthesis were used to locate all H atoms. In the final cycles one scale factor, the atomic coordinates for all atoms, anisotropic thermal parameters for the heavy atoms and the isotropic thermal parameters for the H

Table 2. Final coordinates ($\times 10^4$) for the non-hydrogen atoms

	x	y	z
C(1)	6870 (2)	8274 (2)	3210 (3)
C(2)	7999 (2)	7986 (1)	5012 (2)
C(3)	9106 (2)	7737 (2)	6842 (2)
C(4)	8521 (1)	6499 (1)	5467 (2)
C(5)	7946 (1)	6171 (1)	4487 (2)
C(6)	7327 (2)	6715 (1)	3690 (2)
C(8)	8730 (2)	5107 (1)	5506 (2)
C(1')	8885 (1)	2961 (1)	3259 (2)
C(2')	7915 (1)	2980 (1)	4039 (2)
C(3')	7408 (1)	3741 (1)	4238 (2)
C(4')	7706 (1)	4592 (1)	3592 (2)
C(5')	8479 (1)	4406 (1)	2538 (1)
C(6')	8998 (2)	5228 (1)	2015 (2)
C(7')	9550 (2)	2290 (2)	1409 (2)
C(8')	6754 (2)	1903 (1)	5066 (2)
C(9')	6602 (2)	915 (2)	5249 (3)
C(10')	10189 (1)	5404 (1)	329 (2)
C(11')	10352 (2)	5244 (2)	−1064 (2)
N(1)	7428 (1)	7627 (1)	4002 (2)
N(3)	8549 (1)	7400 (1)	5747 (2)
N(7)	8094 (1)	5252 (1)	4524 (1)
N(9)	9015 (1)	5845 (1)	6115 (1)
N(1')	7648 (1)	2134 (1)	4481 (2)
O(2)	7999 (2)	8783 (1)	5213 (2)
O(6)	6772 (1)	6472 (1)	2829 (2)
O(0')	9255 (1)	3827 (1)	3009 (1)
O(1')	8660 (1)	2491 (1)	2138 (1)
O(2')	6132 (1)	2449 (1)	5394 (2)
O(3')	9336 (1)	5019 (1)	738 (1)
O(4')	10733 (1)	5837 (1)	1013 (2)

atoms (363 variables in all) were varied. The final $R = 0.046$ for all data and 0.037 for the 2140 reflexions having $|F_o| > 2\sigma_{F_o}$; $R_w = 0.048$.

Scattering factors given by *International Tables for X-ray Crystallography* (1974) and (for H) by Stewart, Davidson & Simpson (1965) were used.

The calculations were carried out on the PDP-11 computer at the Medical Foundation of Buffalo with local versions of Nonius programs.

Atomic positional parameters are listed in Tables 2 and 3.*

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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 34187 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Positional parameters* ($\times 10^3$) *for the hydrogen atoms*

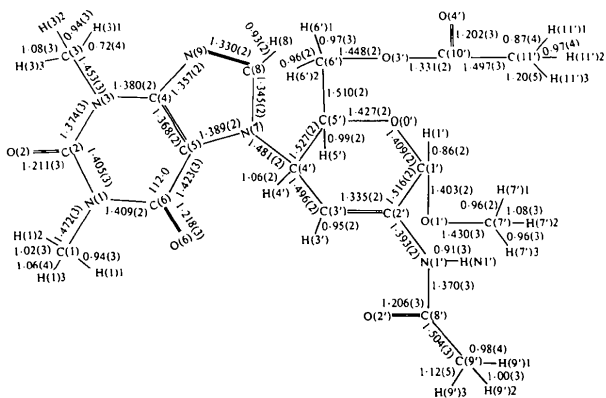|	x	y	z
H(1)1	669 (2)	797 (2)	246 (3)
H(1)2	746 (3)	869 (2)	300 (3)
H(1)3	610 (3)	861 (2)	383 (3)
H(3)1	918 (4)	741 (2)	733 (4)
H(3)2	971 (2)	806 (2)	670 (2)
H(3)3	861 (3)	822 (2)	729 (3)
H(8)	892 (2)	453 (2)	571 (2)
H(1')	935 (2)	270 (1)	371 (2)
H(3')	681 (2)	372 (1)	474 (2)
H(4')	705 (2)	488 (1)	318 (2)
H(5')	810 (2)	412 (1)	184 (2)
H(6')1	855 (2)	575 (2)	202 (2)
H(6')2	958 (2)	539 (1)	252 (2)
H(7')1	1014 (2)	219 (1)	192 (2)
H(7')2	939 (3)	165 (2)	96 (4)
H(7')3	963 (3)	277 (2)	81 (4)
H(9')1	726 (3)	62 (2)	514 (3)
H(9')2	615 (3)	78 (2)	598 (3)
H(9')3	603 (4)	74 (3)	449 (5)
H(11')1	1004 (4)	568 (3)	−145 (3)
H(11')2	1107 (3)	517 (2)	−126 (4)
H(11')3	960 (4)	489 (3)	−146 (4)
H(N1')	814 (2)	172 (2)	430 (3)

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

Description and discussion of the structure

The structural formula with the atom numbering and interatomic distances is given in Fig. 1. Bond angles are listed in Table 4. The packing is illustrated in Fig. 2. The conformation of the sugar moiety is shown in Fig. 3 and Table 5. In the preparation of the title compound, a D compound was used as the starting material, *i.e.* a 2-acetamido-2-deoxy-D-glucal derivative (Kojić-Prodić, Danilov & Pravdić, 1976). There is no reason to expect conversion from the D to L enantiomer. During the structure determination the D enantiomer was chosen, and the configuration and conformation were defined in accordance with its torsion angles (Table 6).

Base

The theophylline base (1,3-dimethylxanthine) realizes the glycosyl bond through the N(7)H form. The data in the literature (Katritzky & Boulton, 1971,
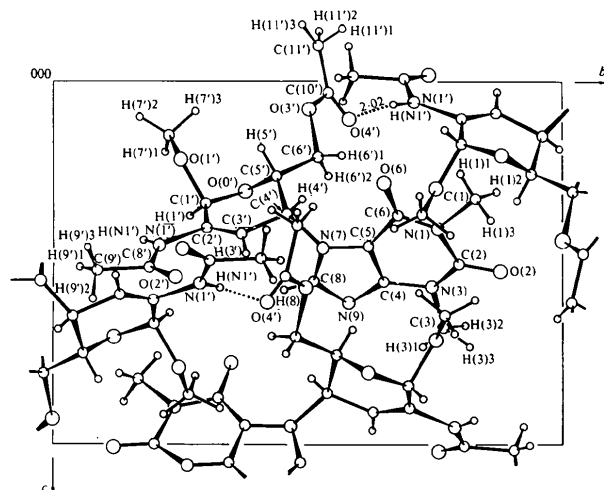

Fig. 2. A view of the crystal structure along a showing the packing arrangement and hydrogen bonds. Intermolecular hydrogen bonds between the amide H atom and the carbonyl O of the C(6') acetyl group (both of sugar moieties) are marked with dotted lines. The N(1')···O(4') distance is 2·917 Å. A right-handed coordinate system has been chosen so that the positive direction of a is away from the reader.
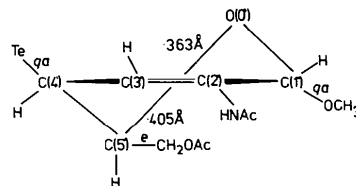

Fig. 3. Schematic drawing of the $^0H_5$ half-chair conformation illustrating the displacement of O(0') (above) and C(5') (below) the plane defined by C(1'), C(2'), C(3') and C(4'), and orientation of the substituents.

Table 4. *Bond angles* (°)

C(1)–N(1)–C(2)	116·2 (1)	C(3′)–C(4′)–C(5′)	110·3 (1)
C(1)–N(1)–C(6)	117·2 (1)	C(3′)–C(4′)–N(7)	111·0 (1)
C(2)–N(1)–C(6)	126·6 (1)	C(5′)–C(4′)–N(7)	111·8 (1)
N(1)–C(2) O(2)	120·5 (2)	O(0′)–C(5′)–C(4′)	109·6 (1)
N(1)–C(2)–N(3)	117·5 (1)	O(0′)–C(5′)–C(6′)	107·3 (1)
O(2)–C(2)–N(3)	122·2 (2)	C(4′)–C(5′)–C(6′)	114·5 (1)
C(2)–N(3)–C(3)	119·2 (1)	O(3′)–C(6′)–C(5′)	107·4 (1)
C(2)–N(3)–C(4)	119·3 (1)	N(1′)–C(8′)–O(2′)	122·6 (1)
C(3)–N(3)–C(4)	121·4 (1)	N(1′)–C(8′)–C(9′)	114·8 (2)
N(3)–C(4)–C(5)	121·7 (1)	O(2′)–C(8′)–C(9′)	122·6 (2)
N(3)–C(4)–N(9)	125·7 (1)	O(3′)–C(10′)–O(4′)	122·7 (1)
C(5)–C(4)–N(9)	112·6 (1)	O(3′)–C(10′)–C(11)	111·5 (1)
C(4)–C(5)–N(7)	104·8 (1)	O(4′)–C(10′)–C(11′)	125·8 (1)
C(4)–C(5)–C(6)	123·6 (1)	C(1′)–O(1′)–C(7′)	112·5 (1)
C(6)–C(5)–N(7)	131·5 (1)	N(7)–C(8)–H(8)	119·8 (9)
N(1)–C(6)–C(5)	111·3 (1)	N(9)–C(8)–H(8)	126·0 (9)
N(1)–C(6)–O(6)	121·3 (1)	H(1′)–C(1′)–C(2′)	108·2 (9)
C(5)–C(6)–O(6)	127·4 (1)	C(2′)–C(3′)–H(3′)	118·7 (8)
C(5)–N(7)–C(8)	105·6 (1)	C(2′)–N(1′)–H(N1′)	111·5 (1)
C(5)–N(7)–C(4′)	126·4 (1)	C(8′)–N(1′)–H(N1′)	122·0 (1)
C(8)–N(7)–C(4′)	127·8 (1)	O(0′)–C(1′)–H(1′)	105·7 (9)
N(7)–C(8)–N(9)	114·2 (1)	O(1′)–C(1′)–H(1′)	112·3 (9)
C(4)–N(9)–C(8)	102·8 (1)	C(2′)–C(1′)–H(1′)	108·2 (9)
C(1′)–O(0′)–C(5′)	112·1 (1)	C(2′)–C(3′)–H(3′)	118·7 (8)
O(0′)–C(1′)–O(1′)	112·2 (1)	C(4′)–C(3′)–H(3′)	119·4 (9)
O(0′)–C(1′)–C(2′)	111·9 (1)	C(3′)–C(4′)–H(4′)	108·5 (8)
O(1′)–C(1′)–C(2′)	106·6 (1)	C(5′)–C(4′)–H(4′)	108·7 (8)
N(1′)–C(2′)–C(1′)	112·0 (1)	N(7)–C(4′)–H(4′)	106·3 (8)
N(1′)–C(2′)–C(3′)	126·6 (1)	O(0′)–C(5′)–H(5′)	110·4 (7)
C(1′)–C(2′)–C(3′)	121·4 (1)	C(4′)–C(5′)–H(5′)	106·9 (8)
C(2′)–C(3′)–C(4′)	121·6 (1)	C(6′)–C(5′)–H(5′)	108·2 (8)

Table 5. *Displacements from least-squares planes through the base and sugar* (Å)

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

Theophylline		Sugar	
N(1)*	0·014 (2)	C(1′)*	0·013 (3)
C(2)*	−0·005 (4)	C(2′)*	−0·028 (3)
N(3)*	−0·007 (2)	C(3′)*	0·028 (3)
C(4)*	0·012 (2)	C(4′)*	−0·013 (3)
C(5)*	0·012 (2)	C(5′)	−0·403 (2)
C(6)*	−0·021 (3)	O(0′)	0·363 (2)
N(7)*	0·004 (2)	N(1′)	−0·135 (3)
C(8)*	−0·007 (3)	H(N1′)	−0·072 (47)
N(9)*	−0·002 (2)	H(3′)	0·004 (35)
O(2)	−0·017 (3)		
O(6)	−0·066 (2)		
C(1)	0·055 (4)		
C(3)	−0·100 (4)		

and references therein) give evidence of the appearance of the xanthines predominantly in the N(7)H form. Calculations by the CNDO (complete neglect of differential overlap) method confirm this (Pullman, 1970).

The base conformation is defined by the mean torsion angles of 1·8 and 0·0° for the six- and five-membered rings respectively (Table 6). The base is

nearly planar as can also be seen from the displacements of the atoms from the least-squares planes (Table 5). Bond distances and angles are in agreement with the expected values for a given type and hybridization.

Sugar moiety

Bond lengths and angles (Fig. 1 and Table 4) are comparable to values found in some peracetylated 2,3-dideoxyaldopyranoses (Rogić, 1975; Kojić-Prodić, Rogić & Ružić-Toroš, 1976). The asymmetry of the endocyclic C(1′)–O(0′) [1·409 (2) Å] and C(5′)–O(0′) [1·427 (2) Å] bonds is pronounced. The exocyclic anomeric C(1′)–O(1′), 1·403 (2) Å, is significantly shorter than the normal C–O single bond of 1·428 Å (Sundaralingam, 1968). The exocyclic O(0′)–C(1′)–O(1′) bond angle is 112·2 (1) and larger than the 110·5 (2)° found in peracetylated 2,3-dideoxy-α-D-*threo*-aldopyranose (Kojić-Prodić, Rogić & Ružić-Toroš, 1976).

The sugar ring exhibits the $^0H_5$ half-chair conformation (Fig. 3). The best least-squares plane is defined by C(1′), C(2′), C(3′) and C(4′); O(0′) and C(5′) are displaced from this plane by 0·363 and −0·403 Å, respectively (Table 5). The puckering of the sugar ring required by the $^0H_5$ conformation can be described by the values of the C(2′)–C(3′)–C(4′)–C(5′) (11·4°) and C(3′)–C(4′)–C(5′)–O(0′) (−47·0°) torsion angles (Table 6); these values are very similar to the 7·1 and −40·5° found in peracetylated 2,3-dideoxy-α-D-*threo*-aldopyranose (Kojić-Prodić, Rogić & Ružić-Toroš, 1976) appearing in the $^0H_5$ conformation. Sundaralingam (1968) has defined the conformation about C(5)–C(6) in pyranosides by the angle $\varphi_{00} =$ O(5)–C(5)–C(6)–O(6). In the present structure this angle is described by the sequence O(0′)–C(5′)–C(6′)–O(3′) and its value of 82·5 (2)° is in the range ±60 ± 30° common to pyranoside derivatives.

The ring substituents are attached at C(1′) and C(4′) in quasi-axial and C(5′) in equatorial positions (Fig. 3) (Stoddart, 1971).

Nucleoside conformation and molecular packing

The orientation of the base relative to the sugar ring, described in terms of rotation about the N(7)–C(4′) glycosyl bond for the sequence C(8)–N(7)–C(4′)–C(5′) is *anti* (−81·0°) (Sundaralingam, 1975).

The molecules are connected by N(1′)–H···O(4′), 2·917 (3) Å, hydrogen bonds acting between sugar moieties only (Fig. 2). Each molecule serves as donor and acceptor forming two hydrogen bonds with the neighbouring molecules and thus a three-dimensional network is built up. Base stacking does not occur.

Table 6. *Torsion angles* (°) *describing the conformation of the molecule*

Theophylline		Pyranose ring			
N(1)—C(6)—C(5)—C(4)	3·0 (3)	O(0')—C(1')—C(2')—C(3')	11·1 (2)	H(3')—C(3')—C(4')—H(4')	−43·2 (1)
C(2)—N(1)—C(6)—C(5)	−4·0 (3)	C(1')—C(2')—C(3')—C(4')	6·6 (2)	C(3')—C(4')—C(5')—C(6')	−167·5 (1)
N(3)—C(2)—N(1)—C(6)	2·6 (3)	C(2')—C(3')—C(4')—C(5')	11·4 (2)	C(3')—C(4')—C(5')—H(5')	72·7 (1)
C(4)—N(3)—C(2)—N(1)	0·0 (3)	C(3')—C(4')—C(5')—O(0')	−47·0 (2)	N(7)—C(4')—C(5')—O(0')	77·1 (2)
C(5)—C(4)—N(3)—C(2)	−1·0 (3)	C(4')—C(5')—O(0')—C(1')	68·4 (2)	H(4')—C(4')—C(5')—H(5')	−46·2 (1)
C(6)—C(5)—C(4)—N(3)	0·0 (3)	C(5')—O(0')—C(1')—C(2')	−48·8 (2)	O(0')—C(5')—C(6')—O(3')	82·5 (2)
C(4)—C(5)—N(7)—C(8)	0·0 (2)	*Others*		C(5')—C(6')—O(3')—C(10')	−147·3 (2)
C(5)—N(7)—C(8)—N(9)	0·0 (2)			C(6')—O(3')—C(10')—C(11')	−170·8 (2)
N(7)—C(8)—N(9)—C(4)	0·0 (2)	O(0')—C(1')—C(2')—N(1')	−169·2 (1)	C(6')—O(3')—C(10')—O(4')	7·8 (3)
C(8)—N(9)—C(4)—C(5)	0·0 (2)	O(1')—C(1')—C(2')—C(3')	−111·9 (2)	O(0')—C(1')—O(1')—C(7')	67·1 (2)
N(9)—C(4)—C(5)—N(7)	0·0 (2)	H(1')—C(1')—C(2')—C(3')	127·1 (2)	C(1')—C(2')—N(1')—C(8')	−170·9 (2)
		C(1')—C(2')—C(3')—H(3')	−180·0 (2)	C(2')—N(1')—C(8')—O(2')	−8·6 (3)
		C(2')—C(3')—C(4')—N(7)	−113·1 (2)	C(2')—N(1')—C(8')—C(9')	170·4 (2)
		C(2')—C(3')—C(4')—H(4')	130·4 (1)	C(8)—N(7)—C(4')—C(5')	−81·0 (2)

References

BLOCH, A. (1975). *Ann. NY Acad. Sci.* **255**, 576–596.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

HAUPTMAN, H. & KARLE, J. (1953). *Solution of the Phase Problem.* I. *The Centrosymmetric Crystal,* ACA Monograph No. 3. Pittsburgh: Polycrystal Book Service.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

KATRITZKY, A. R. & BOULTON, A. J. (1971). *Adv. Heterocycl. Chem.* **13**, 77–159.

KOJIĆ-PRODIĆ, B., DANILOV, B. & PRAVDIĆ, N. (1976). *Carbohydr. Res.* **52**, C7–C11.

KOJIĆ-PRODIĆ, B., ROGIĆ, V. & RUŽIĆ-TOROŠ, Ž. (1976). *Acta Cryst.* B**32**, 1833–1838.

PRAVDIĆ, N. (1979). To be published.

PULLMAN, B. (1970). *Quantum Aspects of Heterocyclic Compounds in Chemistry and Biochemistry,* p. 292. New York: Academic Press.

ROGIĆ, V. (1975). PhD Thesis. Faculty of Science, Univ. of Zagreb.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

STODDART, J. F. (1971). *Stereochemistry of Carbohydrates,* pp. 50–93. New York: John Wiley.

STOUT, G. H. & JENSEN, L. H. (1968). *X-ray Structure Determination.* New York: Macmillan.

SUNDARALINGAM, M. (1968). *Biopolymers,* **6**, 189–213.

SUNDARALINGAM, M. (1975). *Ann. NY Acad. Sci.* **255**, 3–42.

The Crystal and Molecular Structure of 7-(Methyl 2-acetamido-6-*O*-acetyl-2,3,4-trideoxy-β-D-*erythro*-hex-2-enopyranosid-4-yl)theophylline, $C_{18}H_{23}N_5O_7$

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Abstract

The title compound crystallizes in the monoclinic space group *C*2 with $a = 24\cdot093$ (1), $b = 5\cdot003$ (3), $c = 19\cdot002$ (2) Å, $\beta = 118\cdot85$ (4)°, $Z = 4$. The structure was refined to an *R* of 0·051. The orientation of the base relative to the sugar ring, defined in terms of rotation about the C(4')—N(7) glycosyl bond, is *syn* (105·1°). The theophylline base is planar within the limits of experimental error. The sugar moiety exhibits a half-chair $^5H_0$ conformation. The molecules are connected by N(1')—H···O(2), 3·09 Å, hydrogen bonds acting between the sugar and base moieties forming an infinite chain along **a**. Base stacking occurs along **b**.