in the $\mathrm{NS^{VI}O}$ pairs. All four sulfaguanidine structures exhibit such parameters (see above).

(5) The presence of $110° < \mathrm{NS^{VI}O'} > \mathrm{NS^{VI}O}$ inequalities in 9 of 32 structures of (aryl)$\mathrm{SO_2N}X,X'$ type helped to shed light on the role of the different spatial arrangement of the trigonal-pyramidal lp:N[$\mathrm{S^{VI}}$,X,X'] groups relative to the $\mathrm{S^{VI}}$[O′,O,N,C] tetrahedra from those found in the other 23 structures (Fig. 8). Of course, a deeper understanding of these phenomena is still ahead together with a study of any further correlations between the parameters of the retrieved *N*-substituted arylsulfonamides.

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Structure of 7-(Methyl 2-acetamido-2,3,4-trideoxy-α-D-*erythro*-hex-2-enopyranosid-4-yl)theophylline Monohydrate

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Abstract

$\mathrm{C_{16}H_{21}N_5O_6.H_2O}$, $M_r = 397{\cdot}40$, crystallizes in the orthorhombic space group $P2_12_12_1$ with $a = 14{\cdot}520\,(5)$, $b = 13{\cdot}844\,(6)$, $c = 9{\cdot}347\,(3)$ Å, $Z = 4$, $U = 1878{\cdot}89$ Å^3, $D_c = 1{\cdot}409$ Mg m^{-3}, μ(Cu $K\alpha$) = $2{\cdot}271$ mm^{-1}. Final $R = 0{\cdot}059$ for 1434 observed reflexions [$I > 2\sigma(I)$]. The interatomic distances and angles are in agreement with the given atom type and hybridization. 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* (75·5°). The theophylline conformation is defined by the mean torsion angles of 3·9 and 0·6° for the six-and five-membered rings, respectively. The sugar moiety exhibits a half-chair $^0H_5$ conformation. The packing is dominated by hydrogen bonds. Sugar–base interaction is realized through O(3′)···O(2), 2·730 (7) Å. The water molecule is hydrogen bonded to the sugar residue by O(W)···O(3′), 2·860 (8), and N(1′)···O(W), 2·951 (6) Å, as well as to the base moiety by O(W)···N(9), 3·046 (7) Å. There is no base stacking.

Experimental

The space group was deduced from Weissenberg photographs recorded with Cu $K\alpha$ radiation. The

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diffraction symmetry and extinctions determined $P2_12_12_1$ uniquely. A crystal 0·08 × 0·08 × 0·52 mm was used for the data collection. The intensities were collected on a Philips PW 1100 computer-controlled four-circle diffractometer in the ω scan mode [scan width = 1·20° (θ), scan speed = 0·03° (θ) s^{-1}] with graphite-monochromated Cu $K\alpha$ radiation. 1434 independent reflections $I > 2\sigma(I)$ in the range $9 < 2\theta < 140°$ were used in the calculations. Three standard reflections were measured every 2 h. The data were corrected for background, Lorentz and polarization effects but not for absorption.

Table 2. *Positional* (×10³) *parameters for* H *atoms*

	x	*y*	*z*		*x*	*y*	*z*
H(1)1	1063	629	477	H(6′)2	714	751	908
H(1)2	1136	552	557	H(7′)1	383	772	822
H(1)3	1086	523	396	H(7′)2	407	722	982
H(3)1	925	266	860	H(7′)3	488	790	894
H(3)2	971	219	701	H(9′)1	315	385	400
H(3)3	1043	282	826	H(9′)2	275	460	537
H(8)	661	419	782	H(9′)3	285	504	356
H(1′)	473	724	632	H(N1′)	380	595	585
H(3′)	608	486	500	H(O3′)	833	807	743
H(4′)	729	639	554	H(*W*)1	774	815	534
H(5′)	635	598	826	H(*W*)2	758	742	373
H(6′)1	799	653	852				

Structure determination and refinement

Overall temperature (B = 2·70 Å²) and scale factors were determined (Wilson, 1942) and used to compute normalized structure amplitudes by the routine *NORMAL* included in *MULTAN* 78. The structure was solved with *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) based on 300 reflexions with $|E| \geq 1·20$. The E map corresponding to the solution with the best figure of merit (ABS FOM = 1·240, PSI ZERO = 1·232, RESID = 32·84) revealed 27 non-hydrogen atoms out of 28. The remaining C atom of a methyl group [C(7′)] was located by Fourier synthesis. Refinement was by least squares minimizing $\sum w||F_o| - |F_c||^2$. A weighting scheme of type 3 from XRAY (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) was used. The weights were assigned as $w = w_1w_2$, where $w_1 = 1$ for $|F_o| \leq 20$ and $w_1 = 20/|F_o|$ for $|F_o| > 20$; $w_2 = 1$ for $\sin\theta \geq 0·3$ and $w_2 = (\sin\theta)/0·3$ for $\sin\theta < 0·3$. Anisotropic refinement and a subsequent weighted difference synthesis located the H atoms which were included in the structure factor calculations only. For the H atoms the isotropic thermal parameters were those of the bonded atoms plus one. A scale factor, heavy-atom coordinates and anisotropic thermal parameters (253 variables in all) were refined. Anisotropic thermal parameters (of the non-hydrogen atoms) are in the usual range: maximum values of U_{11} for C(7′) and C(9′) of 0·114 (6) and 0·111 (6) Å² were obtained, respectively. The final R = 0·059 and R_w = 0·069 for 1434 reflexions having $I > 2\sigma(I)$.

Table 1. *Final atomic coordinates* (×10⁴) *and isotropic thermal parameters* (×10²) *for non-hydrogen atoms*

	x	*y*	*z*	U_{eq} (Å²)*
O(2)	11029 (2)	3991 (3)	6524 (5)	5·90
O(6)	8973 (2)	6269 (2)	5021 (5)	5·19
O(0′)	5995 (2)	7193 (2)	7075 (4)	4·87
O(1′)	4654 (2)	6539 (2)	8069 (4)	5·23
O(2′)	4787 (2)	4264 (3)	4082 (6)	6·58
O(3′)	7806 (3)	7737 (4)	7133 (6)	8·02
O(*W*)	7733 (3)	8182 (3)	4147 (5)	6·66
N(1)	9980 (2)	5089 (3)	5727 (6)	4·43
N(3)	9529 (3)	3717 (3)	7061 (5)	4·46
N(7)	7500 (3)	5074 (3)	6646 (5)	4·22
N(9)	7897 (3)	3652 (3)	7536 (6)	4·86
N(1′)	4340 (2)	5539 (3)	5466 (5)	4·77
C(1′)	5130 (3)	6767 (4)	6779 (7)	4·46
C(2′)	5211 (3)	5857 (4)	5936 (6)	3·98
C(3′)	6032 (3)	5480 (4)	5594 (6)	4·28
C(4′)	6903 (3)	5883 (4)	6211 (6)	4·21
C(5′)	6677 (3)	6512 (4)	7540 (7)	4·60
C(6′)	7472 (4)	7069 (5)	8169 (8)	6·46
C(7′)	4332 (6)	7397 (6)	8774 (10)	8·74
C(8′)	4170 (4)	4756 (4)	4627 (7)	5·14
C(9′)	3166 (4)	4556 (5)	4368 (10)	7·34
C(1)	10740 (4)	5587 (5)	4969 (9)	6·12
C(2)	10220 (3)	4250 (4)	6454 (7)	4·70
C(3)	9743 (4)	2793 (4)	7776 (8)	5·69
C(4)	8649 (3)	4078 (4)	7040 (6)	4·24
C(5)	8447 (3)	4945 (4)	6417 (6)	3·93
C(6)	9105 (3)	5511 (4)	5692 (6)	4·46
C(8)	7220 (4)	4277 (4)	7360 (7)	4·97

* Defined according to Willis & Pryor (1975).

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

The calculations were carried out on the Univac 1110 computer at the University Computing Centre in Zagreb with the XRAY system.

Atom coordinates are listed in Tables 1 and 2.*

Description and discussion of the structure

The structural formula, atom numbering and bond lengths are given in Fig. 1. Bond angles are listed in Table 3. The molecular packing and hydrogen bonds are illustrated in Fig. 2. The conformation of the sugar moiety is shown in Fig. 3 and Tables 4 and 5. Torsion angles defining the conformation of the molecule are presented in Table 5.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35843 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

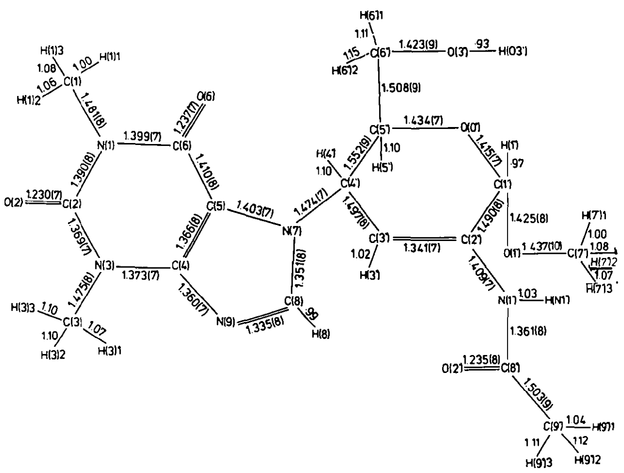

Fig. 1. The structural formula with the atom numbering and bond lengths (Å).

Table 3. *Bond angles* (°)

The e.s.d. for all listed bond angles involving non-hydrogen atoms is 0·5°.

C(1)–N(1)–C(2)	115·9	C(2′)–C(3′)–C(4′)	120·9
C(1)–N(1)–C(6)	118·1	C(3′)–C(4′)–C(5′)	109·8
C(2)–N(1)–C(6)	126·0	C(3′)–C(4′)–N(7)	108·6
N(1)–C(2)–O(2)	120·6	C(5′)–C(4′)–N(7)	109·3
N(1)–C(2)–N(3)	118·0	O(0′)–C(5′)–C(4′)	105·8
O(2)–C(2)–N(3)	121·4	O(0′)–C(5′)–C(6′)	108·1
C(2)–N(3)–C(3)	120·1	C(4′)–C(5′)–C(6′)	115·9
C(2)–N(3)–C(4)	118·7	O(3′)–C(6′)–C(5′)	109·1
C(3)–N(3)–C(4)	121·2	N(1′)–C(8′)–O(2′)	123·0
N(3)–C(4)–C(5)	121·8	N(1′)–C(8′)–C(9′)	114·5
N(3)–C(4)–N(9)	125·7	O(2′)–C(8′)–C(9′)	122·4
C(5)–C(4)–N(9)	112·5	C(1′)–O(1′)–C(7′)	111·2
C(4)–C(5)–N(7)	104·9	C(2′)–N(1′)–C(8′)	126·3
C(4)–C(5)–C(6)	123·3	N(7)–C(8)–H(8)	126
C(6)–C(5)–N(7)	131·8	N(9)–C(8)–H(8)	120
N(1)–C(6)–C(5)	111·8	C(2′)–N(1′)–H(N1′)	114
N(1)–C(6)–O(6)	120·4	C(8′)–N(1′)–H(N1′)	120
C(5)–C(6)–O(6)	127·7	O(0′)–C(1′)–H(1′)	110
C(5)–N(7)–C(8)	105·5	O(1′)–C(1′)–H(1′)	103
C(5)–N(7)–C(4′)	129·1	C(2′)–C(1′)–H(1′)	112
C(8)–N(7)–C(4′)	125·4	C(2′)–C(3′)–H(3′)	121
N(7)–C(8)–N(9)	113·8	C(4′)–C(3′)–H(3′)	118
C(4)–N(9)–C(8)	103·4	C(3′)–C(4′)–H(4′)	117
C(1′)–O(0′)–C(5′)	113·5	C(5′)–C(4′)–H(4′)	102
O(0′)–C(1′)–O(1′)	110·9	N(7)–C(4′)–H(4′)	110
O(0′)–C(1′)–C(2′)	112·7	O(0′)–C(5′)–H(5′)	109
O(1′)–C(1′)–C(2′)	107·3	C(4′)–C(5′)–H(5′)	102
N(1′)–C(2′)–C(1′)	111·0	C(6′)–C(5′)–H(5′)	116
N(1′)–C(2′)–C(3′)	127·0	C(6′)–O(3′)–H(O3′)	113
C(1′)–C(2′)–C(3′)	121·7	H(*W*)1–O(*W*)–H(*W*)2	108

In the preparation of the title compound (Pravdić & Danilov, 1981) the D enantiomer of the starting compound was used. In the present structure determination the D enantiomer was selected and the configuration and conformation were defined in accordance with its torsion angles (Table 5).

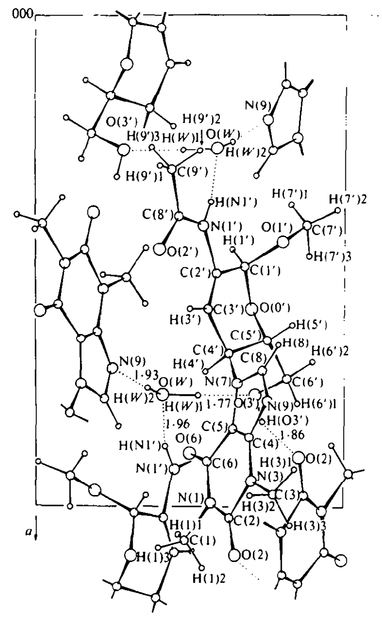

Fig. 2. A view of the crystal structure along **b** showing the packing and hydrogen bonds (Å). The water molecule is hydrogen bonded to the sugar residue through O(*W*)···O(3′), 2·860 (8), and N(1′)···O(*W*), 2·951 (6) Å, as well as to the base moiety by O(*W*)···N(9), 3·046 (7) Å. Sugar–base contact is through O(3′)···O(2), 2·730 (7) Å. A right-handed coordinate system has been chosen so that the positive direction of **b** is away from the reader.

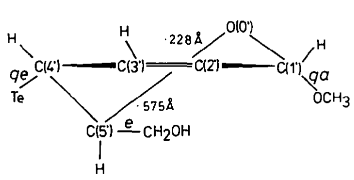

Fig. 3. Schematic drawing of the ${}^{0}H_{5}$ half-chair conformation illustrating the displacement of O(0′) above and C(5′) below the plane defined by C(1′), C(2′), C(3′) and C(4′) and the orientation of the substituents.

Base

The theophylline base (1,3-dimethylxanthine) realizes the glycosyl bond through the N(7)H form. Bond distances and angles are in good agreement with those in 7-(methyl 2-acetamido-6-*O*-acetyl-2,3,4-trideoxy-α-D-*threo*-hex-2-enopyranosid-4-yl)theophylline (Kojić-Prodić, 1979).

The base conformation is defined by the mean torsion angles of 3·9 and 0·6° for the six- and five-membered rings, respectively (Table 5). The departure of the base from planarity is more pronounced (Tables 4 and 5) than in 7-(methyl 2-acetamido-6-*O*-acetyl-2,3,4-trideoxy-α-D-*threo*-hex-2-enopyranosid-4-yl)theophylline (1·8 and 0°) (Kojić-Prodić, 1979) and its *β-erythro* stereoisomer (1 and 0·8°) (Kojić-Prodić, Coffou & Ružić-Toroš, 1979). In the title compound C(3) is nearly in the least-squares plane [0·007 (11) Å] whereas in both quoted structures it is displaced by 0·1 Å. The neighbouring O(2) is involved in a hydrogen bond with the sugar moiety [O(3′)–H(O3′)···O(2), 2·730 Å]. In the *α-threo* stereoisomer a base is not involved in hydrogen bonding at all but in the *β-erythro* isomer O(2) acts as an acceptor in N(1′)···O(2), 3·09 Å. Thus the hydrogen bond itself could not be an explanation for the deviation of the base from planarity.

Sugar moiety

Bond lengths and angles (Fig. 1 and Table 3) are in good agreement with those in the theophylline nucleoside with the 7-methyl 2-acetamido-6-*O*-acetyl-2,3,4-trideoxy-α-D-*threo*-hex-2-enopyranose moiety (Kojić-Prodić, 1979) and also in 1,4,6-tri-*O*-acetyl-2-(*N*-acetylacetamido)-2,3-dideoxy-α-D-*erythro*-hex-2-enopyranose (Ružić-Toroš, Rogić & Kojić-Prodić, 1980).

Table 4. *Displacements* (Å) *from least-squares planes through the base and sugar*

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

Theophylline		Sugar	
N(1)*	−0·037 (8)	C(1′)*	0·016 (9)
C(2)*	0·049 (10)	C(2′)*	−0·035 (9)
N(3)*	−0·004 (8)	C(3′)*	0·035 (9)
C(4)*	−0·014 (9)	C(4′)*	−0·016 (9)
C(5)*	0·013 (9)	C(5′)	−0·575 (10)
C(6)*	−0·014 (9)	O(0′)	0·228 (6)
N(7)*	0·031 (9)	N(1′)	−0·030 (8)
C(8)*	−0·003 (10)		
N(9)*	−0·020 (8)		
O(2)	0·147 (8)		
O(6)	−0·080 (7)		
C(1)	−0·108 (12)		
C(3)	0·007 (11)		

Bond-length differences between the endocyclic C(1′)–O(0′) [1·415 (7) Å] and C(5′)–O(0′) [1·434 (7) Å] bonds are $<3\sigma$. The exocyclic anomeric C(1′)–O(1′), 1·425 (8) Å, is close to the normal C–O single-bond length of 1·428 Å (Sundaralingam, 1968). The exocyclic O(0′)–C(1′)–O(1′) bond angle is 110·9 (5)°, close to the value of 110·5 (5)° in 1,4,6-tri-*O*-acetyl-2-(*N*-acetylacetamido)-2,3-dideoxy-α-D-*erythro*-hex-2-enopyranose (Ružić-Toroš, Rogić & Kojić-Prodić, 1980). The endocyclic C(5′)–O(0′)–C(1′) angle is 113·5 (5)°.

The shortening of C(1′)–C(2′) [1·490 (8) Å] and C(3′)–C(4′) [1·497 (8) Å] is due to the contiguous C(2′)=C(3′) [1·341 (7) Å]. The C–O lengths in the ring substituents are 1·437 (10) and 1·423 (9) Å.

The carbohydrate moiety 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 0·228 (6) and −0·575 (10) Å, respectively (Table 4). The ring substituents are attached at C(1′) in quasi-axial, at C(4′) in quasi-equatorial and at C(5′) in equatorial positions (Fig. 3) (Stoddart, 1971), as expected for the $^0H_5$ half-chair conformation. The highly distorted conformations seem to be due to the departure of C(5′) from the plane to avoid steric hindrance between substituents at C(4′) and C(5′). The same order of distortion is observed in 7-(methyl 2-acetamido-6-*O*-acetyl-2,3,4-trideoxy-*β*-D-*erythro*-hex-2-enopyranosid-4-yl)theophylline (Kojić-Prodić, Coffou & Ružić-Toroš, 1979) appearing in the $^5H_0$ conformation.

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′) [17·0 (8)°] and C(3′)–C(4′)–C(5′)–O(0′) [−52·8(6)°] torsion angles (Table 5).

Sundaralingam (1968) has defined the conformation about C(5)–C(6) in pyranosides by the angle φ_{OO} = O(5)–C(5)–C(6)–O(6). In the present structure

Table 5. *Torsion angles* (°) *describing the conformation of the molecule*

Theophylline					
N(1)–C(6)–C(5)–C(4)	−1·5 (9)	C(5)–C(4)–N(3)–C(2)	0·8 (9)	N(7)–C(8)–N(9)–C(4)	−0·8 (7)
C(2)–N(1)–C(6)–C(5)	−4·2 (9)	C(6)–C(5)–C(4)–N(3)	3 (1)	C(8)–N(9)–C(4)–C(5)	0·3 (6)
N(3)–C(2)–N(1)–C(6)	8·2 (9)	C(4)–C(5)–N(7)–C(8)	−0·8 (7)	N(9)–C(4)–C(5)–N(7)	0·3 (6)
C(4)–N(3)–C(2)–N(1)	−6·0 (9)	C(5)–N(7)–C(8)–N(9)	1·0 (7)		
Pyranose ring					
O(0′)–C(1′)–C(2′)–C(3′)	3·7 (9)	C(2′)–C(3′)–C(4′)–C(5′)	17·0 (8)	C(4′)–C(5′)–O(0′)–C(1′)	69·4 (6)
C(1′)–C(2′)–C(3′)–C(4′)	8·3 (9)	C(3′)–C(4′)–C(5′)–O(0′)	−52·8 (6)	C(5′)–O(0′)–C(1′)–C(2′)	−44·5 (7)
Others					
O(0′)–C(1′)–C(2′)–N(1′)	−170·5 (5)	H(3′)–C(3′)–C(4′)–H(4′)	89	C(5′)–C(6′)–O(3′)–H(O3′)	−175
O(1′)–C(1′)–C(2′)–C(3′)	−118·7 (6)	C(3′)–C(4′)–C(5′)–C(6′)	−172·6 (5)	O(0′)–C(1′)–O(1′)–C(7′)	71·6 (6)
H(1′)–C(1′)–C(2′)–C(3′)	128	C(3′)–C(4′)–C(5′)–H(5′)	61	C(1′)–C(2′)–N(1′)–C(8′)	177·3 (6)
C(1′)–C(2′)–C(3′)–H(3′)	−179	N(7)–C(4′)–C(5′)–O(0′)	−171·9 (4)	C(2′)–N(1′)–C(8′)–O(2′)	−6 (1)
C(2′)–C(3′)–C(4′)–N(7)	136·5 (6)	H(4′)–C(4′)–C(5′)–H(5′)	−174	C(2′)–N(1′)–C(8′)–C(9′)	176·5 (6)
C(2′)–C(3′)–C(4′)–H(4′)	−98	O(0′)–C(5′)–C(6′)–O(3′)	−56·2 (7)	C(8)–N(7)–C(4′)–C(5′)	75·5 (7)

Table 6. *Hydrogen bonds*

X–H···*Y*	*X*···*Y*	*X*–H	H···*Y*	*X*–H···*Y*	Symmetry operation on *Y*
O(*W*)–H(*W*)1···O(3′)	2·860 (8) Å	1·11 Å	1·77 Å	163°	x,y,z
O(*W*)–H(*W*)2···N(9)	3·046 (7)	1·15	1·93	163	$\frac{1}{2}-x+1, 1-y, \frac{1}{2}+z-1$
O(3′)–H(O3′)···O(2)	2·730 (7)	0·93	1·86	155	$-x+2, \frac{1}{2}+y, \frac{1}{2}-z+1$
N(1′)–H(N1′)···O(*W*)	2·951 (6)	1·03	1·96	160	$\frac{1}{2}+x-1, \frac{1}{2}-y+1, -z+1$

this angle is described by the sequence O(0′)—C(5′)—C(6′)—O(3′) and its value of −56·2(7)° is in the range ±60–±30° common for pyranoside derivatives.

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* [75·5 (7)°] (Sundaralingam, 1975).

The packing is dominated by hydrogen bonds (Table 6). The water molecule acts as a donor to the carbohydrate O(3′) and to the base N(9) as well as an acceptor to the acetamido N(1′)—H. Thus the water molecule is involved in hydrogen bonds with the sugar moiety by O(*W*)—H(*W*)1···O(3′), 2·860 (8), and N(1′)—H(N1′)···O(*W*), 2·951 (6) Å, and with the base residue by O(*W*)—H(*W*)2···N(9), 3·046 (7) Å. Sugar–base interaction is realized through the O(3′)—H(O3′)···O(2), 2·730 (7) Å, hydrogen bond. Base stacking does not occur.

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The Structure of Ethoxycarbonylcholine Iodide*

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Abstract

$C_8H_{18}NO_3^+.I^-$ crystallizes in space group $P\bar{1}$ with a = 11·632 (6), b = 14·658 (8), c = 15·24 (1) Å, α = 97·75 (6), β = 89·44 (3), γ = 101·07 (5)°, Z = 8, U = 2527 Å^3, D_m = 1·609, D_c = 1·59 Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 2·56 mm^{-1}, $F(000)$ = 1200. The final R = 0·062 for 5525 reflections. The ethoxycarbonylcholine ions adopt a variety of conformations. The potential originating in the ester moiety seems to make some packing patterns especially favourable.

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* IUPAC name: (ethoxycarbonyloxyethyl)trimethylammonium iodide.

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