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Methyl 1-deoxy-1-(N^1-thyminyl)-β-D-psicofuranoside

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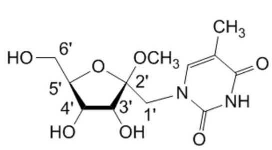
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.132; data-to-parameter ratio = 12.2.

In the structure of the title compound, $C_{12}H_{18}N_2O_7$, the furanosyl ring adopts the *S*-type sugar pucker with the following pseudorotational parameters: P_S = 159.6° (C2′-*endo* according to the designation of the ribofuranose ring of natural nucleosides; C3′-*endo* according to the numbering of the title compound) and ν_{max} = 35.9°. The conformation around the C5′—C6′ bond is *ap* (*gauche–trans*; *gt*; −*g*), with a torsion angle γ of −170.3 (2)°. The structure of the thymine base is very similar to that of thymidine. There are intermolecular N—H···O and O—H···O hydrogen bonds.

Related literature

For related literature, see: Chekhlov (1995); Kulak *et al.* (2005); Miles *et al.* (1967, 1970); Pradeepkumar *et al.* (2004); Roivainen *et al.* (2002, 2006); Seela *et al.* (1999); Young *et al.* (1969).



Experimental

Crystal data

$C_{12}H_{18}N_2O_7$
M_r = 302.28
Orthorhombic, $P2_12_12_1$
a = 5.7476 (7) Å
b = 15.6207 (12) Å
c = 15.6735 (12) Å
V = 1407.2 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 (2) K
0.35 × 0.16 × 0.16 mm

Data collection

Siemens P4 diffractometer
Absorption correction: none
3072 measured reflections
2335 independent reflections
2102 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
3 standard reflections every 97 reflections intensity decay: 0.4%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.132
S = 1.05
2335 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O4^{i}	0.86	2.00	2.842 (3)	167
O3′—H3O···O3′^{ii}	0.84	2.46	3.2745 (16)	163
O4′—H4O···O6′^{iii}	0.84	1.86	2.662 (3)	160
O6′—H6O···O2^{iv}	0.84	1.89	2.721 (2)	170

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iii) $x-1, y, z$; (iv) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2046).

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supplementary materials

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Methyl 1-deoxy-1-(N^1-thyminyl)-*β*-D-psicofuranoside

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Comment

In a search of new approaches to the synthesis of conformationally rigid 1,3-anhydro-β-*D*-psicofuranosyl nucleosides, we have briefly reported on the condensation of methyl 1,3-anhydro-4,6-di-O-toluoyl -β-*D*-psicofuranoside (I, scheme 1) with persilylated thymine (Roivainen *et al.*, 2002). Conventional work-up of the reaction mixture followed by deprotection of the product gave thymine nucleoside, structure of which was tentatively proposed as 1-(1,3-anhydro -β-*D*-psicofuranosyl)thymine (II).

Later on, careful comparison of the NMR spectroscopy data for isolated compound with those for 9-(1,3-anhydro-β-*D*-psicofuranosyl)adenine (Roivainen *et al.*, 2002), 1-(1,3-anhydro-β-*D*-psicofuranosyl)uracil (Kulak *et al.*, 2005) and 1-(1,3-anhydro-β-*D*-psicofuranosyl)thymine (II) (Pradeepkumar *et al.*, 2004), as well as the CD spectroscopy data for isolated compound with those for uracil and thymine nucleosides (Miles *et al.*, 1967, 1970; Kulak *et al.*, 2005) showed essential differences pointing to the unusual structure of the former. We have, therefore, undertaken the determination of the crystal and molecular structure of isolated thymime derivative. Recently, we have determined the single-crystal X-ray structure of 9-(1,3-anhydro-β-*D*-psicofuranosyl)adenine (Roivainen *et al.*, 2006).

The molecular structure of the new glycoside (Fig.1) was found to be methyl 1-deoxy-1-(N^1-thyminyl)-β-*D*-psicofuranoside (III). It became obvious that its formation from methyl glycoside (I) and persilylated thymine results from nucleophilic attack of the nitrogen-atom N1 of the base onto the carbon atom C1' of the sugar.

As might be expected, the structure of the thymine base of (III) was found to be very similar to that of thymidine (Young *et al.*, 1969; Chekhlov, 1995). The C1'-N1 bond length of 1.464 (2) Å is shorter than the glycosidic bond length of thymidine by 0.016 Å (Young *et al.*, 1969; Chekhlov, 1995). The furanosyl ring of (III) in the solid state adopts the S-type sugar pucker with the following pseudorotational parameters: P_S = 159.6° (C2'-*endo* according to the designation of the ribofuranose ring of natural nucleosides; C3'-*endo*; according to the atom numbering indicated in Fig. 1) and ν_{max} = 35.9°. The conformation around the C5'-C6' bond is *ap* (*gauche*,*trans*; gt;−g) with a torsion angle γ of −170.3 (2)°. It is noteworthy that the C5'-O5' bond is longer than O5'-C2' as it is the case for the most nucleosides (Seela *et al.*, 1999; Roivainen *et al.*, 2006).

In solid state the molecules are linked to each other *via* four hydrogen bonds of different strengths. From the thymine base the oxygen atoms (O2, O4) act as acceptors and the NH-group (N3) as donor of hydrogen bonds. From the sugar moiety the hydroxyl group of O4' acts as donor and the hydroxyl groups of O3' and O6' as donor as well as acceptor groups. In summary, a three dimensional hydrogen bonding scheme results (Fig. 2).

Experimental

The synthesis of compound (I) has been described earlier (Roivainen *et al.*, 2002). Samples for X-ray analyses were crystallized from a mixture of methanol and propanol-2. Single crystals suitable for X-ray diffraction were selected directly from the sample as prepared.

Refinement

In the absence of suitable anomalous scattering, Friedel equivalents could not be used to determine the absolute structure. Therefore, Friedel equivalents were merged before the final refinements.

The known configuration of the parent molecule was used to define the enantiomer employed in this structure refinement.

All H atoms were initially found in a difference Fourier synthesis. In order to maximize the data/parameter ratio, the H atoms bonded to carbon were placed in geometrically idealized positions (C—H=0.93–0.98 Å) and constrained to ride on their parent atoms with a common isotropic displament parameter. The hydrogen atoms of the OH und NH groups were first refined with the restriction of a common O—H and N—H bond length (*DFIX*). After refinement the positions of these hydrogen atoms were also constrained (AFIX 3).

Figures

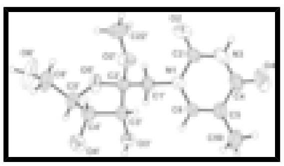


Fig. 1. The molecular structure of nucleoside (III) with the numbering scheme used. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary size.

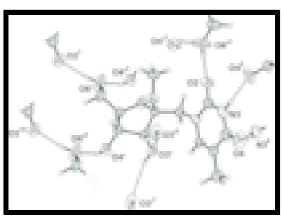


Fig. 2. Part of the crystal structure of nucleoside (III) showing the main structural features of the hydrogen bonding scheme. Dotted lines indicate hydrogen bonds. Symmetry codes for the generation of the different molecules are as follows: 1) 1 − *x*, −1/2 + *y*, 1/2 − *y*; 2) 1 − *x*, *y*, *z*; 3) −*x*, 1/2 + *y*,1/2 − *z*; 4) 1 − *x*, 1/2 + *y*, 1/2 − *y*; 5) 1/2 + *x*, 3/2 − *y*, 1 − *z*; 6) −1/2 + *x*, 3/2 − *y*, 1 − *z*; 7) −1/2 + *x*, 1/2 − *y*, 1 − *z*; 8) 1/2 + *x*, 1/2 − *y*, 1 − *z*; 9) −1 + *x*, *y*,*z*; 10) −*x*,-1/2 + *y*, 1/2 − *z*.

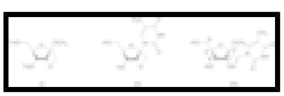


Fig. 3. The structures of (I), (II) and (III).

Methyl 1-deoxy-1-(N^1-thyminyl)-β-*D*-psicofuranoside

Crystal data

$C_{12}H_{18}N_2O_7$	F_{000} = 640
M_r = 302.28	D_x = 1.427 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 65 reflections
a = 5.7476 (7) Å	θ = 5.5–12.4°
b = 15.6207 (12) Å	μ = 0.12 mm^{-1}
c = 15.6735 (12) Å	*T* = 293 (2) K
V = 1407.2 (2) Å^3	Needle, colourless
Z = 4	0.35 × 0.16 × 0.16 mm

Data collection

Siemens P4 diffractometer	$R_{int} = 0.021$
Radiation source: fine-focus sealed tube	$\theta_{max} = 30.0°$
Monochromator: graphite	$\theta_{min} = 1.8°$
$T = 293(2)$ K	$h = -8 \rightarrow 1$
$2\theta/\omega$ scans	$k = -21 \rightarrow 1$
Absorption correction: none	$l = -1 \rightarrow 22$
3072 measured reflections	3 standard reflections
2335 independent reflections	every 97 reflections
2102 reflections with $I > 2\sigma(I)$	intensity decay: 0.4%

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0728P)^2 + 0.2397P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.044$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.132$	$\Delta\rho_{max} = 0.25$ e Å^{-3}
$S = 1.05$	$\Delta\rho_{min} = -0.22$ e Å^{-3}
2335 reflections	Extinction correction: SHELXTL (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
192 parameters	Extinction coefficient: 0.021 (4)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983)
Secondary atom site location: difference Fourier map	Flack parameter: 1.7 (14)
Hydrogen site location: inferred from neighbouring sites	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
N1	0.1653 (3)	0.49559 (9)	0.48231 (10)	0.0332 (4)
C2	0.2349 (5)	0.57613 (13)	0.45747 (13)	0.0383 (5)

O2	0.4073 (4)	0.58835 (11)	0.41307 (12)	0.0553 (5)
N3	0.0962 (5)	0.64153 (11)	0.48671 (14)	0.0487 (5)
H3	0.1333	0.6923	0.4706	0.075 (3)*
C4	−0.0964 (5)	0.63477 (13)	0.53910 (15)	0.0443 (5)
O4	−0.2060 (5)	0.69882 (12)	0.56133 (16)	0.0728 (7)
C5	−0.1533 (4)	0.54875 (13)	0.56644 (13)	0.0369 (4)
C50	−0.3524 (5)	0.53625 (19)	0.62618 (18)	0.0531 (6)
H501	−0.4222	0.5906	0.6387	0.075 (3)*
H502	−0.2973	0.5106	0.6781	0.075 (3)*
H503	−0.4658	0.4994	0.6002	0.075 (3)*
C6	−0.0208 (4)	0.48472 (12)	0.53712 (12)	0.0339 (4)
H6	−0.0562	0.4294	0.5547	0.075 (3)*
C1'	0.2955 (4)	0.42076 (12)	0.45255 (12)	0.0328 (4)
H1'1	0.3118	0.3804	0.4992	0.075 (3)*
H1'2	0.4502	0.4386	0.4354	0.075 (3)*
C2'	0.1763 (4)	0.37591 (12)	0.37717 (11)	0.0304 (4)
O2'	0.1290 (3)	0.43351 (11)	0.30982 (10)	0.0426 (4)
C3'	−0.0556 (4)	0.33227 (13)	0.39528 (12)	0.0349 (4)
H3'	−0.1868	0.3719	0.3893	0.075 (3)*
O3'	−0.0464 (4)	0.29518 (10)	0.47810 (10)	0.0512 (5)
H3O	−0.1711	0.2672	0.4789	0.075 (3)*
C4'	−0.0604 (4)	0.26182 (13)	0.32716 (13)	0.0339 (4)
H4'	−0.1183	0.2847	0.2729	0.075 (3)*
O4'	−0.1915 (3)	0.18980 (10)	0.35188 (11)	0.0466 (4)
H4O	−0.3134	0.1845	0.3229	0.075 (3)*
C5'	0.1973 (4)	0.23823 (13)	0.31950 (13)	0.0350 (4)
H5'	0.2278	0.1885	0.3559	0.075 (3)*
O5'	0.3284 (3)	0.30973 (10)	0.35161 (10)	0.0390 (3)
C6'	0.2637 (5)	0.21586 (19)	0.22929 (16)	0.0491 (6)
H6'1	0.1545	0.1745	0.2063	0.075 (3)*
H6'2	0.2585	0.2667	0.1939	0.075 (3)*
O6'	0.4901 (4)	0.18126 (19)	0.22886 (14)	0.0791 (8)
H6O	0.5052	0.1505	0.1853	0.075 (3)*
C22'	0.3241 (6)	0.4601 (2)	0.26075 (16)	0.0610 (8)
H221	0.2739	0.4990	0.2170	0.075 (3)*
H222	0.3957	0.4110	0.2349	0.075 (3)*
H223	0.4345	0.4882	0.2972	0.075 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0448 (9)	0.0214 (6)	0.0335 (7)	−0.0003 (7)	0.0067 (7)	0.0013 (5)
C2	0.0525 (12)	0.0267 (8)	0.0356 (9)	−0.0049 (9)	−0.0002 (9)	0.0056 (7)
O2	0.0691 (12)	0.0419 (9)	0.0549 (10)	−0.0134 (9)	0.0151 (10)	0.0100 (7)
N3	0.0715 (14)	0.0209 (7)	0.0536 (10)	−0.0022 (9)	0.0011 (11)	0.0046 (7)
C4	0.0599 (14)	0.0256 (8)	0.0475 (11)	0.0090 (10)	−0.0042 (11)	−0.0010 (8)
O4	0.0989 (18)	0.0346 (9)	0.0848 (14)	0.0265 (11)	0.0097 (15)	−0.0036 (9)
C5	0.0430 (11)	0.0318 (9)	0.0360 (9)	0.0022 (9)	−0.0012 (9)	−0.0047 (7)

C50	0.0507 (14)	0.0545 (13)	0.0540 (13)	0.0041 (12)	0.0113 (12)	−0.0138 (11)
C6	0.0456 (11)	0.0241 (7)	0.0319 (8)	−0.0033 (8)	0.0051 (8)	−0.0018 (6)
C1'	0.0391 (10)	0.0285 (8)	0.0307 (8)	0.0054 (8)	0.0002 (8)	−0.0003 (7)
C2'	0.0338 (9)	0.0290 (8)	0.0285 (7)	0.0049 (7)	0.0042 (7)	0.0003 (6)
O2'	0.0471 (9)	0.0461 (8)	0.0345 (7)	−0.0037 (7)	−0.0035 (7)	0.0128 (6)
C3'	0.0367 (10)	0.0297 (8)	0.0382 (9)	0.0025 (8)	0.0077 (8)	−0.0010 (7)
O3'	0.0813 (13)	0.0363 (8)	0.0361 (7)	−0.0176 (9)	0.0171 (9)	−0.0027 (6)
C4'	0.0340 (9)	0.0319 (8)	0.0359 (9)	0.0023 (8)	0.0013 (8)	−0.0009 (7)
O4'	0.0498 (9)	0.0383 (8)	0.0517 (9)	−0.0087 (7)	−0.0008 (8)	−0.0014 (7)
C5'	0.0361 (10)	0.0345 (9)	0.0345 (8)	0.0073 (8)	−0.0021 (8)	−0.0082 (7)
O5'	0.0323 (7)	0.0406 (7)	0.0440 (7)	0.0080 (6)	−0.0010 (6)	−0.0156 (6)
C6'	0.0446 (12)	0.0605 (15)	0.0423 (10)	0.0053 (12)	0.0000 (10)	−0.0230 (11)
O6'	0.0506 (11)	0.122 (2)	0.0651 (12)	0.0234 (13)	−0.0032 (10)	−0.0587 (14)
C22'	0.074 (2)	0.0713 (17)	0.0374 (10)	−0.0288 (17)	0.0071 (13)	0.0091 (11)

Geometric parameters (Å, °)

N1—C2	1.376 (2)	C2'—C3'	1.524 (3)
N1—C6	1.382 (3)	O2'—C22'	1.422 (3)
N1—C1'	1.464 (2)	C3'—O3'	1.423 (2)
C2—O2	1.226 (3)	C3'—C4'	1.534 (3)
C2—N3	1.375 (3)	C3'—H3'	0.9800
N3—C4	1.382 (4)	O3'—H3O	0.8389
N3—H3	0.8600	C4'—O4'	1.408 (3)
C4—O4	1.232 (3)	C4'—C5'	1.531 (3)
C4—C5	1.448 (3)	C4'—H4'	0.9800
C5—C6	1.338 (3)	O4'—H4O	0.8389
C5—C50	1.491 (3)	C5'—O5'	1.438 (3)
C50—H501	0.9600	C5'—C6'	1.505 (3)
C50—H502	0.9600	C5'—H5'	0.9800
C50—H503	0.9600	C6'—O6'	1.409 (4)
C6—H6	0.9300	C6'—H6'1	0.9700
C1'—C2'	1.535 (3)	C6'—H6'2	0.9700
C1'—H1'1	0.9700	O6'—H6O	0.8389
C1'—H1'2	0.9700	C22'—H221	0.9600
C2'—O5'	1.412 (2)	C22'—H222	0.9600
C2'—O2'	1.413 (2)	C22'—H223	0.9600
C2—N1—C6	120.86 (17)	C2'—O2'—C22'	116.0 (2)
C2—N1—C1'	119.41 (18)	O3'—C3'—C2'	108.65 (18)
C6—N1—C1'	119.69 (15)	O3'—C3'—C4'	110.10 (16)
O2—C2—N3	122.86 (19)	C2'—C3'—C4'	101.94 (16)
O2—C2—N1	122.5 (2)	O3'—C3'—H3'	111.9
N3—C2—N1	114.6 (2)	C2'—C3'—H3'	111.9
C2—N3—C4	127.30 (17)	C4'—C3'—H3'	111.9
C2—N3—H3	116.4	C3'—O3'—H3O	101.2
C4—N3—H3	116.4	O4'—C4'—C5'	110.30 (17)
O4—C4—N3	121.0 (2)	O4'—C4'—C3'	113.03 (17)
O4—C4—C5	123.7 (3)	C5'—C4'—C3'	102.12 (17)
N3—C4—C5	115.31 (19)	O4'—C4'—H4'	110.4

C6—C5—C4	117.6 (2)	C5'—C4'—H4'	110.4
C6—C5—C50	123.7 (2)	C3'—C4'—H4'	110.4
C4—C5—C50	118.7 (2)	C4'—O4'—H4O	112.1
C5—C50—H501	109.5	O5'—C5'—C6'	112.1 (2)
C5—C50—H502	109.5	O5'—C5'—C4'	107.02 (15)
H501—C50—H502	109.5	C6'—C5'—C4'	112.00 (19)
C5—C50—H503	109.5	O5'—C5'—H5'	108.5
H501—C50—H503	109.5	C6'—C5'—H5'	108.5
H502—C50—H503	109.5	C4'—C5'—H5'	108.5
C5—C6—N1	124.18 (18)	C2'—O5'—C5'	110.08 (15)
C5—C6—H6	117.9	O6'—C6'—C5'	109.1 (2)
N1—C6—H6	117.9	O6'—C6'—H6'1	109.9
N1—C1'—C2'	112.45 (17)	C5'—C6'—H6'1	109.9
N1—C1'—H1'1	109.1	O6'—C6'—H6'2	109.9
C2'—C1'—H1'1	109.1	C5'—C6'—H6'2	109.9
N1—C1'—H1'2	109.1	H6'1—C6'—H6'2	108.3
C2'—C1'—H1'2	109.1	C6'—O6'—H6O	108.6
H1'1—C1'—H1'2	107.8	O2'—C22'—H221	109.5
O5'—C2'—O2'	111.93 (16)	O2'—C22'—H222	109.5
O5'—C2'—C3'	105.49 (15)	H221—C22'—H222	109.5
O2'—C2'—C3'	104.80 (17)	O2'—C22'—H223	109.5
O5'—C2'—C1'	106.05 (16)	H221—C22'—H223	109.5
O2'—C2'—C1'	111.72 (16)	H222—C22'—H223	109.5
C3'—C2'—C1'	116.81 (16)		

C6—N1—C2—O2	175.6 (2)	C1'—C2'—O2'—C22'	74.0 (2)
C1'—N1—C2—O2	−2.3 (3)	O5'—C2'—C3'—O3'	80.69 (18)
C6—N1—C2—N3	−4.2 (3)	O2'—C2'—C3'—O3'	−161.01 (16)
C1'—N1—C2—N3	177.94 (19)	C1'—C2'—C3'—O3'	−36.8 (2)
O2—C2—N3—C4	−177.7 (2)	O5'—C2'—C3'—C4'	−35.54 (19)
N1—C2—N3—C4	2.1 (4)	O2'—C2'—C3'—C4'	82.75 (18)
C2—N3—C4—O4	179.6 (3)	C1'—C2'—C3'—C4'	−153.02 (17)
C2—N3—C4—C5	0.9 (4)	O3'—C3'—C4'—O4'	36.9 (3)
O4—C4—C5—C6	179.6 (3)	C2'—C3'—C4'—O4'	152.08 (17)
N3—C4—C5—C6	−1.8 (3)	O3'—C3'—C4'—C5'	−81.6 (2)
O4—C4—C5—C50	−1.2 (4)	C2'—C3'—C4'—C5'	33.6 (2)
N3—C4—C5—C50	177.4 (2)	O4'—C4'—C5'—O5'	−141.60 (16)
C4—C5—C6—N1	−0.3 (3)	C3'—C4'—C5'—O5'	−21.2 (2)
C50—C5—C6—N1	−179.4 (2)	O4'—C4'—C5'—C6'	95.2 (2)
C2—N1—C6—C5	3.6 (3)	C3'—C4'—C5'—C6'	−144.4 (2)
C1'—N1—C6—C5	−178.6 (2)	O2'—C2'—O5'—C5'	−90.3 (2)
C2—N1—C1'—C2'	−100.1 (2)	C3'—C2'—O5'—C5'	23.1 (2)
C6—N1—C1'—C2'	82.0 (2)	C1'—C2'—O5'—C5'	147.67 (17)
N1—C1'—C2'—O5'	175.76 (15)	C6'—C5'—O5'—C2'	122.23 (19)
N1—C1'—C2'—O2'	53.6 (2)	C4'—C5'—O5'—C2'	−0.9 (2)
N1—C1'—C2'—C3'	−67.1 (2)	O5'—C5'—C6'—O6'	69.4 (3)
O5'—C2'—O2'—C22'	−44.8 (3)	C4'—C5'—C6'—O6'	−170.3 (2)
C3'—C2'—O2'—C22'	−158.6 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O4^{i}	0.86	2.00	2.842 (3)	167
O3'—H3O···O3'^{ii}	0.84	2.46	3.2745 (16)	163
O4'—H4O···O6'^{iii}	0.84	1.86	2.662 (3)	160
O6'—H6O···O2^{iv}	0.84	1.89	2.721 (2)	170

Symmetry codes: (i) *x*+1/2, −*y*+3/2, −*z*+1; (ii) *x*−1/2, −*y*+1/2, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) −*x*+1, *y*−1/2, −*z*+1/2.

Fig. 1

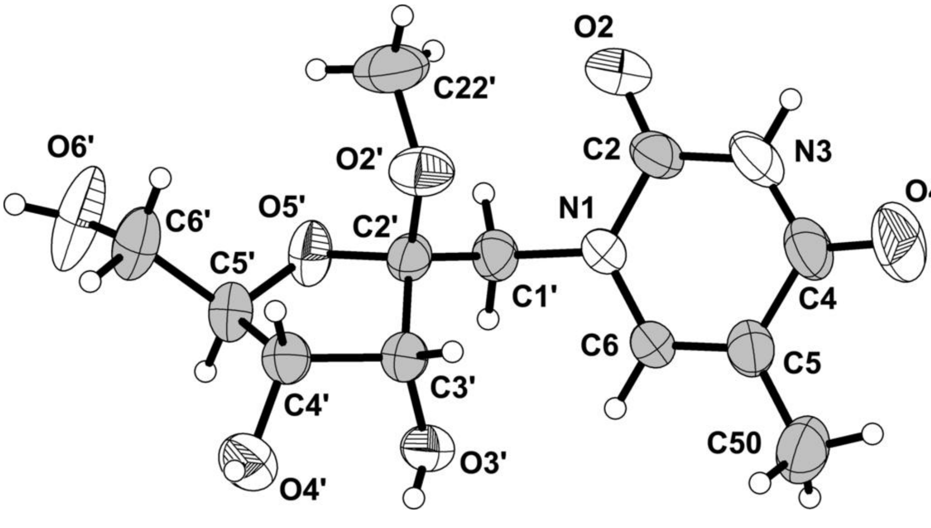


Fig. 2

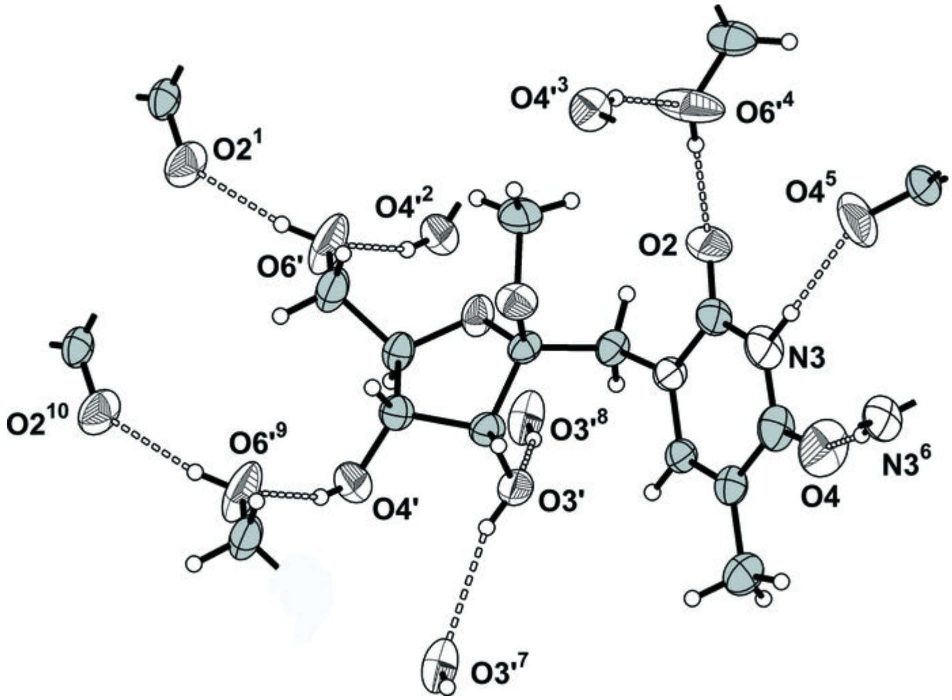
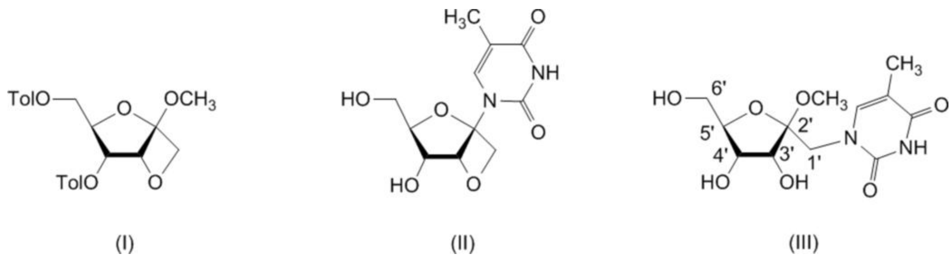


Fig. 3


TolO
OCH3
O
TolO
O
(I)
H3C
O
NH
N
O
HO
O
HO
O
(II)
CH3
HO
6'
O
OCH3
O
5'
2'
4'
3'
N
NH
1'
HO
OH
O
(III)