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7-Methyl-1-(β-D-*erythro*-pentofuranosyl)-1*H*-benzimidazol-2(3*H*)-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.080
Data-to-parameter ratio = 9.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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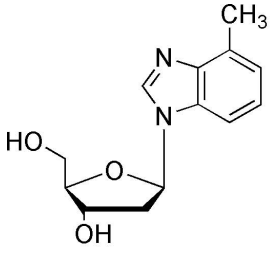
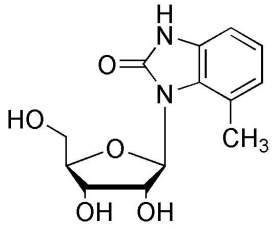
The title nucleoside, $C_{13}H_{16}N_2O_5$, was prepared and characterized by X-ray crystallographic techniques. The crystal structure determination reveals that the furanose ring adopts a C2′-*endo* conformation, while the orientation of the benzimidazole moiety with respect to the sugar moiety is mid-*anti*. The crystal structure is stabilized by intermolecular O—H···O and N—H···O hydrogen bonds.

Comment

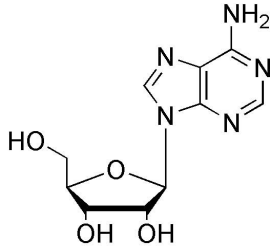
The potential of benzimidazole nucleosides to help understand the structure and function of RNA and DNA has been well documented (Guckian *et al.*, 1998; Parsch & Engels, 2002). For example, 1-[2-deoxy-β-D-*erythro*-pentofuranosyl]-4-methyl-1*H*-benzimidazole, (I), reported by Guckian *et al.* (1998), is a close mimic of 2′-deoxyadenosine and was shown to be selectively inserted into DNA pairing with 2′-deoxythymidine. At the same time, some benzimidazole nucleosides are known for their antiviral activities (Zou *et al.*, 1996, 1997). The unique structural features and interesting biological activities of benzimidazole nucleosides have rendered them attractive targets for structure analysis. The title compound, (II), an analog of adenosine (III), was prepared for further study of RNA, as well as for a pharmaceutical test. We report here the crystal structure of 7-methyl-1-(β-D-*erythro*-pentofuranosyl)-1*H*-benzimidazol-2(3*H*)-one, (II).



(I)



(II)



(III)

A perspective view of (II) with the atom-labeling scheme is shown in Fig. 1. The bond lengths in the sugar moiety are in agreement with those found in adenosine (Lai & Marsh, 1972), within experimental error (Table 1). In the furanose ring, atom C10 is displaced by 0.538 (3) Å on the same side of the C9/O2/C12/C11 plane as atom C13. Thus, the furanose ring is in a C2′-*endo* conformation compared to the C3′-*endo* conformation observed in adenosine. The orientation of the benzimidazole moiety with respect to the sugar moiety is represented by the O2—C9—N2—C1 torsion angle about the glycosidic bond and the torsion angle value of 69.80 (19)° suggests that the

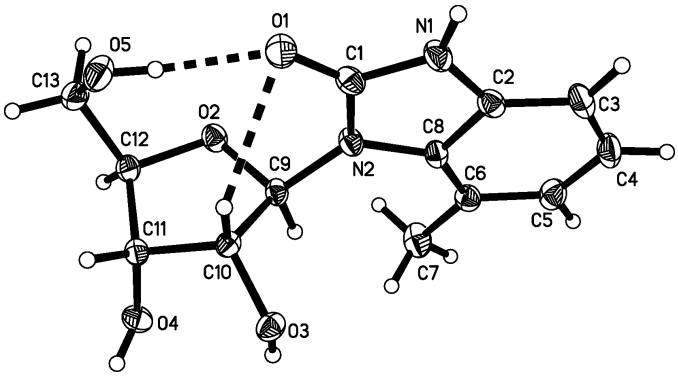


Figure 1
The structure of (II), showing 30% probability displacement ellipsoids and the atom-labeling scheme. Hydrogen bonds are shown as dashed lines.

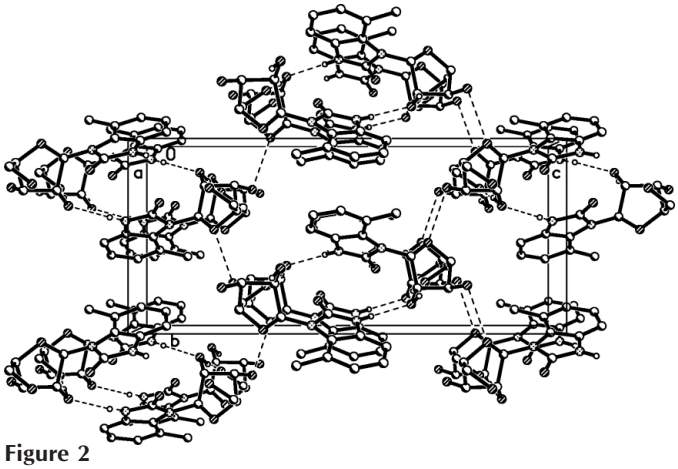


Figure 2
Packing of molecules in (II), viewed along the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

glycosidic bond rotation is mid-*anti*. The dihedral angle between the C9/O2/C12/C11 plane and the benzimidazole ring system is 84.83 (6)°.

Atom O1 forms two intramolecular hydrogen bonds, *viz*. C10—H10···O1 and O5—H5···O1. O—H···O and N—H···O intermolecular hydrogen bonds (Table 2) link the molecules into a three-dimensional network (Fig. 2).

Experimental

4-Methylbenzimidazolone was prepared as described by Harrison *et al.* (1963) and silylated by a procedure similar to that described by Nishimura & Iwai (1964). For the preparation of compound (II) (Niedballa & Vorbruggen (1974), silylated 4-methylbenzimidazolone (3.8 mmol) and 1-*O*-acetyl-2,3,5-tri-*O*-benzoyl-β-D-ribofuranose (3.6 mmol) were dissolved in acetonitrile (30 ml) and a mixture of tin tetrachloride (0.84 ml) in acetonitrile (18 ml) was added dropwise with stirring under a nitrogen atmosphere at room temperature. The solution was stirred overnight at room temperature. Sodium bicarbonate (7.5 g) in water (50 ml) was added and stirring continued for 1 h. The resulting solution was extracted three times with chloroform, the organic layers were collected, dried over anhydrous sodium sulfate, filtered, and evaporated to give a solid. The solid was dissolved in methanol (40 ml) and the solution was saturated with ammonia gas and stirred overnight in a sealed container. The resulting solution was evaporated to dryness to give the crude product. The crude product was purified by silica-gel column chromatography using chloroform–methanol (10:1 *v*/*v*) as eluent. Single crystals suitable for X-ray diffraction measurements were obtained from water by slow evaporation at room temperature.

Crystal data

$C_{13}H_{16}N_2O_5$
$M_r = 280.28$
Orthorhombic, $P2_12_12_1$
$a = 6.741$ (4) Å
$b = 9.119$ (5) Å
$c = 20.465$ (11) Å
$V = 1258.0$ (11) Å^3
$Z = 4$
$D_x = 1.480$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2668 reflections
$\theta = 2.4$–$26.8°$
$\mu = 0.12$ mm^{-1}
$T = 293$ (2) K
Plate, colourless
0.38 × 0.22 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.940$, $T_{max} = 0.989$
8479 measured reflections
1775 independent reflections
1496 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 28.1°$
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 12$
$l = -24 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.080$
$S = 1.09$
1775 reflections
185 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C1	1.232 (2)	N2—C1	1.390 (2)
O2—C9	1.433 (2)	N2—C8	1.416 (2)
O2—C12	1.465 (2)	N2—C9	1.443 (2)
O3—C10	1.406 (2)	C9—C10	1.535 (2)
O4—C11	1.422 (2)	C10—C11	1.527 (2)
O5—C13	1.413 (2)	C11—C12	1.521 (2)
N1—C1	1.349 (2)	C12—C13	1.512 (3)
N1—C2	1.390 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O3^{i}	0.86	2.06	2.876 (3)	157
O3—H3···O5^{ii}	0.82	1.94	2.725 (2)	160
O4—H4···O2^{iii}	0.82	2.01	2.830 (2)	173
O5—H5···O1	0.82	1.87	2.676 (2)	168
C10—H10···O1	0.98	2.40	3.072 (3)	125

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å, and with U_{iso}(H) = 1.5U_{eq}(carrier atom) for methyl and hydroxy H atoms and 1.2U_{eq}(carrier atom) for other H atoms. The methyl group was allowed to rotate freely about the C—C bond. In the absence of significant anomalous scattering, Friedel equivalents were merged before the final refinement.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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