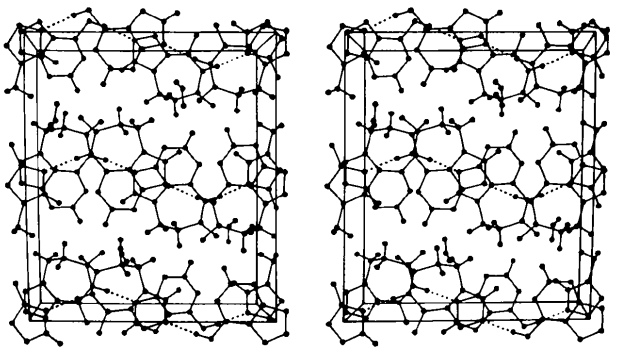

Fig. 2. Stereoscopic diagram showing the molecular packing viewed along the *b* axis. For the origin at the upper rear left-hand corner, **a** is to the right, **c** is down and **b** is out of the paper.

Table 2. *Hydrogen-bond geometry*

N(6)···N(9^{i})	2·967 (3) Å	H(6)···N(9^{i})	2·035 (23) Å
C(6)–N(6)···N(9^{i})	107·0 (1)°	H(6)–N(6)···N(9^{i})	6·4 (14)°
C(13)–N(6)···N(9^{i})	124·6 (1)	N(6)···N(9^{i})···H(6)	2·9 (6)
N(6)···N(9^{i})–C(4^{i})	144·8 (1)	H(6)···N(9^{i})–C(4^{i})	143·7 (7)
N(6)···N(9^{i})–C(8^{i})	111·0 (1)	H(6)···N(9^{i})–C(8^{i})	112·3 (7)
N(6)–H(6)···N(9^{i})	170·6 (20)		

Symmetry code: superscript: none x,y,z; (i) $-\frac{1}{2}+x,\frac{1}{2}-y,1-z$.

of C(6)–N(6)–C(13)–O(13) being 175·8 (2)°. Although this conformation is similar to that of ε-caprolactam (Winkler & Dunitz, 1975), shortening of C(13)–O(13) and lengthening of C(13)–N(6) are observed.

The crystal structure is shown in Fig. 2. Hydrogen-bond distances and angles are given in Table 2. The N(6)–H(6) group is the donor for a hydrogen bond to N(9) of the neighbouring molecule related by 2_1 symmetry along the *a* axis. The carbonyl oxygen, O(13), of the amide group does not participate in hydrogen bonding.

Parallel adenine moieties approach each other across inversion centres. The spacings are 3·323 and 3·226 Å. For the latter spacing, the shortest contact (3·258 Å) is between C(4) and C(8).

Figs. 1 and 2 were drawn by *TSD*: *XTAL*, which is a computer-graphics interactive modelling program for the Nova 3 computer (Takenaka & Sasada, 1978). The present work was supported by a Grant-in-Aid for Scientific Research from the Ministry of Education and by the Kawakami Foundation, to which the authors' thanks are due.

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5,6-Dimethyl-1-(α-D-ribofuranosyl)benzimidazole

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Abstract. $C_{14}H_{18}N_2O_4$, $M_r = 278{\cdot}31$, monoclinic $P2_1$, $a = 10{\cdot}849\ (3)$, $b = 5{\cdot}460\ (3)$, $c = 11{\cdot}717\ (6)$ Å, $\beta = 103{\cdot}94\ (1)°$, $Z = 2$, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å. Final $R = 0{\cdot}067$ for 1241 diffractometer data. The molecule is in the *syn* conformation. The ribose moiety has the C(3′)-*endo*, $^3T_2$ conformation. The staggered conformation about C(4′)–C(5′) is *trans*. The bases form a stacked column along **b** with an interplanar base separation of 5·460 Å. The molecules are connected by O(2′)–H(O2′)···N(9) [2·701 (6) Å] hydrogen bonds.

Introduction. $C_{14}H_{18}N_2O_4$ (ribazole) is of biological interest since its nucleotide counterpart occurs as a fragment of vitamin B_{12}. The aim of this structure deter-

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mination was to obtain information on molecular dimensions and conformation, and to compare it with the original fragment in vitamin B_{12}.

The intensities were collected on a Philips PW 1100 computer-controlled four-circle diffractometer in the ω-scan mode (scan width = 1·2° θ, scan speed = 0·03° θ s^{-1}) with graphite-monochromated Cu $K\alpha$ radiation. 1241 independent reflexions were observed ($I \geq 2\sigma$) and used in the calculations. The data were corrected for Lorentz and polarization factors.

The structure was solved with *MULTAN* (Declercq, Germain, Main & Woolfson, 1973). The solution was based on 300 reflexions with $|E| \geq 1{\cdot}2$. The E map corresponding to the solution with the best figure of merit revealed the positions of all non-hydrogen atoms.

Table 1. *Positional parameters* (×10⁴) *for non-hydrogen atoms*

	x	y	z
O(1′)	8474 (4)	224 (13)	2842 (4)
O(2′)	7775 (4)	−3286 (12)	727 (4)
O(3′)	10289 (4)	−3017 (16)	1128 (5)
O(5′)	10467 (4)	−723 (15)	4865 (4)
N(7)	6388 (4)	149 (0)	1662 (4)
N(9)	4290 (5)	861 (0)	1043 (5)
C(1)	6308 (5)	−3488 (16)	2976 (5)
C(2)	5825 (5)	−1657 (15)	2173 (5)
C(3)	4517 (5)	−1266 (15)	1767 (5)
C(4)	3659 (5)	−2762 (16)	2141 (5)
C(5)	4116 (6)	−4638 (17)	2934 (6)
C(6)	5443 (6)	−4963 (16)	3366 (5)
C(8)	5419 (6)	1602 (17)	1005 (6)
C(10)	3181 (7)	−6306 (18)	3330 (7)
C(11)	5933 (7)	−6949 (17)	4250 (6)
C(1′)	7706 (6)	696 (16)	1686 (6)
C(2′)	8250 (5)	−872 (17)	835 (6)
C(3′)	9641 (6)	−1031 (19)	1512 (6)
C(4′)	9512 (5)	−1346 (17)	2781 (6)
C(5′)	10665 (6)	−545 (21)	3701 (6)

Table 2. *Positional* (×10³) *and isotropic thermal* (×10²) *parameters for hydrogen atoms*

	x	y	z	U (Å²)
H(1)	722	−419	347	3·1
H(4)	278	−250	176	3·3
H(8)	556	344	82	3·6
H(10)1	222	375	309	4·8
H(10)2	315	375	412	4·8
H(10)3	333	200	309	4·8
H(11)1	590	281	494	3·9
H(11)2	685	313	435	3·9
H(11)3	574	160	388	3·9
H(1′)	778	272	162	3·7
H(2′)	796	0	0	4·4
H(3′)	1018	31	147	4·4
H(4′)	926	−300	294	3·8
H(5′)1	1148	−156	368	5·1
H(5′)2	1100	138	355	5·1
H(O2′)	722	−345	−20	3·6
H(O3′)	964	−375	73	5·3
H(O5′)	1115	−62	545	5·6

A full-matrix least-squares procedure minimizing $\sum w(|F_o| - |F_c|)^2$ with $w = 1/\sigma^2_{F_o}$ was used for the refinement. Heavy-atom coordinates, isotropic thermal parameters and a scale factor were refined to R = 0·113. The H atoms were located from a difference synthesis after anisotropic refinement (R = 0·085). In the final cycle one scale factor, the atomic positional parameters and the anisotropic thermal parameters for non-hydrogen atoms were varied. The H atom positions were kept fixed and isotropic thermal parameters were those of the atoms to which they were bonded. The final R = 0·067 and R_w = 0·068.

Scattering factors given by Cromer & Mann (1968) and (for H) by Stewart, Davidson & Simpson (1965) were used. The calculations were carried out on the Univac 1110 computer at the University Computing Centre in Zagreb with XRAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Positional parameters are listed in Tables 1 and 2.*

Discussion. The atom numbering and bond lengths are shown in Fig. 1, molecular packing and hydrogen bonds in Fig. 2 and base stacking in Fig. 3. Interatomic angles are listed in Table 3. The α-D-5,6-dimethylbenzimidazole ribonucleoside appears as a fragment of vitamin B_{12}. Thus the geometries of both molecules are compared. Displacements of atoms from the least-squares planes through the base and sugar in ribazole and in vitamin B_{12} are given in Table 4, torsion angles for both molecules in Table 5.

The benzimidazole moiety is planar within experimental error (Table 4). C(10) and C(11) deviate −0·005 and 0·113 Å from the ring, respectively. The angle between the plane of the base and that of the ribose is 71 (2)°. Bond distances (Fig. 1) and angles (Table 3) correspond to the values for the relevant type

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34113 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

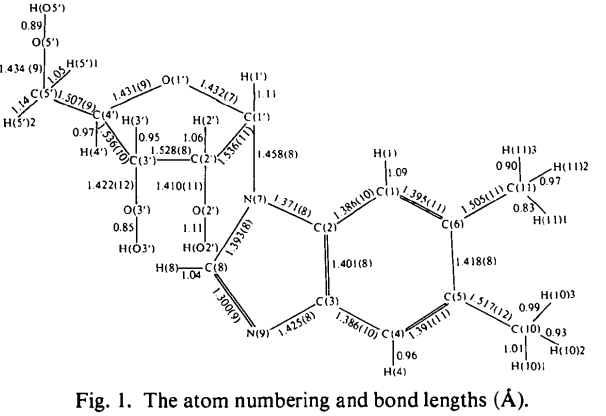

Fig. 1. The atom numbering and bond lengths (Å).

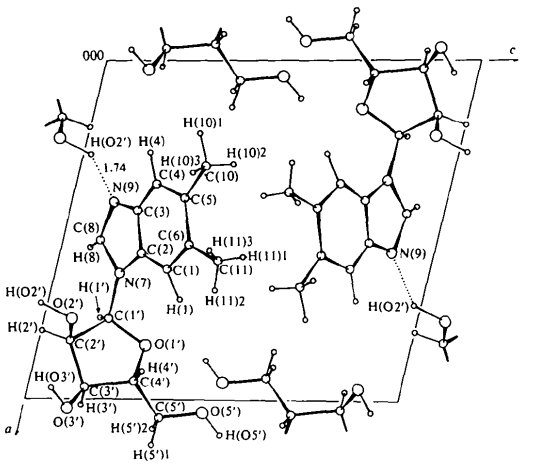

Fig. 2. A view of the crystal structure along **b** showing the packing arrangement and hydrogen bonds. A right-handed coordinate system has been chosen so that the positive direction of **b** is away from the reader.

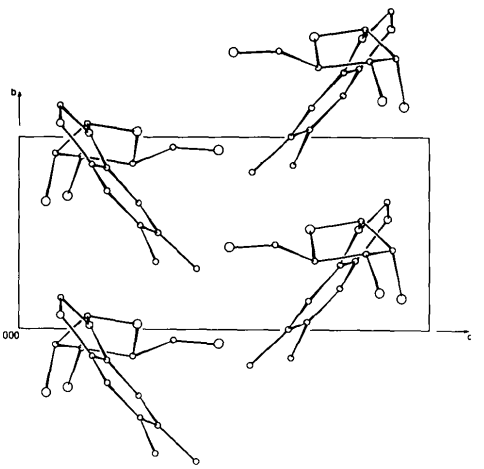

Fig. 3. The base stacking along **b** with interplanar base separation of 5·460 (3) Å.

Table 3. *Bond angles* (°)

C(2)–C(1)–C(6)	117·7 (5)	C(3)–N(9)–C(8)	104·2 (5)
C(1)–C(2)–C(3)	121·8 (6)	C(2')–C(1')–N(7)	113·8 (5)
C(1)–C(2)–N(7)	132·8 (5)	N(7)–C(1')–O(1')	109·0 (6)
C(3)–C(2)–N(7)	105·3 (6)	C(2')–C(1')–O(1')	106·7 (5)
C(2)–C(3)–C(4)	120·4 (6)	C(1')–C(2')–C(3')	100·4 (5)
C(2)–C(3)–N(9)	110·0 (6)	C(1')–C(2')–O(2')	112·9 (6)
C(4)–C(3)–N(9)	129·5 (5)	C(3')–C(2')–O(2')	106·7 (7)
C(3)–C(4)–C(5)	119·1 (5)	C(2')–C(3')–C(4')	101·5 (5)
C(4)–C(5)–C(6)	119·9 (7)	C(2')–C(3')–O(3')	112·4 (6)
C(4)–C(5)–C(10)	119·3 (6)	C(4')–C(3')–O(3')	112·8 (7)
C(6)–C(5)–C(10)	120·8 (7)	C(3')–C(4')–C(5')	114·0 (6)
C(1)–C(6)–C(5)	121·1 (7)	C(3')–C(4')–O(1')	103·7 (5)
C(1)–C(6)–C(11)	119·2 (6)	C(5')–C(4')–O(1')	108·6 (7)
C(5)–C(6)–C(11)	119·7 (7)	C(4')–C(5')–O(5')	111·8 (6)
N(7)–C(8)–N(9)	113·3 (6)	C(1')–O(1')–C(4')	110·2 (5)
C(2)–N(7)–C(8)	107·1 (5)		

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

Atoms included in calculation of least-squares plane are denoted by an asterisk.

	Ribazole		Ribazole moiety in vitamin B_{12}
Benzimidazole			
C(1)*	0·01 (1)		−0·02
C(2)*	−0·04 (1)		0·03
C(3)*	−0·04 (1)		0·02
C(4)*	−0·02 (1)		0·02
C(5)*	0·00 (1)		−0·03
C(6)*	0·04 (1)		−0·02
N(7)*	−0·02 (1)		0·09
C(8)*	0·03 (1)		−0·13
N(9)*	0·04 (1)		−0·05
Ribose			
C(1')*	0·00 (1)	C(1')*	0·00
C(4')*	0·00 (1)	C(4')*	−0·01
O(1')*	0·00 (1)	O(1')*	0·01
C(2')	−0·12 (1)	C(3')*	−0·00
C(3')	0·54 (1)	C(2')	0·74
C(5')	0·90 (1)	C(5')	−0·96

Table 5. *Torsion angles* (°) *describing the conformation of the ribazole molecule and the ribazole moiety in vitamin* B_{12}

Greek letters correspond to Sundaralingam's (1975) notation. The right-hand convention for the torsion angles is used throughout.

		Ribazole	Ribazole moiety in vitamin B_{12}
χ	O(1')–C(1')–N(7)–C(8)	142·7 (7)	−46
τ_0	C(4')–O(1')–C(1')–C(2')	4·5 (8)	28
τ_1	O(1')–C(1')–C(2')–C(3')	−28·3 (8)	−46
τ_2	C(1')–C(2')–C(3')–C(4')	39·7 (8)	46
τ_3	C(2')–C(3')–C(4')–O(1')	−38·4 (8)	−31
τ_4	C(3')–C(4')–O(1')–C(1')	21·3 (8)	1
	O(1')–C(1')–N(7)–C(2)	−38·1 (9)	165
	C(2')–C(1')–N(7)–C(2)	81·0 (9)	−87
	C(2')–C(1')–N(7)–C(8)	−98·2 (7)	61
	O(2')–C(2')–C(3')–O(3')	42·4 (8)	34
	O(5')–C(5')–C(4')–O(1')	61·2 (9)	−43
ψ	O(5')–C(5')–C(4')–C(3')	176·1 (8)	74
	N(7)–C(1')–C(2')–O(2')	−35·4 (7)	−30
	N(7)–C(1')–C(2')–C(3')	−148·7 (6)	−149
	N(7)–C(1')–O(1')–C(4')	127·8 (6)	139
ψ'	C(5')–C(4')–C(3')–O(3')	83·3 (9)	84
	C(5')–C(4')–C(3')–C(2')	−156·1 (8)	−161
	C(5')–C(4')–O(1')–C(1')	142·8 (7)	127

of hybridization and are also in agreement with those found in other compounds, *e.g.* 2-chloro-1-(β-D-ribofuranosyl)benzimidazole (Sprang & Sundaralingam, 1973) and the ribazole moiety in vitamin B_{12} (Brink-Shoemaker, Cruickshank, Crowfoot-Hodgkin, Kamper & Pilling, 1964).

The ribose appears in the C(3′)-*endo* mode of puckering (Table 4) and is in the asymmetrical $^3T_2$ twist conformation having a phase angle of pseudorotation P = 12·4° (Sundaralingam, 1975). The staggered conformation about the exocyclic C(4′)–C(5′) is *trans* (ψ = 176·1°).

The ribose moiety in the analogous nucleotide fragment in vitamin B_{12} exhibits a C(2′)-*exo*, $_2E$ envelope conformation (Tables 4 and 5). The phase angle of pseudorotation P of 343·3° also confirms this type of conformation. The conformation about C(4′)–C(5′) is *gauche*$^+$ (Table 5).

The orientation of the base relative to the sugar ring, described in terms of rotation about C(1′)–N(7) for the sequence O(1′)–C(1′)–N(7)–C(8), is 142·7° (Sundaralingam, 1975) and the molecule is *syn*. The α-D-5,6-dimethylbenzimidazole ribonucleotide moiety (in vitamin B_{12}) with χ_{CN} −46° (Table 5) occurs in the *anti* conformation.

The molecules are connected by hydrogen bonds O(2′)–H(O2′)···N(9), 2·701 (6) Å, acting between sugar and base moieties. The pronounced base stacking appears in the lattice along **b** with an interplanar separation of 5·460 (3) Å.

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2-Ethoxy-1,7,9-triethyl-7,9-dihydro-1*H*-purine-6,8-dione; a Tetraethyl Derivative of Uric Acid

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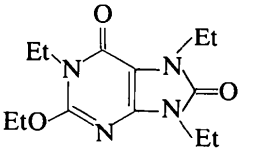
Abstract. $C_{13}H_{20}N_4O_3$, M_r = 280·33; monoclinic, $P2_1/n$; a = 15·491 (6), b = 4·794 (2), c = 19·597 (5) Å, γ = 95·26 (3)°, Z = 4, D_c = 1·285, D_m = 1·28 Mg m^{-3} (flotation); final R = 0·048 (852 reflections). Uric acid was ethylated and four isomers of tetraethyluric acid were isolated. One isomer, m.p. 387·2–387·7 K, was analyzed.

Introduction. For a quantitative measurement of uric acid in sera by isotope-dilution mass spectrometry, a stable derivative of uric acid was required. The title compound (I) was prepared by ethylating uric acid with triethylphenylammonium carbonate and purified by preparative thin-layer chromatography. As the structure of the title compound could not be determined with certainty by standard chemical techniques, it was solved by single-crystal X-ray diffraction.



(I) m.p. 387·2–387·7 K

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