only close contact occurs between C(13), C(7), and C(8) of a symmetry-related molecule. A view of the crystal structure down the b axis is shown in Fig. 6.

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Molecular and Crystal Structure of the tRNA Minor Constituent Dihydrouridine

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The tRNA minor constituent dihydrouridine crystallized from aqueous n-butanol as needles in the space group $P2_12_12_1$ with $a = 11\cdot779$, $b = 8\cdot150$, $c = 23\cdot068$ Å, and two molecules of the nucleoside plus one molecule of water in the asymmetric unit. The structure was solved by direct methods using the tangent formula and refined to an R value of 5·1 %. The oxygen atom O(5′) of one molecule is threefold disordered with the occupational parameters 0·5:0·25:0·25 but generally bond distances and angles are similar in both molecules. Owing to the saturated C(5)–C(6) bond the two nucleobases are puckered so that atom C(6) is on opposite sides of the plane through atoms N(1), C(2), N(3), C(4), i.e. the two nucleosides within an asymmetric unit are conformational isomers. Both molecules exhibit the usual anti conformation; the conformation of the ribose residues is C(2′)-endo. The packing of the molecules in the crystal is determined by a network of numerous hydrogen bonds and shows no base stacking.

Introduction

Transfer ribonucleic acids (tRNA's) are macromolecules of molecular weight 25000 to 30000 consisting of about 75 to 85 nucleosides. These nucleosides are not only adenosine, guanosine, uridine, cytidine but about 10 % are modified nucleosides such as dihydrouridine. Since tRNA's are synthesized in the cell on DNA-cistrons it is generally assumed that the 'virgin' tRNA does not contain modified nucleosides a priori but that

the modification takes place enzymatically at a later stage. It is not clear whether the occurrence of dihydro-uridine in tRNA's is a result of the hydrogenation of uridine or of cytidine with subsequent deamination (Zachau, 1970). In the cloverleaf model which was proposed by Holley, Apgar, Everett, Madison, Mar-quisee, Merrill, Penswick & Zamir (1965) for the sec-ondary structure of tRNA's, dihydrouridine is a reg-ular constituent of the 'dihydrouridine loop' (for an exception see Doctor, Loebel, Sodd & Winter, 1969) but it also occurs in other non-helical, non-base-paired regions (Zachau, 1965). Since the role of the modified nucleosides in tRNA's is still uncertain we undertook the X-ray analysis described below to contribute to an understanding of the structural features of dihydro-uridine.

Experimental

Dihydrouridine (purchased from P-L Biochemicals, Inc. Milwaukee, Wisconsin, U.S.A.) crystallized as needles when a hot, concentrated aqueous n-butanol solution was allowed to cool down slowly. The crystal-lographic data presented in Table 1 were obtained from Weissenberg and precession photographs and measure-ments on a four circle Siemens diffractometer. The density of the crystals as determined by the flotation method with a methyl iodide/toluene mixture is in agreement with the density calculated from the crys-tallographic data only if one assumes the asymmetric unit to consist of two molecules of dihydrouridine plus one of water.

Using the five-point method in which the intensity of every reflexion is measured twice, we collected 2335 data with Ni-filtered Cu $K\alpha$ radiation in the θ, 2θ scan mode. The intensity measurements were averaged and corrected for geometrical factors but not for absorp-tion due to the small linear absorption coefficient.
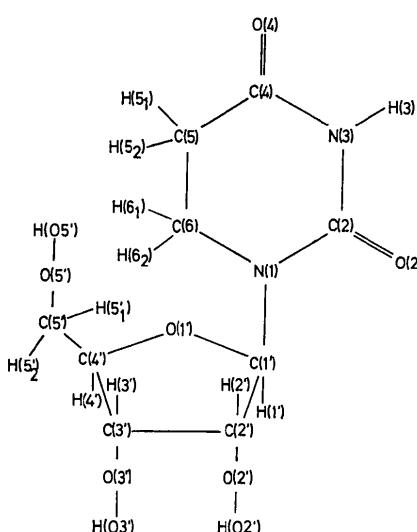

Fig. 1. Chemical structure and numbering scheme for dihy-drouridine.

Table 1. Crystallographic data

Space group $P2_12_12_1$, $Z=8$
Systematic extinctions when $h00$, $0k0$, $00l$ were odd.

$a = 11.779 \pm 0.003$ Å
$b = 8.150 \pm 0.002$
$c = 23.068 \pm 0.006$

Chemical formula $C_9H_{14}N_2O_6 \cdot \frac{1}{2}H_2O$

Density observed (toluene/CH$_3$I) 1.51 g.cm^{-3}
Density calculated ($Z=8$) 1.53 g.cm^{-3}

Linear absorption coefficient (Cu $K\alpha$)$\mu = 11.4$ cm^{-1}
Size of crystal $0.23 \times 0.38 \times 0.15$ mm

Solution and refinement of the structure

An overall temperature ($B = 3.26$ Å^2) and scale fac-tor were evaluated (Wilson, 1942) and used to com-pute normalized structure factors (E's; Karle & Haupt-man, 1956). The E-statistics suggested an acentric struc-ture (Computing Methods in Crystallography, 1965).

The structure was solved by direct methods through application of the tangent formula in the form pro-grammed by Main, Woolfson & Germain (1970) and by Zechmeister (1969). Of the 200 greatest E's the triple products were determined from which the reliabilities α_h (Karle & Karle, 1966) of the phases to be expected from the tangent formula refinement were estimated (Germain, Main & Woolfson, 1970). These α's were then used to select the best starting set by an iterative procedure ('convergence') and further starting phases were evaluated from a Σ_1 relationship (Hauptman & Karle, 1953).

The starting phases were

h	k	l	phase	
0	1	23	90°	
10	0	7	90°	to determine the origin
9	0	9	90°	
3	4	4	45°	to determine the enantiomorph
8	0	12	0°	these phases were varied by 90°
0	7	2	90°	(344) and 180° → 16 permuta-
9	1	0	90°	tions
6	0	0	0°	from Σ_J relationship
0	2	0	180°	

This phase information was extended by iterative (12 cycles) weighted tangent formula refinement to yield the phases of all 200 strongest E's. Since the phases of reflexions 8,0,12, 072, 910, 344 were assigned arbi-trarily they were allowed to vary in turn by 180° for the equatorial reflexions and by 90° for 344 and we ob-tained phase sets for all 16 possible combinations. One of these sets seemed to be the correct one, judging by probability characteristics such as the 'absolute figure of merit', the 'sum of the α's' and the 'sum of the resid-uals', and the phases of this set were further extended to yield the phases of the 300 strongest E's which were used to compute a Fourier synthesis.

This E map revealed the whole structure, but atoms

O(5') of molecule B and the water oxygen atom O(W) were weakly indicated. They were confirmed from an electron density distribution calculated from the 1600 strongest structure factors phased with the positional and overall isotropic temperature parameters of the 33 well-resolved atoms of the E map. A structure factor calculation based on the parameters of all the atoms yielded a residual $R = \sum ||F_{obs}| - |F_{cal}||/\sum|F_{obs}|$ of 19·1% for the 1600 strongest reflexions.

After four cycles of isotropic full-matrix least-squares refinement (Busing, Martin & Levy, 1962) the crystallographic reliability value, R was 11·2% for the 2143 significant reflexions. We applied a weighting scheme based on counter statistics (Stout & Jensen, 1968) and all data with $F_{obs} \leq 3·0$. σF_{obs} were treated as unobserved and not included in the refinement. A difference Fourier synthesis computed after the isotropic refinement provided us with the positions of 15 hydrogen atoms. Since the isotropic temperature factor of atom O(5') in molecule B was unusually high (13·4 Å²) a further isotropic refinement cycle and difference Fourier synthesis were computed but with the 15 hydrogen atoms included and atoms O(5') and C(5') of molecule B omitted. This synthesis revealed the position of atom C(5') and three peaks of weights 0·5:0·25:0·25 (compared to other oxygen atoms) in locations which could

Table 2. *Atomic coordinates of dihydrouridine molecules A (above) and B (below) in fractions of the crystallographic axes and thermal parameters in the form* $T = \exp[-(+\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$

Estimated standard deviation separated by a slash.

ATOM	X	Y	Z	BETA(11)	BETA(22)	BETA(33)	BETA(12)	BETA(13)	BETA(23)
N(1)	.5375/2	.3450/4	.3815/1	.0038/2	.0093/6	.0009/1	-.0013/3	-.0002/1	.0006/1
C(2)	.6440/3	.4083/5	.3827/1	.0042/3	.0106/7	.0010/1	-.0008/4	-.0000/1	.0006/2
O(2)	.6842/2	.4974/4	.3453/1	.0054/2	.0178/6	.0015/1	-.0029/3	-.0003/1	.0025/2
N(3)	.7097/2	.3671/4	.0306/1	.0040/2	.0125/6	.0012/1	-.0010/3	-.0004/1	.0011/2
C(4)	.6730/3	.2907/5	.4797/2	.0051/3	.0111/7	.0031/1	-.0003/4	-.0003/1	.0004/2
O(4)	.7390/2	.2690/4	.5203/1	.0066/2	.0231/8	.0013/1	-.0015/4	-.0011/1	.0020/2
C(5)	.5504/3	.2447/5	.4809/1	.0060/3	.0140/8	.0011/1	-.0015/4	-.0003/1	.0005/2
C(6)	.5065/3	.2124/5	.4204/1	.0055/3	.0114/7	.0011/1	-.0019/4	-.0002/1	.0011/2
C(1')	.4664/3	.3774/4	.3318/1	.0040/2	.0075/6	.0008/1	.0002/3	-.0000/1	.0002/2
C(2')	.4396/3	.2318/5	.2930/1	.0048/3	.0093/6	.0010/1	.0003/4	.0001/1	-.0005/2
O(2')	.5335/2	.1766/4	.2661/1	.0065/2	.0124/5	.0015/1	.0023/3	.0005/1	-.0009/1
C(3')	.3362/3	.2930/5	.2596/1	.0049/3	.0106/7	.0009/1	-.0012/4	-.0000/1	-.0003/2
O(3')	.3677/2	.3921/4	.2100/1	.0061/2	.0150/5	.0008/1	-.0001/3	.0002/1	.0001/1
C(4')	.2744/3	.3324/5	.3066/1	.0040/2	.0135/7	.0008/1	.0006/4	-.0001/1	.0002/2
C(5')	.1793/3	.2935/6	.3343/1	.0049/3	.0207/1	.0015/1	-.0019/5	.0007/1	-.0000/3
O(5')	.1346/2	.3720/4	.3841/1	.0053/2	.0250/7	.0014/1	.0033/4	.0009/1	.0014/2
O(1')	.3583/2	.4317/3	.3511/1	.0040/2	.0143/5	.0010/0	.0013/3	-.0001/1	-.0011/1
O(W)	.8861/4	.4362/6	.2680/2	.0170/5	.0352/12	.0032/1	.0010/7	.0010/2	.0000/3
H(3)	.2081	-.0856	.0711	.0034	.0082	.0010	.0000	.0000	.0000
H(5$_1$)	.0542	.3751	.4907	.0051	.0106	.0013	.0000	.0000	.0000
H(5$_2$)	.4723	-.1700	.0089	.0051	.0106	.0013	.0000	.0000	.0000
H(6$_1$)	.4272	.1938	.4167	.0044	.0092	.0012	.0000	.0000	.0000
H(6$_2$)	.4533	-.3992	.0900	.0044	.0092	.0012	.0000	.0000	.0000
H(1')	.4949	.4676	.3102	.0033	.0070	.0009	.0000	.0000	.0000
H(2')	.4141	.1219	.3117	.0040	.0083	.0010	.0000	.0000	.0000
H(3')	.3308	.2006	.2449	.0042	.0082	.0011	.0000	.0000	.0000
H(4')	.2374	.4900	.2922	.0046	.0085	.0011	.0000	.0000	.0000
H(5$_1$')	.1980	.1986	.3384	.0057	.0118	.0015	.0000	.0000	.0000
H(5$_2$')	.1215	.2563	.3074	.0057	.0118	.0015	.0000	.0000	.0000
H(O5')	.1777	.3370	.0161	.0052	.0252	.0020	.0000	.0000	.0000

ATOM	X	Y	Z	BETA(11)	BETA(22)	BETA(33)	BETA(12)	BETA(13)	BETA(23)
N(1)	.5484/2	.2368/5	.0160/1	.0042/2	.0175/7	.0010/1	.0010/3	.0003/1	.0004/2
C(2)	.4713/3	.2726/5	.0575/2	.0043/3	.0149/8	.0013/1	-.0005/4	.0002/1	.0007/2
O(2)	.3720/2	.3022/5	.0492/1	.0037/2	.0365/10	.0015/1	.0008/4	.0001/1	.0002/2
N(3)	.5133/2	.2800/4	.1139/1	.0044/2	.0138/6	.0010/1	-.0001/3	.0005/1	.0003/1
C(4)	.6255/3	.2957/5	.1288/2	.0049/3	.0100/7	.0013/1	.0304/4	.0001/1	.0006/2
O(4)	.6524/2	.3347/4	.1782/1	.0065/2	.0198/7	.0015/1	-.0004/3	-.0001/1	-.0009/2
C(5)	.7091/3	.2647/6	.0813/2	.0039/3	.0195/10	.0015/1	.0002/4	.0003/1	.0007/2
C(6)	.6568/3	.1633/6	.0335/2	.0056/3	.0180/9	.0011/1	.0025/5	.0004/1	.0004/2
C(1')	.5134/3	.2346/5	-.0440/2	.0049/3	.0141/8	.0012/1	-.0017/4	.0000/1	.0002/2
C(2')	.5921/3	.3223/5	-.0857/2	.0041/3	.0092/7	.0012/1	-.0000/3	.0000/1	.0005/2
O(2')	.5730/2	.4920/3	-.0862/1	.0038/2	.0101/5	.0017/1	.0004/3	-.0003/1	-.0000/1
C(3')	.5717/4	.2342/5	-.1434/2	.0072/4	.0113/8	.0013/1	.0008/4	.0002/1	-.0002/2
O(3')	.4819/3	.3096/4	-.1761/1	.0122/3	.0204/7	.0018/1	.0030/5	-.0018/1	-.0005/2
C(4')	.5374/4	.0614/6	-.1244/2	.0071/4	.0134/8	.0017/1	-.0012/5	.0003/2	-.0005/2
C(5')	.6254/6	-.0663/7	-.1323/3	.0183/9	.0154/11	.0052/2	.0027/9	.0050/4	.0009/4
O(5$_1$)	.6766/8	-.0995/10	-.1758/4	.0147/9	.0214/16	.0044/3	.0031/11	.0039/5	-.0015/6
O(5$_2$)	.7306/10	-.0316/18	-.1184/6	.0046/11	.0169/24	.0032/4	.0016/13	.0007/5	.0040/8
O(5$_3$)	.5862/28	-.0641/26	-.2070/12	.0285/31	.0218/40	.0065/10	-.0079/31	.0092/15	-.0062/16
O(1')	.5090/3	.0703/4	-.0637/1	.0133/4	.0127/6	.0015/1	-.0039/4	.0003/1	-.0000/2
H(3)	.4622	.2992	.1480	.0042	.0089	.0011	.0000	.0000	.0000
H(5$_1$)	.1948	-.2929	.4070	.0057	.0119	.0015	.0000	.0000	.0000
H(5$_2$)	.2758	-.1312	.4251	.0057	.0119	.0015	.0000	.0000	.0000
H(6$_1$)	.3437	-.4471	.4498	.0047	.0098	.0012	.0000	.0000	.0000
H(6$_2$)	.2079	.3149	.0034	.0047	.0098	.0012	.0000	.0000	.0000
H(1')	.0720	-.2649	.4517	.0048	.0100	.0012	.0000	.0000	.0000
H(2')	.1753	.2071	.0726	.0042	.0087	.0011	.0000	.0000	.0000
H(3')	.1393	.2876	.1707	.0057	.0120	.0015	.0000	.0000	.0000
H(4')	.0370	-.0596	.3638	.0052	.0108	.0013	.0000	.0000	.0000

be assigned to a disordered atom O(5'). In three cycles of anisotropic least-squares refinement the hydrogen atoms were assigned the isotropic temperature factors of the atoms to which they were bound covalently and the atomic parameters of all the non-hydrogen atoms and the occupational parameters of the disordered atom O(5') were varied. From a subsequent difference Fourier synthesis we were able to pick the positions of seven more hydrogen atoms but the hydrogen atoms attached to O(2') and O(3') in molecule A and to O(2'), O(3'), C(5'), O(5') in molecule B and to the water oxygen atom O(W) could not be located. Prior to the third cycle of refinement the reflexions 004, 012, 022, 101, 104, 020, 021, 200, 210, 211, 312 were corrected for secondary extinction (Stout & Jensen, 1968).

After two further cycles of anisotropic refinement the average parameter changes were less than one third of the standard deviations estimated from the correlation matrix. The final R value is 5·1% for the 2143 significant and 5·7% for all 2335 data.

Results and discussion

In Tables 2 to 7 we have listed the final atomic coordinates, intramolecular bond distances and angles (standard deviations are given in the Tables 2 and 3), the deviations of the atoms from least-squares planes through nucleobases and riboses, some dihedral angles, the geometrical data for the hydrogen bonds and the observed and calculated structure factors. In Figs. 2 to 5 are presented bond distances and angles averaged from the two molecules in the asymmetric unit together with comparative values, a schematic sketch of the pucker of the dihydrouracil residues, a thermal ellipsoids plot and the projection of the crystal structure along the b axis.
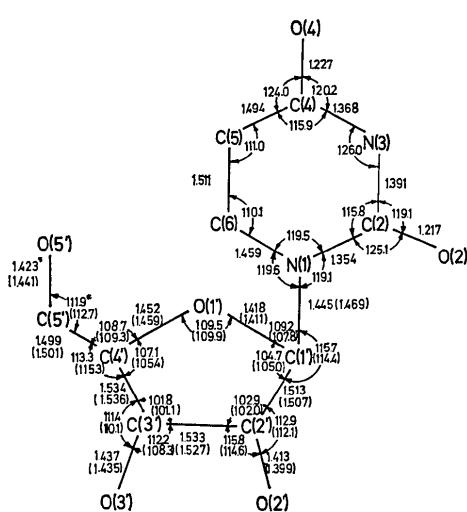
Fig. 2. Bond distances and angles averaged from molecules A and B. Hydrogen atoms are omitted. The figures in parentheses represent data averaged from several C(2')-*endo* ribose units (Saenger & Eckstein, 1970).

Table 3. *Bond distances and angles*

Estimated standard deviations for data not involving hydrogen atoms are 0·005 Å and 0·5°. In molecule B the standard deviations are 0·008 Å for C(4')-C(5'), 0·8° for C(3')-C(4')-C(5') and standard deviations for data involving O(5') are meaningless due to the disorder of this atom. Bond distances C(5')-O(5'₁) and C(5')-O(5'₂) are shorter than normal, a feature common to distances involving atoms with highly anisotropic thermal motion (Busing & Levy, 1964). C(5')-O(5'₃) however is too long for a C-O bond but position O(5'₃) was found in a difference Fourier synthesis and did not disappear in later refinement cycles.

	Bond lengths (Å)	
	Molecule *A*	Molecule A
N(1)—C(2)	1·356	1·351
C(2)—O(2)	1·223	1·210
C(2)—N(3)	1·389	1·393
N(3)—C(4)	1·364	1·371
C(4)—O(4)	1·229	1·225
C(4)—C(5)	1·492	1·495
C(5)—C(6)	1·512	1·510
C(6)—N(1)	1·450	1·467
N(1)—C(1')	1·444	1·445
C(1')—C(2')	1·510	1·516
C(2')—O(2')	1·424	1·401
C(2')—C(3')	1·534	1·531
C(3')—O(3')	1·435	1·438
C(3')—C(4')	1·538	1·529
C(4')—C(5')	1·518	1·480
C(5')—O(5')	1·423	
C(5')—O(5'₁)		1·20
C(5')—O(5'₂)		1·31
C(5')—O(5'₃)		1·78
C(4')—O(1')	1·461	1·442
O(1')—C(1')	1·420	1·415
N(3)—H(3)	1·04	1·00
C(5)—H(5₁)	1·18	1·25
C(5)—H(5₂)	0·78	0·88
C(6)—H(6₁)	0·95	0·98
C(6)—H(6₂)	1·05	1·06
C(1')—H(1')	0·95	1·04
C(2')—H(2')	1·03	1·05
C(3')—H(3')	0·93	1·03
C(4')—H(4')	0·97	0·92
C(5')—H(5'₁)	0·81	
C(5')—H(5'₂)	0·97	
O(5')—H(O5')	0·94	

	Bond angles (°)	
N(1)—C(2)—O(2)	124·7	125·4
N(1)—C(2)—N(3)	116·0	115·6
O(2)—C(2)—N(3)	119·2	118·9
C(2)—N(3)—C(4)	126·5	125·4
N(3)—C(4)—O(4)	119·9	120·4
N(3)—C(4)—C(5)	115·9	115·9
O(4)—C(4)—C(5)	124·2	123·7
C(4)—C(5)—C(6)	111·0	111·0
C(5)—C(6)—N(1)	110·8	109·4
C(6)—N(1)—C(2)	120·3	118·6
C(6)—N(1)—C(1')	118·7	120·5
C(2)—N(1)—C(1')	118·9	119·3
N(1)—C(1')—O(1')	109·2	109·2
N(1)—C(1')—C(2')	116·1	115·3
O(1')—C(1')—C(2')	103·9	105·4
C(1')—C(2')—O(2')	113·8	111·9
C(1')—C(2')—C(3')	102·1	103·6
O(2')—C(2')—C(3')	116·0	115·6
C(2')—C(3')—O(3')	112·5	111·8
C(2')—C(3')—C(4')	100·6	103·0
O(3')—C(3')—C(4')	112·3	110·5

Table 3 (cont.)

	Bond angles (°)	
	Molecule A	Molecule B
C(3′)–C(4′)–C(5′)	111·3	115·3
C(3′)–C(4′)–O(1′)	107·0	107·1
O(1′)–C(4′)–C(5′)	108·9	108·5
C(4′)–O(1′)–C(1′)	108·5	110·5
C(4′)–C(5′)–O(5′)	111·9	
C(4′)–C(5′)–O(5′₁)		127·7
C(4′)–C(5′)–O(5′₂)		118·7
C(4′)–C(5′)–O(5′₃)		86·0
C(2)—N(3)—H(3)	114	122
C(4)—N(3)—H(3)	120	112
C(4)—C(5)—H(5₁)	107	112
C(4)—C(5)—H(5₂)	100	104
H(5₁)–C(5)–H(5₂)	120	108
C(6)—C(5)—H(5₁)	100	109
C(6)—C(5)—H(5₂)	108	113
C(5)—C(6)—H(6₁)	116	103
C(5)—C(6)—H(6₂)	102	105
H(6₁)–C(6) –H(6₂)	107	118
N(1)–C(6)—H(6₁)	108	119
N(1)–C(6)—H(6₂)	113	102
N(1)–C(1′)–H(1′)	111	111
O(1′)–C(1′)–H(1′)	104	99
C(2′)–C(1′)–H(1′)	112	115
C(1′)–C(2′)–H(2′)	121	106
O(2′)–C(2′)–H(2′)	100	112
C(3′)–C(2′)–H(2′)	105	107
C(2′)–C(3′)–H(3′)	106	119
O(3′)–C(3′)–H(3′)	107	109
C(4′)–C(3′)–H(3′)	118	103
C(3′)–C(4′)–H(4′)	114	100
O(1′)–C(4′)–H(4′)	111	94
C(5′)–C(4′)–H(4′)	104	128
C(4′)–C(5′)–H(5′₁)	113	
C(4′)–C(5′)–H(5′₂)	112	
H(5′₁)–C(5′)–H(5′₂)	96	
O(5′₁)–C(5′)–H(5′₁)	111	
O(5′)–C(5′)–H(5′₂)	112	
C(5′)–O(5′)–H(O5′)	108	

Table 4. Least-squares planes through heterocycles and riboses

The plane equations are of the form $lX+mY+nZ+p=0$ where X, Y, Z are along a, b, c. Atoms which define the planes are marked by†.

(1) Heterocycles

Molecule A $l=-0·3146$ $m=0·575$ $n=0·4072$ $p=-4·022$
Molecule B $l=0·0301$ $m=0·9857$ $n=-0·1656$ $p=-2·069$

	Displacements (Å)	
	Molecule A	Molecule B
N(1)†	−0·019	−0·033
C(2)†	0·040	0·069
N(3)†	−0·042	−0·072
C(4)†	0·021	0·036
C(5)	0·166	−0·001
C(6)	−0·466	−0·652
O(2)	0·162	0·303
O(4)	0·006	0·171
C(1′)	0·004	0·166
C(2′)	−1·272	1·058
H(3′)	−0·03	−0·07
H(5₁)	−0·33	−0·50
H(5₂)	1·20	0·90
H(6₁)	−0·34	−1·60
H(6₂)	−1·49	−0·32

Table 4 (cont.)

(2) Ribose units

Molecule A $l=-0·1672$ $m=-0·8579$ $n=0·4859$ $p=-0·181$
Molecule B $l=0·9492$ $m=-0·1597$ $n=0·2712$ $p=-5·175$

	Displacements (Å)	
	Molecule A	Molecule B
C(1′)+	−0·019	−0·016
C(3′)+	0·017	0·015
C(4′)+	−0·029	−0·025
O(1′)+	0·030	0·026
C(2′)	0·625	0·489
C(5′)	1·157	1·075
O(2′)	0·449	0·051
O(3′)	−1·285	−1·292
O(5′)	1·258	
O(5′₁)		1·413
O(5′₂)		2·293
O(5′₃)		0·167

(a) Heterocycles

In contrast to pyrimidine nucleobases the dihydrouracil moiety is not planar owing to the saturation of the C(5)–C(6) double bond. The deviations of atoms C(5) and C(6) from the least-squares plane through the nearly coplanar atoms N(1), C(2), N(3) and C(4) are indicated in Fig. 3 and Table 4. In both nucleoside molecules atom C(6) is deviating from this plane more than atom C(5). The nucleobase in molecule A exhibits a half-chair conformation with atom C(6) and atom C(5) displaced by 0·466 and 0·166 Å on opposite sides of this plane, a geometry which is similar to the one observed in dihydrouracil (Rohrer & Sundaralingam, 1970) and in dihydrothymidine (Konnert, Karle & Karle, 1970). In molecule B, however, atom C(6) is deviating by 0·652 Å from this plane whereas C(5) is

Fig. 3. Schematic view along the N(1), C(2), N(3), C(4) plane through the dihydrouracil residues. Atom C(2′) is a reference point to demonstrate that the heterocycles are conformational isomers.

almost coplanar with it. These differences in the pucker of the two bases are also reflected in the absolute values of the dihedral angles N(3)–C(4)–C(5)–C(6) and C(2)–N(1)–C(6)–C(5) which differ by 7° for molecules A and

Table 5. *Some dihedral angles describing the conformation of dihydrouridine molecules*

These four-atom angles A–B–C–D are defined as zero if, when looking from B to C, bonds A–B and C–D are cisplanar and counted positive if the rear bond C–D is rotated clockwise with respect to the front bond A–B.

	Molecule A	Molecule B
C(2)–N(1)–C(6)–C(5)	+44·1	−51·0
N(3)–C(4)–C(5)–C(6)	+28·8	−21·1
C(4)–C(5)–C(6)–N(1)	−49·3	+50·4
C(2')–C(1')–N(1)–C(2)	+111·8	+134·2
C(2')–C(1')–N(1)–C(6)	−51·6	−60·6
O(1')–C(1')–N(1)–C(6)	+65·4	+57·8
O(1')–C(1')–N(1)–C(2)	−131·3	−107·4
O(2')–C(2')–C(3')–O(3')	−41·9	−32·8
O(5')–C(5')–C(4')–O(1')	−52·0	
O(5')–C(5')–C(4')–C(3')	−169·6	
O(5'$_1$)–C(5')–C(4')–O(1')		−173·7
O(5'$_2$)–C(5')–C(4')–O(1')		−77·3
O(5'$_3$)–C(5')–C(4')–O(1')		+162·0
O(5'$_1$)–C(5')–C(4')–C(3')		−53·6
O(5'$_2$)–C(5')–C(4')–C(3')		+42·7
O(5'$_3$)–C(5')–C(4')–C(3')		−78·0
C(1')–C(2')–C(3')–C(4')	−37·3	−28·7
C(2')–C(3')–C(4')–O(1')	+20·5	+15·6
C(3')–C(4')–O(1')–C(1')	−5·4	−4·8
C(4')–O(1')–C(1')–C(2')	−29·8	−23·6
O(1')–C(1')–C(2')–C(3')	+42·3	+32·5
N(1)–C(1')–C(2')–O(2')	−72·1	−81·9
N(1)–C(1')–C(2')–C(3')	+162·1	+153·0
N(1)–C(1')–O(1')–C(4')	−154·2	−148·0
C(5')–C(4')–C(3')–O(3')	+141·9	+135·3
C(5')–C(4')–C(3')–C(2')	−98·3	−105·2
C(5')–C(4')–O(1')–C(1')	+125·8	+129·8

B (Table 5) whereas the dihedral angle C(4)–C(5)–C(6)–N(1) is similar in both cases and comparable to the corresponding angle in dihydrouracil.

Table 6. *Geometrical data for hydrogen bonds and close intermolecular contacts*

Atom 1 in	Atom 2	in position			Distance (Å)
x y z					
N(3)A	O(2')B	$\frac{1}{2}-x$	$1-y$	$\frac{1}{2}+z$	2·832*
O(2')A	O(4)B	x	y	z	2·682
O(3')A	O(2')A	$1-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$	2·680
O(5')A	O(4)A	$-\frac{1}{2}+x$	$\frac{1}{2}-y$	$1-z$	2·774†
O(2')B	O(5')A	$\frac{1}{2}-x$	$1-y$	$-\frac{1}{2}+z$	2·771
O(3')B	O(W)	$\frac{1}{2}-x$	$1-y$	$-\frac{1}{2}+z$	2·893
O(5$_1$)B	O(3')A	$\frac{1}{2}+x$	$\frac{1}{2}-y$	$\bar{z}$	2·93
O(5$_2$)B	O(3')A	$\frac{1}{2}+x$	$\frac{1}{2}-y$	$\bar{z}$	2·91
O(5$_2$)B	O(2)B	$\frac{1}{2}+x$	$\frac{1}{2}-y$	$\bar{z}$	2·97
O(3')B	O(5$_3$)B	$1-x$	$\frac{1}{2}+y$	$-\frac{1}{2}-\bar{z}$	3·00
O(W)	O(5$_3$)B	$\frac{1}{2}-x$	$\bar{y}$	$\frac{1}{2}+z$	2·94
N(3)B	O(3')A	x	y	z	2·962‡

* Distance H(3)A···O(2')B 1·80 Å; angle H(3)A–N(3)A–O(2')B 6°

† Distance H(05')A···O(4)A 1·85 Å; angle H(05')A–O(5')A–O(4)A 8°

‡ Distance H(3)B···O(3')A 1·98 Å; angle H(3)B–N(3)B–O(3')A 9°.

It is interesting to note that in molecule A, C(6) is located on the same side of the plane through atoms N(1), C(2), N(3), C(4) as atom C(2') but on the opposite side in molecule B (Fig. 3), *i.e.* molecules A and B are conformational isomers. One has to assume that in solution as well these two modes of puckering of the dihydrouracil residue exist.

The four atoms comprising the N(1), C(2), N(3), C(4) planes and their substituents O(2) and O(4) should be
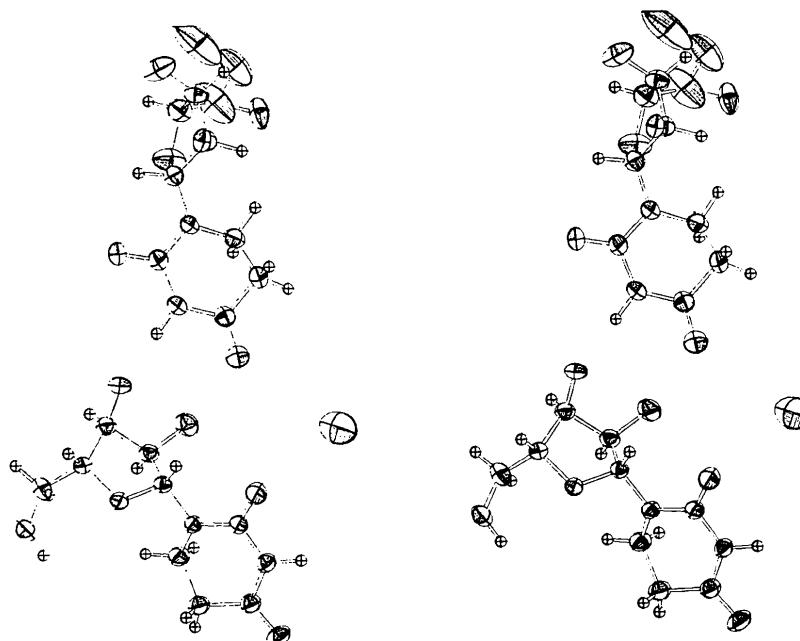
Fig. 4. Stereoscopic view along **b** of the 50 % probability thermal ellipsoids representing one asymmetric unit (Johnson, 1965.)

coplanar in order to allow optimum resonance. In both molecules however, atoms N(3) and C(2) are deviating from the least squares planes by about 0·07 Å and atom O(2) by as much as 0·3 Å. This non-planarity of the conjugated system of the dihydrouracil residue might in part be responsible for the association constant of the 1-cyclohexyldihydrouracil: 9-ethyladenine complex to be about one third the constant of the corresponding 1-cyclohexyluracil: 9-ethyladenine complex (Kyogoku, Loid & Rich, 1967). The diffeient association constants are due mainly to the pK-differences of the amino-N(3)-groups of the uracil residues; but since for optimum base-pairing a coplanar complex is required, the pucker of the dihydrouracil residue should further reduce the association constant of the corresponding complex with adenine.

Bond angles and distances are very similar in the two dihydrouracil residues except for bond N(1)–C(6) which differs by 0·017 Å. Both heterocycles exist in the usual diketo form, i.e. C(2)–O(2) and C(4)–O(4) have double bond character.

Comparable geometrical data have been reported for dihydrouracil (Rohrer & Sundaralingam, 1970). Due to the substitution at N(1) however, the N(1)–C(2) bond in the nucleoside is longer by 0·02 Å.

(b) Riboses

The riboses in molecules A and B exist in the envelope form with carbon atom C(2′) displaced by 0·625 and 0·489 Å respectively from the best least-squares plane through atoms C(1′), C(3′), C(4′) and O(1′) and on the same side as atom C(5′) (Table 4). Thus the ribose units are in the C(2′)-endo conformation. Most of the bond distances and angles are very similar in both ribose units and compare well with averaged data for C(2′)-endo riboses (Saenger, Eckstein,

1970); see Fig. 2. Significant differences between the dihydrouridine molecules occur only in the bonds C(2′)–O(2′) and O(1′)–C(4′) and the dihedral angle O(2′)–C(2′)–C(3′)–O(3′). The hydrogen bonds involving atoms O(2′) and O(3′) could be responsible for the relatively long C(2′)–O(2′) bond distance of 1·424 Å in molecule A [compared to othei C(2′)-endo nucleosides] and for the difference of about 10° in the O(2′)–C(2′)–C(3′)–O(3′) dihedral angles of the two molecules (Table 5). The differences around atom C(5′) have to be explained with the disorder of atom O(5′) in molecule B.

The conformation about the C(4′)–C(5′) bond is defined by the dihedral angles φ_{OO}, O(5′)–C(5′)–C(4′)–O(1′) and φ_{OC}, O(5′)–C(5′)–C(4′)–C(3′) (Shefter & Trueblood, 1965). These are $+52\cdot0°$ and $+169\cdot6°$ for molecule A and $-173\cdot7°$ and $-53\cdot6°$ for the most preferred location O(5′)₁ in molecule B, i.e. the conformations are gauche, trans and trans, gauche respectively. In most of the nucleosides investigated so far a gauche, gauche conformation was observed with O(5′) located above the ribose.

As in all pyrimidine nucleosides, except 4-thiouridine (Saenger & Scheit, 1970) the conformation about the glycosidic bond N(1)–C(1′) is anti in dihydrouridine, i.e. atom O(2) is pointing away from the ribose (Donohue & Trueblood, 1960). Owing to the pucker of the nucleobase the quantitative measure of this conformation should not be the angle C(2′)–C(1′)–N(1)–C(6) (Saenger & Scheit, 1970) but rather the angle [180° + C(2′)–C(1′)–N(1)–C(2)] which is $-68\cdot2°$ for molecule A and $-45\cdot8°$ for molecule B respectively (Table 5), i.e. both dihedral angles are in the anti range.

(c) Packing of the molecules and hydrogen bonding scheme

In Fig. 5 we have presented a projection of the crys-
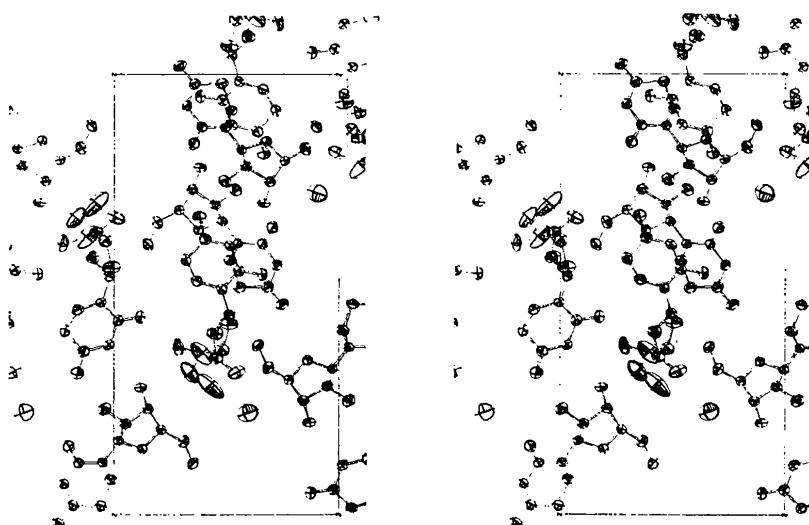
Fig. 5. Stereoscopic view along the b axis of the crystal structure. One should note the peculiar packing of the molecules around the disordered O(5′)-hydroxyl group in molecule B and how poorly the dihydrouracil residues of the molecules in the centre do overlap.

tal structure along the *b* axis. In contrast to the crystal structure of dihydrouracil (Rohrer & Sundaralingam, 1970) and other nucleoside constituents the nucleobases in this structure are hardly stacked. They are arranged at $c = 0$ and $c = \frac{1}{2}$ along the *a,b* plane and form a more hydrophobic region. In the more hydrophilic region

Table 7. *Observed and calculated structure factors for dihydrouridine multiplied by* 10

Unobserved data are marked by an asterisk.

Table 7 (*cont.*)

H	K	L	FOBS	FCAL
4	7	17	52	48
4	7	18	53	53
4	8	0	138	134
4	8	1	67	70
4	8	2	82	84
4	8	3	112	112
4	8	4*	19	22
4	8	5*	24	24
4	8	6	125	125
4	8	7	51	52
4	8	8	46	40
4	8	9	58	62
4	8	10	38	35
4	8	11	65	59
4	8	12	49	52
4	8	13	74	75
4	8	14*	16	5
5	0	1	499	523
5	0	2	462	465
5	0	3	347	323
5	0	4	363	363
5	0	5	146	160
5	0	6	653	[illegible]
5	0	7	556	659
5	0	8	65	59
5	0	9	135	128
5	0	10	29	32
5	0	11	192	182
5	0	12	345	333
5	0	13	235	243
5	0	14	31	7
5	0	15	234	215
5	0	16	192	178
5	0	17	236	210
5	0	18	133	128
5	0	19	53	40
5	0	20	285	275
5	0	21*	5	2
5	0	22	33	10
5	0	23	60	68
5	0	24	84	90
5	0	25	134	122
5	0	26*	53	49
5	1	0	140	126
5	1	1	598	590
5	1	2	258	269
5	1	3	182	151
5	1	4	127	123
5	1	5	222	214
5	1	6	434	421
5	1	7	537	537
5	1	8	26	21
5	1	9	152	145
5	1	10	112	119
5	1	11	126	123
5	1	12	155	134
5	1	13	139	125
5	1	14	224	212
5	1	15	59	50
5	1	16	190	190
5	1	17	53	39
5	1	18	158	174
5	1	19	93	97
5	1	20	79	76
5	1	21	114	127
5	1	22	28	19
5	1	23	43	44
5	1	24*	17	23
5	1	25	32	35
5	1	26	45	43
5	2	0	90	132
5	2	1	193	165
5	2	2	139	137
5	2	3	464	436
5	2	4	320	319
5	2	5	374	367
5	2	6	259	233
5	2	7	567	550
5	2	8	47	22
5	2	9	138	131
5	2	10	110	123
5	2	11	33	39
5	2	12	236	220
5	2	13	25	32
5	2	14	193	194
5	2	15	93	97
5	2	16	236	241
5	2	17	229	241
5	2	18	90	37
5	2	19	73	97
5	2	20	161	150
5	2	21	45	35
5	2	22	66	79
5	2	23	39	45
5	2	24	137	74
5	2	25	51	78
5	3	0	33	53
5	3	1	135	85
5	3	2	395	391
5	3	3	241	249
5	3	4	65	72
5	3	5	93	93
5	3	6	194	161
5	3	7	32	32
5	3	8	221	213
5	3	9	83	91
5	3	10*	0	24
5	3	11	132	139
5	3	12	334	341
5	3	13	381	352
5	3	14	48	57
5	3	15	273	262
5	3	16	61	57
5	3	17	75	34
5	3	18	59	85
5	3	19	148	147
5	3	20	145	145
5	3	21	41	46
5	3	22	125	128
5	3	23	43	28
5	3	24	23	22
5	4	0	131	125
5	4	1	68	67
5	4	2	76	73
5	4	3	225	219
5	4	4	156	155
5	4	5	117	125
5	4	6	132	97
5	4	7	94	116
5	4	8	97	95
5	4	9	79	[illegible]
5	4	10	146	162
5	4	11	157	154
5	4	12	155	162
5	4	13	76	36
5	4	14	51	40
5	4	15	156	141
5	4	16	92	61
5	4	17	111	122
5	4	18	58	66
5	4	19	45	44
5	4	20	74	78
5	4	21	83	37
5	4	22	133	97
5	4	23	53	59
5	5	0	94	71
5	5	1	133	133
5	5	2	154	157
5	5	3	179	152
5	5	4	46	51
5	5	5	106	99
5	5	6	171	163
5	5	7	303	303
5	5	8	64	64
5	5	9	76	74
5	5	10	87	80
5	5	11	137	131
5	5	12*	12	25
5	5	13	229	212
5	5	14	126	117
5	5	15	131	132
5	5	16*	14	7
5	5	17	139	139
5	5	18*	21	39
5	5	19	46	49
5	5	20	66	60
5	5	21	93	85
5	5	22	27	20
5	6	0	110	121
5	6	1	149	142
5	6	2	171	175
5	6	3	211	205
5	6	4	141	131
5	6	5	174	183
5	6	6	93	99
5	6	7	54	59
5	6	8	214	215
5	6	9	64	73
5	6	10	94	98
5	6	11	81	84
5	6	12	193	185
5	6	13*	24	36
5	6	14	47	54
5	6	15	62	66
5	6	16	53	52
5	6	17	33	29
5	6	18	97	100
5	6	19	37	27
5	6	20	22	20
5	7	0	81	85
5	7	1	144	142
5	7	2	173	187
5	7	3	165	163
5	7	4	65	70
5	7	5	142	161
5	7	6	112	114
5	7	7*	5	22
5	7	8	58	70
5	7	9	52	51
5	7	10	36	74
5	7	11	135	125
5	7	12	94	88
5	7	13	78	79
5	7	14*	17	8
5	7	15	64	87
5	7	16	79	78
5	7	17	37	36
5	8	0	19	18
5	8	1	60	71
5	8	2	77	82
5	8	3	125	117
5	8	4	145	143
5	8	5	137	96
5	8	6	31	32
5	8	7	52	45
5	8	8*	23	33
5	8	9	40	52
5	8	10	37	28
5	8	11	94	92
5	8	12	52	49
5	8	13	83	81
6	0	0	863	977
6	0	1	27	27
6	0	2	128	155
6	0	3	90	88
6	0	4	264	253
6	0	5	39	59
6	0	6	179	196
6	0	7	337	324
6	0	8	77	105
6	0	9	245	251
6	0	10	417	430
6	0	11	125	115
6	0	12	44	27
6	0	13	359	365
6	0	14	334	329
6	0	15	171	159
6	0	16	154	153
6	0	17	51	44
6	0	18	254	270
6	0	19	217	216
6	0	20	39	51
6	0	21	53	43
6	0	22	135	105
6	0	23	149	136
6	0	24	34	28
6	0	25	61	65
6	1	0	615	[illegible]
6	1	1	245	251
6	1	2	447	424
6	1	3	133	136
6	1	4	455	451
6	1	5	335	334
6	1	6	121	114
6	1	7	157	168
6	1	8	[illegible]	[illegible]
6	1	9	150	147
6	1	10	257	259
6	1	11	137	129
6	1	12	51	53
6	1	13	414	421
6	1	14	122	127
6	1	15	174	174
6	1	16	131	137
6	1	17	137	135
6	1	18	110	117
6	1	19	45	67
6	1	20	75	61
6	1	21	57	78
6	1	22	91	90
6	1	23	34	33
6	1	24	93	93
6	1	25	22	62
6	2	0	214	241
6	2	1	254	274
6	2	2	217	231
6	2	3	275	259
6	2	4	114	83
6	2	5	127	111
6	2	6	386	388
6	2	7	33	74
6	2	8	211	187
6	2	9	73	75
6	2	10	157	146
6	2	11	43	49
6	2	12	165	164
6	2	13	95	83
6	2	14	277	284
6	2	15	43	34
6	2	16	135	139
6	2	17	145	165
6	2	18	73	67
6	2	19	105	195
6	2	20	57	77
6	2	21	120	137
6	2	22	134	98
6	2	23	32	34
6	2	24	91	91
6	3	0	64	63
6	3	1	146	177
6	3	2	337	337
6	3	3	228	231
6	3	4	169	167
6	3	5	192	239
6	3	6	156	171
6	3	7	147	141
6	3	8	117	107
6	3	9	227	213
6	3	10	169	163
6	3	11	73	72
6	3	12	159	165
6	3	13	237	228
6	3	14	274	281
6	3	15	96	95
6	3	16	32	29
6	3	17	69	72
6	3	18	55	60
6	3	19	117	115
6	3	20	84	76
6	3	21	43	52
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10	6	7	50	57
10	6	8	76	75
10	6	9	33	31
10	6	10	78	77
10	7	0	26	23
10	7	1	68	41
10	7	2	102	98
10	7	3	61	63
11	0	1	269	231
11	0	2	33	29
11	0	3	47	60
11	0	4	59	57
11	0	5	72	66
11	0	6	278	287
11	0	7	165	175
11	0	8	50	70
11	0	9	62	71
11	0	10	61	57

close to the *a*,*b* plane at $c=\frac{1}{4}$ and $c=\frac{3}{4}$ there are numerous hydrogen bonds between ribose and water hydroxyl groups. In Table 6 the geometrical data for these hydrogen bonds are listed together with short intermolecular contacts between the disordered atom O(5′) of molecule *B* and oxygen atoms respectively. It might be due to these many short intermolecular O(5′) $\cdots$O contacts that atom O(5′) is disordered.

Table 7 (cont.)

The hydroxyl group O(2') of molecule A is involved in two short hydrogen bonds. It acts as an acceptor for atom O(3') of another molecule A related to the first one by the twofold screw axis parallel to **b** in $a=\frac{1}{2}$, $c=\frac{1}{4}$ and as a donor for atom O(4) of molecule B. By means of the screw operation a ribbon of hydrogen bonds is formed along the mentioned twofold screw axis. Atom O(5') in molecule A is the donor for a hydrogen bond of 2·774 Å to O(4) in a symmetry related molecule A and the acceptor for a hydrogen bond from O(2') of molecule B (2·771 Å).

Around the water molecule there are located atoms O(3') and the disordered O(5'$_3$) of molecule B. Another hydrogen bond exists between N(3) in molecule A to O(2') in molecule B and from N(3) in molecule B to O(3') in molecule A. Since in some cases the hydrogen atoms could not be traced from a difference Fourier synthesis, intermolecular distances close to or shorter than the sum of the van der Waals radii which are given in Table 6 served to indicate hydrogen bonds.

The computations were carried out on the IBM 360/91 computer at München/Garching, on the UNIVAC 1108 at Göttingen/Nikolausberg and on the IBM 7094 computer at Deutsches Rechenzentrum, Darmstadt.

We are pleased to acknowledge the interest and support of Professors F. Cramer and W. Hoppe. We thank Professor D. Mootz for making available his Siemens diffractometer for the data collection and Miss U. Wittenberg for skilful technical assistance.

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