gree of anisotropic vibration, assuming that systematic errors in the experimental data are insignificant.

Table 6. *Ellipsoids of vibration to the unit-cell axes (neutron refinement)*

	i	$(\overline{r_i^2})^{1/2}$	a_{ia}	a_{ib}	a_{ic}
Y(1)	1	0·078 Å	0·577	0·577	0·577
	2	0·040	0·816	−0·408	−0·408
	3	0·040	0·000	0·707	−0·707
Y(2)	1	0·082	0·000	0·403	0·915
	2	0·066	1·000	0·000	0·000
	3	0·047	0·000	0·915	−0·403
O	1	0·080	−0·468	0·837	−0·282
	2	0·060	0·355	0·471	0·808
	3	0·045	0·809	0·278	−0·518

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The Crystal Structure of 5-Methyluridine*

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5-Methyluridine was crystallized from aqueous ethanol as the hemihydrate in the form of orthorhombic needles, space group $P2_12_12$ with $a=14{\cdot}026$, $b=17{\cdot}302$, $c=4{\cdot}861$ Å and four molecules per cell. The crystal structure has been determined by a three-dimensional, X-ray diffraction analysis. Intensity data were collected on photographic film and estimated visually. The structure was solved from the Patterson function with the help of Fourier techniques and packing considerations. Refinement by a full-matrix, least-squares method has led to a final R value of 0·075 for 1294 reflections and to estimated standard deviations in bond lengths for the non-hydrogen atoms between 0·006 and 0·007 Å. The torsion angle describing the relative orientations of the sugar residue and the pyrimidine base is −29·4°. Atom C(3′) of the sugar residue is displaced by about 0·60 Å from the least-squares plane through the other four ring atoms and lies on the same side of the plane as C(5′). This plane also makes an angle of 71·9° with the least-squares plane through the six ring atoms of the pyrimidine base. Of the twelve atoms within, or bonded to, the pyrimidine ring, only C(1′) deviates significantly, by 0·083 Å, from the least-squares plane through the six ring atoms.

1. Introduction

The structure of 5-methyluridine (Fig. 1) has been determined as one in a series of compounds being studied in these laboratories to provide accurate bond lengths and angles and conformational information useful in structural studies of the nucleic acids.

As far as the authors are aware, ten X-ray analyses have been reported, to date, for uridine and thymidine derivatives (Huber, 1957; Trueblood, Horn & Luzzati, 1961; Harris & McIntyre, 1964; Shefter, Barlow, Sparks & Trueblood, 1964; Shefter & Trueblood, 1965; Camerman & Trotter, 1965; Haschemeyer & Sobell, 1965; Iball, Morgan & Wilson, 1966, 1968). The most accurate determinations seem to be for β-adenosine-2′-uridine-5′-phosphoric acid (Shefter *et al.*, 1964) and 5-fluorodeoxyuridine (Harris & MacIntyre, 1964) for which the estimated standard deviations (e.s.d.'s) are slightly larger than those reported here for 5-methyluridine.

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2. Experimental procedure

5-Methyluridine was obtained as a powder from Mann Research Laboratories Inc., New York, N.Y. and crystallized from aqueous ethanol as orthorhombic needles, elongated along **c**. Weissenberg photographs showed systematic absences only amongst $h00$ for $h=2n+1$ and $0k0$ for $k=2n+1$ indicating the space group to be $P2_12_12$.

Unit-cell parameters were obtained from Straumanis type Weissenberg photographs taken about the a and c axes. A least-squares routine minimizing

$$\sum_n w_n(\sin^2\theta_{\text{obs}}-\sin^2\theta_{\text{calc}})^2(4/\lambda^2)$$

was used, w_n, the weight of an observation, being proportional to $1/\sin^2 2\theta$. Systematic corrections for eccentricity and absorption were applied (Klug & Alexander, 1954) and the values obtained were:

$$\begin{aligned} a &= 14{\cdot}026 \pm 0{\cdot}003 \text{ Å} \\ b &= 17{\cdot}302 \pm 0{\cdot}001 \\ c &= \ \ 4{\cdot}861 \pm 0{\cdot}001\ . \end{aligned}$$

The indicated eccentricity and absorption corrections are small and the confidence limits cited are subjective, being approximately ten times the standard deviations obtained from the least squares. The calculated density, assuming four molecules of $C_{10}H_{14}N_2O_6$ and two molecules of water per unit cell is 1·504 g.cm^{-3}; the density measured by flotation is 1·503 g.cm^{-3}.

Intensity data were collected by the multi-film, equi-inclination Weissenberg technique with Cu $K\alpha$ radiation and Eastman No-screen film. Reflection intensities for layers $hk0$–$hk4$ and $0kl$–$5kl$ were estimated visually by comparison with calibrated 'wedges'. Two crystals were used, both cut from needles to give rough cubes of side 0·10 mm, though slightly elongated in the **c** direction. Empirical film factors (corrected for equi-inclination angle) ranged from 2·8 to 4·5; since experience in this laboratory has indicated an average value of 3·8 for this brand of film, this latter value (adjusted for equi-inclination angle) was used in scaling the observations together. The variation of the empirical film factors probably arose from a systematic under-estimation of the very strong intensities.

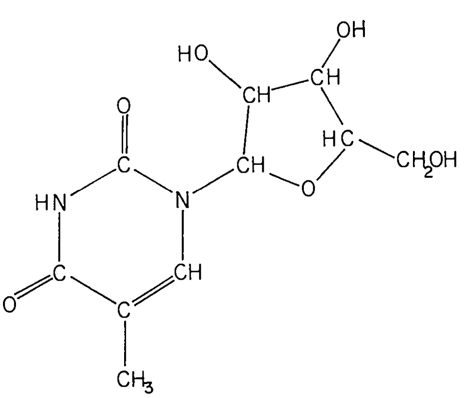

Fig. 1. 5-Methyluridine.

Each intensity reading was allotted a standard deviation according to the formulae

$$\sigma(I)=\sigma(\text{ext})\left\{\left[\frac{I_{\min}}{3}+bI+\left(\frac{0{\cdot}1I^2}{I_{\max}}-I\right)^2\right]\times\left[1+0{\cdot}25\exp\left[-50(0{\cdot}5-\sin^2\theta)^2\right]\right]\right\},$$

where $I_{\min}$ and $I_{\max}$ are the limits within which an intensity can be reliably estimated and b was determined from a statistical analysis of the consistency between reflections multiply observed in the sensitive region of the intensity 'wedge'. $\sigma(\text{ext})$ is a subjective parameter. The exponential term is designed to take account of α_1, α_2 splitting. The data were then corrected for Lorentz and polarization effects and placed on a common arbitrary scale after a least-squares determination of inter-layer scale factors. The final total of 1499 independent reflection intensities (of which 192 were too small to be observed) were then scaled absolutely by the method of Wilson (1942). In view of the size of the crystal and the fact that the calculated absorption coefficient is 11·1 cm^{-1} it was considered unnecessary to make absorption corrections. All calculations were carried out on an IBM 7094 computer using subprograms operating under the *CRYRM* system (Duchamp, 1964).

3. Structure determination

A three-dimensional, origin-removed Patterson function was computed with sharpened coefficients,

$$\left[\frac{(k|F_{\text{obs}}|)^2}{\exp(-2B\sin^2\theta/\lambda^2)\,.\sum n_if_i^2}-1\right].2^{16}.\left(\frac{\sin\theta}{\lambda}\right)^4 \times\exp\left(-19{\cdot}36\,\frac{\sin^2\theta}{\lambda^2}\right),$$

n_i being the number of atoms of type i having atomic scattering factor f_i and B the overall isotropic temperature factor. The approximate orientation of the pyrimidine base was determined from a visual investigation of the heights and positions of the peaks close to the origin of the Patterson function. The location of the base in the unit cell was determined from the Patterson projection, $P(u,v)$, where a large peak was to be expected corresponding to the 'ring–ring' vector from molecules related by the twofold axis. Of several possible peaks, one was selected that corresponded to reasonable packing conditions in the crystal and that best matched the three-dimensional Patterson function. This interpretation turned out to be correct.

Iterative Fourier methods then led to a complete trial structure with an agreement index of $R_{hk0}=0{\cdot}51$. Full-matrix least-squares refinement of the x and y coordinates, a scale factor, and in later cycles isotropic temperature parameters reduced R_{hk0} to 0·15. As expected from the density measurements the unit cell contains two water molecules, these being situated on the twofold axes at (0,0) and ($\frac{1}{2},\frac{1}{2}$).

The z coordinate of the oxygen atom of the water molecule was estimated from the Harker line $P(\frac{1}{2},\frac{1}{2},w)$. To determine trial z coordinates for the remaining atoms it was assumed that the nitrogen atom of the pyrimidine ring participated in a hydrogen bond with the oxygen atom at (0,0). From the Fourier projection it seemed that this geometry was favourable and so the molecule was adjusted in z until the oxygen atom of the water molecule was in the plane of the ring. At this stage a three-dimensional structure factor calculation gave an agreement index of $R_{hkl}=0{\cdot}19$.

4. Refinement

Atomic coordinates, a scale factor and isotropic temperature factors were refined with a full-matrix, least-squares routine minimizing

$$\Sigma\, w(|F_{\rm obs}|^2-1/k^2|F_{\rm calc}|^2)^2\,,$$

where k is the scale factor and $w=1/\sigma^2(|F_{\rm obs}|^2)$ was derived from $\sigma(I)$ defined in § 2. Refinement for three cycles reduced R to 0·15 and was then continued with anisotropic temperature factors of the form

$$\exp[-(h^2b_{11}+k^2b_{22}+l^2b_{33}+hkb_{12}+hlb_{13}+klb_{23})]\,.$$

Three further cycles reduced R to 0·10 whereupon difference Fourier syntheses were calculated in the planes where hydrogen atoms were expected to lie. Peaks ranging from 0·1 to 0·6 e.Å^{-3} were observed in reasonable positions for all hydrogen atoms; these positions with isotropic temperature factors of 4·0 Å^2 were included in subsequent structure-factor calculations but were not refined. For the hydrogen atoms, scattering

Table 1. *The heavy-atom parameters and their estimated standard deviations*

All entries have been multiplied by 10^4.

	X	Y	Z	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
C(5)	2669 (4) Å	1916 (3) Å	4207 (10) Å	29 (3)	19 (1)	288 (18)	9 (4)	−12 (13)	−22 (9)
C(6)	2397 (4)	2457 (3)	2379 (10)	27 (3)	17 (1)	342 (20)	−4 (3)	24 (13)	−35 (9)
N(1)	1537 (3)	2463 (2)	1087 (8)	31 (3)	15 (1)	291 (16)	−2 (3)	−8 (11)	−16 (7)
C(2)	0860 (4)	1905 (3)	1593 (11)	27 (3)	16 (1)	351 (22)	−1 (4)	−35 (14)	−18 (10)
N(3)	1132 (3)	1362 (2)	3509 (9)	31 (3)	16 (1)	436 (21)	−4 (3)	−15 (13)	25 (9)
C(4)	1998 (4)	1317 (3)	4868 (10)	32 (3)	17 (1)	381 (23)	8 (4)	−24 (14)	24 (9)
C(7)	3619 (5)	1924 (3)	5555 (11)	38 (3)	33 (2)	363 (24)	0 (5)	−50 (16)	40 (12)
O(1) (water)	0	0	5605 (11)	27 (3)	31 (2)	401 (24)	3 (5)	0	0
O(2)	0094 (3)	1900 (2)	0508 (8)	30 (2)	25 (1)	566 (20)	−9 (3)	−76 (13)	42 (9)
O(4)	2130 (4)	0795 (2)	6517 (9)	52 (3)	27 (1)	601 (24)	−3 (4)	−73 (15)	122 (9)
C(1′)	1262 (4)	3096 (3)	−0803 (10)	29 (3)	20 (1)	244 (17)	0 (4)	−47 (13)	−17 (9)
C(2′)	0751 (4)	3767 (3)	0639 (10)	29 (3)	19 (1)	305 (20)	0 (3)	−4 (13)	−14 (9)
O(1′)	2091 (3)	3400 (2)	−2039 (6)	43 (2)	18 (1)	247 (13)	3 (3)	54 (10)	−26 (6)
C(3′)	1583 (4)	4307 (3)	1177 (9)	31 (3)	19 (1)	205 (18)	7 (3)	−9 (12)	−8 (8)
C(4′)	2170 (4)	4221 (2)	−1391 (9)	41 (3)	16 (1)	198 (17)	2 (3)	−31 (13)	0 (7)
O(2′)	0100 (3)	4070 (2)	−1280 (8)	37 (2)	24 (1)	492 (19)	15 (3)	−124 (13)	−30 (8)
C(5′)	3207 (5)	4438 (3)	−1203 (12)	41 (4)	25 (2)	373 (24)	−1 (4)	67 (17)	26 (11)
O(5′)	3682 (3)	4102 (2)	1067 (8)	29 (2)	28 (1)	410 (17)	9 (3)	4 (11)	−29 (8)
O(3′)	1257 (3)	5072 (2)	1638 (7)	35 (2)	19 (1)	442 (17)	5 (3)	−18 (12)	−77 (7)

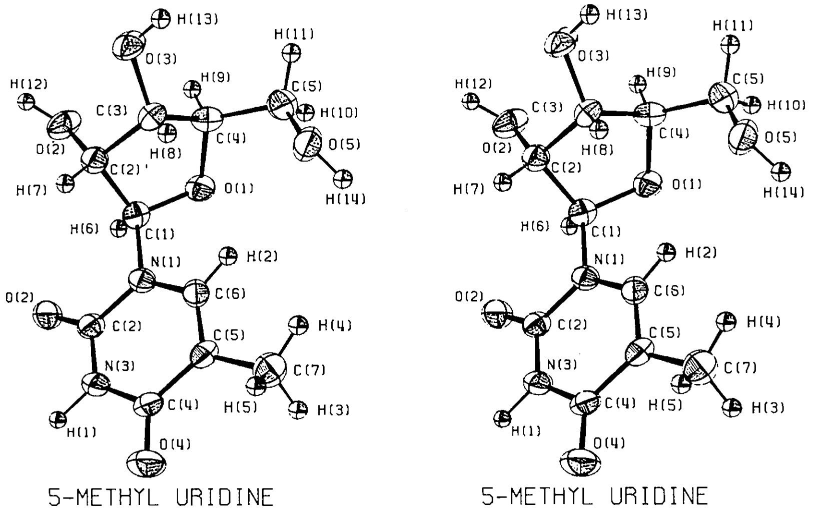

Fig. 2. A stereoscopic view of the molecule. The atoms are represented by ellipsoids (drawn at the 50% probability level) defined by the principal axes of thermal motion (Table 4).

factors calculated by Stewart, Davidson & Simpson (1965) were used and the remaining scattering-factor tables were taken directly from *International Tables for X-ray Crystallography* (1962). At the stage when parameter shifts were less than one fifth of their e.s.d.'s, hydrogen-atom positions alone were refined for four cycles. With the two largest amplitudes (121 and 350) excluded from the calculations and two final cycles of heavy-atom refinement, R converged to 0·075 with a 'goodness of fit' $[\Sigma\, w(|F_{\rm obs}|^2-|F_{\rm calc}|^2/k^2)^2|(m-s)]^{1/2}$, of 1·29, where s is the number of parameters refined and m the number of observations ($m=1294$; 'less thans' were only included if $|F_{\rm calc}|>|F_{\rm obs}|$). All parameter shifts were now less than one tenth of their e.s.d.'s.

5. Results and discussion

For the heavy atoms, the final atomic parameters and their e.s.d.'s are listed in Table 1. The corresponding values for the hydrogen atoms appear in Table 2. Table 3 lists the observed and calculated structure factors and a stereoscopic view of the molecule is shown in Fig. 2 together with the numbering scheme adopted.

Table 4 shows the magnitudes and direction cosines of the principal axes of the thermal ellipsoids which are also represented in Fig. 2. Atoms C(5), C(2′) C(3′) and C(5′) are not significantly anisotropic on the basis that none of their b_{ij} terms (Table 1) differ by more than three e.s.d.'s from the values they would have assuming isotropic motion with B equal to the mean value of the principal axis B_i's. Atoms O(2), O(4), O(2′), and O(3′) appear to be highly anisotropic. As expected, O(2) and O(4) have their maximum amplitudes in a direction approximately perpendicular to the plane of the pyrimidine ring.

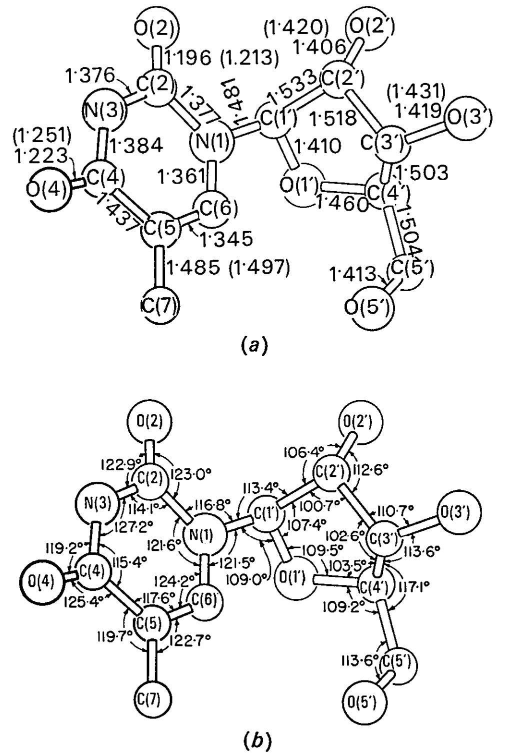

Fig 3. (*a*) Bond distances (in Å) for the non-hydrogen atoms. Values in parentheses include an approximate correction for the effects of thermal motion. (*b*) Bond angles in degrees for the non-hydrogen atoms.

Table 2. *Hydrogen-atom coordinates and their estimated standard deviations* (×10³)

	X	Y	Z
H(1)	061 (4)	089 (3)	370 (11)
H(2)	277 (5)	287 (3)	183 (11)
H(3)	400 (4)	146 (3)	497 (11)
H(4)	393 (5)	244 (3)	554 (11)
H(5)	354 (5)	188 (3)	726 (11)
H(6)	089 (5)	285 (3)	−208 (11)
H(7)	052 (4)	357 (3)	256 (12)
H(8)	190 (4)	419 (3)	259 (11)
H(9)	184 (5)	452 (3)	−279 (12)
H(10)	346 (5)	430 (3)	−301 (12)
H(11)	327 (4)	500 (4)	−122 (10)
H(12)	−038 (4)	435 (3)	−041 (11)
H(13)	170 (5)	537 (3)	198 (11)
H(14)	417 (4)	374 (3)	072 (11)
H(15) (water)	−052 (4)	032 (3)	660 (11)

Intramolecular bond distances and angles for the heavy atoms are shown respectively in Fig. 3(*a*) and (*b*). The values in parentheses in Fig. 3(*a*) include estimated corrections for thermal motion on the basis of a riding model (Busing & Levy, 1964). In the discussion that follows, bond lengths referred to are those uncorrected for thermal motion. The average C–H and O–H distances and the N–H distance are respectively 0·95, 0·94, and 1·10 Å. The average e.s.d.'s in bond lengths are:

$$
\begin{aligned}
\text{C–C} &= 0{\cdot}007\ \text{Å}\\
\text{C–N} &= 0{\cdot}006\\
\text{C–O} &= 0{\cdot}006\\
\text{(C,N,O)–H} &= 0{\cdot}06\,,
\end{aligned}
$$

with the corresponding average e.s.d.'s of bond angles not involving hydrogen atoms being of the order of 0·5°. Fig. 4 is a composite difference electron density map excluding the contributions of the hydrogen atoms to the calculated structure factors.

(*a*) *Packing and hydrogen bonding*

The view of the crystal structure projected down the c axis (Fig. 5) shows the packing arrangement, the hydrogen-bond scheme (broken lines) and the hydrogen bond distances. Some intermolecular contacts less than 3·5 Å are listed in Table 5, where proposed hydrogen bonds are identified by an asterisk. All potential hydrogen-atom donors seem to fulfill that role and O(3′), O(5′), and O(1) (water) also act as acceptors. Some angles associated with the hydrogen bonds are listed in Table 5.

Table 3. *Observed and calculated structure factors*

The four rows in each column are k, $10F_o$, $10F_c$ and the phase angle in degrees. Negative values for $10F_o$ indicate 'less than' reflections; asterisks indicate reflections omitted from the final least-squares cycle.

0 K 0			
0	---*	3650	0
2	182	172	180
4	378	385	180
6	221	213	180
8	261	247	180
10	354	321	0
12	415	424	0
14	96	85	0
16	338	374	0
18	306	298	180
20	87	79	0
22	31	38	0
1 K 0			
1	-11*	89	180
2	180	179	0
3	117	110	180
4	534	636	0
5	172	168	0
6	28	1	180
7	-16	13	180
8	18	1	0
9	76	72	180
10	326	313	180
11	66	63	180
12	156	144	180
13	65	60	180
14	122	113	0
15	63	57	0
16	88	88	180
17	-26	12	180
18	121	107	180
19	67	65	180
20	150	144	0
21	77	74	0
22	66	75	180
2 K 0			
0	552*	676	180
1	605*	865	180
2	153	140	0
3	9*	18	0
4	53	51	180
5	328	324	0
6	367	394	0
7	201	203	0
8	165	143	0
9	178	157	180
10	419	454	0
11	203	197	0
12	202	196	180
13	138	106	0
14	81	84	180
15	116	108	0
16	-43*	36	180
17	205	187	180
18	65	59	0
19	45	43	0
20	26	25	0
21	58	56	0
22	76*	92	0
3 K 0			
1	-18	18	0
2	117	113	0
3	113	104	180
4	297	308	0
5	968*	1230	0
6	133	134	180
7	162	153	0
8	356	364	180
9	187	178	180
10	27	35	0
11	287	286	0
12	63	60	0
13	67	65	180
14	58	50	180
15	141	148	0
16	30	26	180
17	85	78	0
18	39	30	0
19	37	37	0
20	70	61	180
21	32	31	180
4 K 0			
0	182	202	180
1	100	90	0
2	104	84	180
3	159	160	0
4	113	104	180
5	193	179	0
6	135	134	180
7	237	220	180
8	217	205	180
9	160	154	0
10	-15	16	0
11	382	378	180
12	117	110	0
13	85	86	0
14	57	59	180
15	134	149	0
16	110	117	180
17	22	22	0
18	-19	3	0
19	27	24	0
20	-16	8	180
21	-13	0	0
5 K 0			
1	93	94	0
2	167	171	180
3	296	306	0
4	190	182	180
5	499	484	0
6	304	293	180
7	109	105	0
8	219	214	0
9	213	192	0
10	291	317	0
11	29	21	180
12	31	32	180
13	92	96	180
14	71	68	0
15	166	184	0
16	-24	2	0
17	175	167	0
18	-25	3	180
19	-23	13	180
20	50	52	0
21	-14	11	0
6 K 0			
0	338	373	0
1	343	382	180
2	337	338	0
3	556	556	180
4	173	154	0
5	-30	6	180
6	189	168	180
7	130	126	0
8	145	144	180
9	175	178	0
10	67	79	0
11	121	108	0
12	143	144	0
13	190	183	180
14	-43	16	0
15	202	205	180
16	-42	20	180
17	-42	7	180
18	62	50	0
19	46	35	180
20	55	49	0
21	-19	30	0
7 K 0			
1	47	44	180
2	228	202	0
3	204	186	180
4	55	66	0
5	33	17	0
6	96	83	0
7	165	157	0
8	105	101	0
9	47	43	180
10	-40	14	180
11	72	66	180
12	84	75	180
13	43	32	0
14	-43	17	180
15	78	74	180
16	-42	31	0
17	-42	8	0
18	-40	24	0
8 K 0			
0	533	567	0
1	202	201	0
2	132	117	180
3	316	308	0
4	228	232	0
5	196	172	180
6	82	92	0
7	53	40	0
8	-39	19	0
9	-40	1	180
10	345	351	0
11	-42	26	180
12	185	197	0
13	52	57	180
14	51	49	180
15	81	74	0
16	80	69	0
17	-41	17	0
18	150	128	180
19	-31	9	0
20	18*	111	0
9 K 0			
1	-35	25	0
2	51	43	0
3	73	53	180
4	206	206	0
5	107	96	180
6	115	99	180
7	-40	29	0
8	190	181	180
9	-42	26	0
10	104	97	180
11	105	97	180
12	-43	27	0
13	-42	11	180
14	-42	23	0
15	126	104	0
16	87	74	0
17	-39	2	180
18	47	48	180
19	75	74	180
10 K 0			
0	250	225	0
1	54	54	180
2	126	112	0
3	78	64	180
4	280	263	180
5	-40	11	180
6	213	234	0
7	-42	14	180
8	42	26	180
9	-43	11	180
10	105	93	0
11	198	172	0
12	51	47	180
13	-42	18	0
14	126	108	0
15	44	29	180
16	68	48	180
17	41	21	0
18	26*	9	0
11 K 0			
1	131	125	0
2	82	81	180
3	110	103	180
4	94	94	180
5	408	430	0
6	106	117	0
7	-43	24	0
8	74	61	180
9	85	83	180
10	138	127	180
11	115	105	0
12	98	68	0
13	131	103	180
14	158	129	0
15	199	181	0
16	83	79	180
17	44	56	0
12 K 0			
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1	79	77	180
2	111	104	180
3	-43	26	180
4	142	141	0
5	196	197	0
6	85	85	180
7	111	114	180
8	-42	3	180
9	147	127	0
10	-42	16	180
11	188	167	180
12	64	52	180
13	-41	8	0
14	129	121	180
15	-34	25	180
16	98	92	0
13 K 0			
1	91	87	180
2	61	69	180
3	80	90	0
4	142	155	0
5	276	256	0
6	-42	8	180
7	-41	15	0
8	-42	3	180
9	-42	18	0
10	56	59	0
11	122	100	0
12	49	52	180
13	-37	17	0
14	-32	18	180
15	49	50	180
14 K 0			
0	289	251	0
1	96	86	180
2	-42	5	180
3	-41	23	180
4	87	70	180
5	184	176	180
6	-42	13	180
7	84	72	0
8	55	44	180
9	62	55	0
10	164	134	0
11	65	61	0
12	86	77	0
13	113	110	180
15 K 0			
1	-42	21	0
2	67	40	0
3	-42	22	0
4	100	93	0
5	64	47	180
6	159	143	180
7	59	52	180
8	-40	12	0
9	-37	8	180
10	67	57	0
11	-30	21	0
12	69	71	180
16 K 0			
0	84	78	0
1	107	89	180
2	42	42	0
3	58	41	180
4	-40	10	0
5	47	51	0
6	99	79	0
7	49	57	180
8	77	62	0
9	63	63	0
10	76*	91	0
17 K 0			
1	-34	14	0
2	34	31	0
3	-33	23	0
4	44	41	0
5	35	27	180
6	71	61	180
0 K 1			
0	706*	952	0
1	30	31	90
2	74	74	0
3	771*	862	270
4	53	47	0
5	188	159	90
6	432	419	180
7	91	79	270
8	242	228	180
9	315	280	270
10	137	134	0
11	142	123	270
12	216	214	0
13	103	98	270
14	130	134	180
15	57	62	270
16	43	41	0
17	-30	20	270
18	34	8	180
19	51	56	270
20	42	38	0
21	75	77	90
1 K 1			
0	287	326	90
1	395	470	199
2	745*	1176	351
3	547	568	122
4	228	236	69
5	367	355	346
6	279	282	87
7	503	506	129
8	200	192	143
9	278	275	223
10	257	233	203
11	93	94	307
12	117	125	244
13	129	140	179
14	121	125	163
15	178	183	108
16	226	236	58
17	112	106	258
18	77	67	341
19	91	90	96
20	68	69	173
21	44	44	356
2 K 1			
0	442	458	0
1	499	485	179
2	620	580	231
3	610	594	4
4	642	593	134
5	255	239	267
6	64	56	329
7	347	339	241
8	230	214	196
9	227	205	37
10	253	262	348
11	155	164	89
12	204	204	223
13	86	89	231
14	97	105	221
15	186	184	8
16	87	90	319
17	126	119	220
18	117	109	134
19	52	45	316
20	81	75	102
21	42	45	144
3 K 1			
0	308	311	270
1	265	275	267
2	283	277	280
3	629	676	246
4	360	370	272
5	281	278	60
6	299	295	158
7	106	100	353
8	306	282	259
9	205	183	148
10	146	137	18
11	146	137	115
12	22	11	265
13	64	68	103
14	40	38	220
15	179	173	328
16	116	135	254
17	-24	11	8
18	31	36	212
19	95	89	183
20	49	49	267
21	85	83	130
4 K 1			
0	311	314	180
1	389	394	102
2	313	292	196
3	321	283	168
4	386	378	221
5	137	124	353
6	210	184	287
7	99	88	286
8	236	211	125
9	74	76	111
10	138	120	124
11	251	265	120
12	111	113	197
13	135	147	49
14	100	104	110
15	66	67	311
16	120	125	208
17	18	13	205
18	29	24	277
19	53	48	120
20	45	42	111
21	49	50	1
5 K 1			
0	603	635	270
1	244	246	194
2	213	200	223
3	365	339	109
4	270	258	83
5	399	391	5
6	226	225	229
7	252	249	287
8	71	71	304
9	82	79	243
10	174	172	325
11	192	191	240
12	143	156	261
13	183	196	176
14	114	134	304
15	119	139	68
16	43	36	111
17	89	81	327
18	36	40	54
19	-22	11	9
20	28	23	22
21	38*	52	306
6 K 1			
0	369	353	0
1	60	48	353
2	184	168	122
3	198	187	157
4	76	78	70
5	191	173	293
6	221	232	171
7	48	42	5
8	235	240	205
9	32	34	56
10	69	73	113
11	41	41	233
12	115	132	41
13	89	93	157
14	89	93	237
15	130	124	211
16	70	75	88
17	46	42	359
18	76	77	140
19	59	50	99
20	77	78	315
7 K 1			
0	311	298	90
1	259	239	67
2	122	108	174
3	303	295	163
4	267	247	19
5	186	186	197
6	85	97	101
7	85	98	210
8	83	85	293
9	49	52	159
10	95	108	284
11	73	77	217
12	116	143	122
13	-30	12	217
14	124	122	79
15	35	34	268
16	-30	17	138
17	-30	18	129
18	-28	4	67
19	90	99	222
20	58	74	343
8 K 1			
0	101	156	0
1	150	133	224
2	92	89	95
3	212	218	274
4	105	110	240
5	138	147	241
6	101	106	179
7	98	103	315
8	124	144	106
9	41	40	166
10	122	139	343
11	90	109	315
12	109	139	311
13	70	84	274
14	36	38	204
15	-30	5	284
16	52	49	163
17	61	67	206
18	64	65	162
19	88	89	282
9 K 1			
0	280	277	270
1	197	203	214
2	74	71	272
3	104	120	193
4	118	138	116
5	124	142	331
6	225	235	35
7	119	144	135
8	214	225	123
9	75	82	232
10	120	157	186
11	148	195	87
12	118	165	314
13	128	135	197
14	-30	23	320
15	-30	10	304
16	49	50	64
17	26	23	73
18	55	53	172
10 K 1			
0	48	42	180
1	175	167	117
2	228	242	243
3	205	213	312
4	88	96	255
5	29	30	307
6	93	106	25
7	157	183	178
8	209	229	190
9	121	161	334
10	113	143	3
11	37	34	109
12	53	53	131
13	-30	17	224
14	91	92	210
15	62	72	20
16	56	50	224
17	40	39	271
18	22	22	283
11 K 1			
0	93	103	90
1	183	185	98
2	73	80	343
3	41	29	50
4	113	129	277
5	118	142	49
6	69	70	185
7	53	54	220
8	197	210	266
9	101	120	200
10	30	37	83
11	120	111	261
12	60	67	222
13	101	109	87
14	55	48	11
15	37	32	1
16	40	42	8
17	50	47	67
12 K 1			
0	106	118	0
1	107	122	29
2	99	105	212
3	62	67	99
4	94	105	168
5	43	42	94
6	81	89	148
7	80	94	267
8	-30	2	257
9	122	112	49
10	72	65	179
11	104	100	99
12	96	97	190
13	81	67	192
14	39	32	117
15	64	57	193
16	72	73	331
13 K 1			
0	30	35	90
1	108	115	254
2	207	188	224
3	171	161	156
4	110	108	26
5	187	163	9
6	76	71	248
7	80	77	283
8	81	66	323
9	-30	13	157
10	85	82	324
11	53	42	84
12	69	65	153
13	92	91	197
14	76	77	290
14 K 1			
0	105	93	0
1	163	154	196
2	73	67	142
3	88	78	206
4	94	89	175
5	48	42	114
6	55	48	195
7	46	41	358
8	54	50	49
9	42	35	296
10	-28	15	5
11	53	39	79
12	-22	12	342
15 K 1			
0	92	86	270
1	74	58	164
2	-30	19	55
3	73	65	77
4	105	95	12
5	43	42	218
6	106	91	120
7	109	94	217
8	38	30	184
9	52	48	171
10	60	48	95
11	45	44	279
16 K 1			
0	-28	13	0
1	69	56	203
2	68	58	230
3	-28	20	280
4	38	38	134
5	45	38	295
6	69	56	280
7	55	49	244
8	41	44	158
9	30	33	297
17 K 1			
0	39	32	90
1	-22	4	313
2	53	43	34
3	21	20	205
4	-20	8	267
5	32	34	164
6	43	46	189
0 K 2			
0	107	100	0
1	156	141	90
2	131	120	180
3	201	198	270
4	116	109	180
5	196	199	90
6	133	105	0
7	391	420	270
8	116	107	180
9	221	223	270
10	196	187	0
11	207	204	90
12	219	219	180
13	-36	6	270
14	36	38	180
15	63	68	270
16	145	141	0
17	-36	23	270
18	56	57	180
19	-33	15	270
20	40	40	180
1 K 2			
0	105	85	90
1	118	129	336
2	200	204	166
3	118	119	14
4	443	525	67
5	321	325	297
6	212	223	84
7	320	337	76
8	156	159	163
9	101	99	80
10	66	60	321
11	113	116	48
12	46	45	347
13	139	121	321
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15	33	29	90
16	195	186	95
17	30	25	204
18	65	51	63
19	53	51	42
20	25	24	166
2 K 2			
0	325	394	0
1	121	125	351
2	115	117	235
3	81	76	286
4	108	102	42
5	253	246	96
6	184	169	273
7	73	71	331
8	171	162	85
9	176	174	121
10	144	145	100
11	158	165	117
12	250	267	307
13	163	164	286
14	174	185	258
15	53	49	13
16	33	32	249
17	44	40	205
18	23	18	16
19	35	38	169
20	45	58	58
3 K 2			
0	125	121	270
1	253	248	260
2	368	396	315
3	82	82	171
4	294	267	241
5	207	200	11
6	378	383	105
7	193	180	194
8	129	121	326
9	156	158	84
10	135	128	86
11	132	130	3
12	123	122	276
13	107	104	191
14	82	77	185
15	93	92	62
16	65	58	312
17	-25	14	263
18	65	49	310
19	52	48	160
20	32	34	278
4 K 2			
0	36	35	180
1	595	671	98
2	251	239	37
3	186	181	98
4	68	57	15
5	392	424	275
6	291	296	328
7	81	80	137
8	-34*	27	277
9	141	130	66
10	149	153	7
11	144	152	72
12	76	75	198
13	246	283	135
14	125	132	57
15	27	26	116
16	75	62	31
17	87	77	305
18	49	40	13
19	-15	9	130
20	23	21	245
5 K 2			
0	160	155	90
1	305	283	39
2	100	96	208
3	345	347	176
4	65	59	76
5	214	208	56
6	65	68	220
7	203	204	314
8	139	126	254
9	157	155	241
10	96	92	323
11	142	145	259
12	79	74	256
13	112	118	124
14	-39*	38	104
15	73	69	25
16	103	87	115
17	-24	19	24
18	63	57	291
19	78	69	234
20	-20	24	98
6 K 2			
0	224	225	0
1	169	161	72
2	227	215	234
3	119	115	261
4	165	153	15
5	78	74	45
6	52	41	74
7	85	76	56
8	-34	17	63
9	118	116	109
10	128	126	85
11	111	106	185
12	126	133	57
13	146	129	328
14	46	33	197
15	53	41	2
16	108	97	311
17	63	51	151
18	48	52	172
19	-26	17	155
7 K 2			
0	91	85	270
1	101	101	311
2	171	154	163
3	157	162	40
4	274	287	81
5	108	96	47
6	124	127	72
7	71	70	216
8	140	142	95
9	131	136	327
10	174	190	262
11	102	103	354
12	115	116	88
13	-40	39	149
14	78	76	85
15	42	28	352
16	39	33	101
17	111	104	293
18	83	81	116
19	-21	16	197
8 K 2			
0	109	106	0
1	190	181	217
2	168	156	116
3	148	140	306
4	-34	26	81
5	37	32	97
6	215	226	60
7	83	77	215
8	249	263	195
9	37	47	58
10	218	250	300
11	93	84	23
12	-39	43	330
13	142	128	233
14	43	44	220
15	-40	21	286
16	90	75	116
17	-32	16	161
18	50	39	120
9 K 2			
0	217	219	270
1	85	85	345
2	-35	10	213
3	35	26	313
4	179	185	81
5	119	118	294
6	196	211	109
7	116	112	42
8	58	63	351
9	-38	27	270
10	173	155	71
11	163	140	78
12	121	106	197
13	48	53	349
14	71	60	346
15	-37	25	335
16	71	15	93
17	-27	50	67
10 K 2			
0	-36	19	0
1	214	220	69
2	220	256	270
3	74	61	353
4	173	180	287
5	46	43	272
6	73	82	261
7	156	178	235
8	161	143	44
9	67	60	321
10	138	132	90
11	158	166	103
12	48	47	231
13	33	40	73
14	45	44	275
15	-33	12	70
16	81	74	316
11 K 2			
0	129	141	90
1	153	175	85
2	111	116	192
3	55	75	140
4	66	69	313
5	122	104	354
6	57	56	43
7	81	65	237
8	162	146	255
9	79	67	89
10	44	42	121
11	51	47	297
12	48	46	309
13	64	58	177
14	-33	8	30
15	41	27	341
16	39	41	63
12 K 2			
0	67	64	0
1	84	100	144
2	87	91	28
3	67	73	41
4	-39	28	149
5	46	49	85
6	54	60	7
7	79	71	123
8	-41	32	36
9	116	114	72
10	-40	21	13
11	31	37	100
12	64	56	318
13	-31	20	214
14	48	34	0
13 K 2			
0	190	183	90
1	-40	35	198
2	136	121	277
3	111	103	65
4	36	32	188
5	64	57	59
6	35	20	145
7	103	94	253
8	59	50	351
9	38	36	229
10	-36	25	327
11	-33	23	326
12	58	56	199
13	-24	12	356
14 K 2			
0	98	77	0
1	62	60	61
2	-41	24	224
3	87	71	254
4	101	82	77
5	40	47	71
6	-39	16	207
7	-37	24	212
8	62	53	12
9	-33	19	334
10	-30	23	331
11	67	60	77
15 K 2			
0	105	96	270
1	112	106	289
2	55	58	124
3	-37	27	118
4	60	53	39
5	46	37	335
6	82	76	111
7	65	57	326
8	102	93	96
9	30	22	290
16 K 2			
0	58	49	0
1	54	49	207
2	76	68	265
3	-30	13	354
4	81	64	229
5	39	28	85
6	35	30	354
7	42	43	321
0 K 3			
0	492	502	180
1	71	61	270
2	67	70	180
3	146	121	270
4	107	103	180
5	150	144	90
6	30	27	180
7	120	119	270
8	-25	5	0
9	-26	23	270
10	46	44	180
11	62	63	90
12	169	181	180
13	136	150	270
14	-35*	21	180
15	-36*	39	90
16	51	54	180
17	-29	21	270
18	-25	7	180
19	40	33	270
1 K 3			
0	72	68	90
1	198	203	325
2	263	282	214
3	81	81	263
4	195	191	175
5	143	138	96
6	59	44	84
7	64	59	68
8	69	68	0
9	41	27	268
10	212	231	328
11	64	66	11
12	164	153	322
13	91	81	351
14	108	97	188
15	39	26	300
16	92	82	113
17	39	32	141
18	28	35	339
19	-19	13	107
2 K 3			
0	179	187	0
1	24	24	22
2	123	124	217
3	151	159	223
4	121	117	118
5	49	44	298
6	104	103	336
7	58	60	280
8	98	101	92
9	79	80	241
10	130	143	172
11	92	88	107
12	131	133	264
13	54	58	75
14	47	38	285
15	81	76	241
16	75	77	317
17	38	33	269
18	-18	10	65
19	-18*	21	128
3 K 3			
0	159	157	270
1	111	111	202
2	125	124	322
3	149	152	240
4	72	68	255
5	170	172	245
6	145	143	14
7	250	251	108
8	115	122	266
9	87	87	295
10	164	182	128
11	107	107	119
12	77	73	309
13	133	117	176
14	57	49	337
15	63	53	176
16	25	15	346
17	36	33	259
18	60	53	302
19	-14	13	236
4 K 3			
0	71	70	0
1	163	160	126
2	62	62	297
3	112	105	268
4	202	196	2
5	185	180	315
6	116	115	286
7	177	187	286
8	117	110	228
9	124	134	173
10	142	153	40
11	126	136	63
12	78	77	50
13	82	73	291
14	31	27	183
15	28	20	129
16	75	66	359
17	71	68	250
18	28	28	65
5 K 3			
0	-20	1	270
1	63	58	5
2	87	79	261
3	154	149	165
4	37	27	311
5	227	231	110
6	89	89	118
7	299	330	226
8	108	113	276
9	137	138	142
10	60	54	290
11	223	273	312
12	30	27	183
13	91	83	80
14	87	83	198
15	107	97	152
16	22	21	234
17	65	54	171
18	-19*	26	95
6 K 3			
0	108	111	180
1	132	137	6
2	226	229	124
3	125	113	331
4	128	124	296
5	166	155	210
6	131	135	35
7	-28	13	259
8	97	97	171
9	94	92	30
10	66	55	315
11	70	69	279
12	56	52	99
13	39	33	286
14	58	44	200
15	100	87	321
16	-27	16	83
17	37	44	46
18	-15	19	173
7 K 3			
0	-26	7	270
1	82	76	297
2	134	132	256
3	120	116	109
4	171	176	92
5	106	112	328
6	148	149	207
7	51	60	27
8	110	115	282
9	76	79	322
10	125	124	325
11	65	53	98
12	67	67	169
13	137	131	207
14	-31	8	266
15	99	95	18
16	92	91	84
17	60	58	317
8 K 3			
0	38	34	180
1	40	45	182
2	118	124	211
3	173	196	288
4	166	169	125
5	109	111	33
6	51	50	349
7	106	119	170
8	55	57	173
9	40	36	309
10	124	115	187
11	47	35	117
12	137	124	209
13	84	91	264
14	-29	12	333
15	39	36	62
16	31	33	43
9 K 3			
0	200	227	270
1	140	154	23
2	91	98	214
3	80	85	223
4	59	60	297
5	93	102	255
6	140	162	70
7	29	27	55
8	64	59	26
9	71	64	93
10	34	32	238
11	71	65	57
12	88	87	243
13	56	59	354
14	66	60	5
15	38	25	285
16	70	83	260
10 K 3			
0	-32	12	180
1	75	82	125
2	124	141	261
3	57	59	319
4	74	71	306
5	66	78	291
6	65	59	189
7	116	115	245
8	49	34	102
9	34	31	35
10	-32	23	116
11	82	76	155
12	36	33	302
13	32	21	258
14	37	36	104
11 K 3			
0	135	129	90
1	122	103	167
2	127	122	205
3	-35	30	89
4	35	26	300
5	83	82	136
6	35	30	298
7	-34	21	251
8	102	98	285
9	52	44	298
10	57	56	16
11	41	35	160
12	-25	22	245
13	-21	15	196
14	26	37	205
12 K 3			
0	36	16	0
1	47	33	169
2	39	43	299
3	39	36	306
4	42	35	117
5	42	35	188
6	79	66	6
7	60	50	237
8	53	46	11
9	42	41	238
10	-26	7	346
11	68	70	30
12	-19	20	333
13 K 3			
0	-32	10	90
1	-32	14	312
2	43	33	290
3	47	43	88
4	-31	4	277
5	78	67	163
6	-29	21	262
7	109	109	231
8	-26	23	233
9	74	80	123
10	47	49	189
11	39	45	271
14 K 3			
0	78	76	180
1	81	75	313
2	80	75	72
3	45	31	8
4	36	27	15
5	60	66	359
6	54	46	129
7	39	41	253
8	30	34	251
9	43	45	165
15 K 3			
0	-23	4	270
1	71	72	261
2	85	86	249
3	35	37	78
4	30	38	173
5	58	60	341
6	47	53	102
0 K 4			
0	321	338	180
1	33	31	270
2	140	153	180
3	129	124	90
4	33	21	0
5	177	195	90
6	188	203	0
7	40	33	90
8	135	148	0
9	69	71	90
10	81	88	180
11	42	19	90
12	95	91	180
13	30	30	270
14	61	66	180
15	58	59	90
16	47	43	180
17	28	33	90
1 K 4			
0	24	29	90
1	34	43	299
2	84	86	181
3	69	76	303
4	138	146	200
5	45	37	110
6	-25	14	213
7	34	27	125
8	98	103	45
9	81	80	284
10	95	88	314
11	94	87	54
12	55	49	332
13	-26	11	338
14	31	23	22
15	54	53	274
16	-19	5	62
17	29	32	84
2 K 4			
0	-22	15	0
1	129	134	52
2	97	101	40
3	97	98	234
4	40	39	351
5	70	73	67
6	49	49	178
7	-23	13	187
8	150	144	173
9	60	58	229
10	-23	15	4
11	111	109	129
12	25	21	20
13	49	40	110
14	58	52	4
15	43	40	166
16	35	36	351
17	12*	80	348
3 K 4			
0	98	110	90
1	74	72	308
2	38	32	82
3	66	63	229
4	61	53	92
5	57	52	204
6	72	57	105
7	104	104	102
8	45	34	319
9	36	35	262
10	176	172	66
11	73	63	282
12	41	34	182
13	-27*	18	74
14	23	16	129
15	80	79	164
16	59	55	71
4 K 4			
0	171	168	0
1	76	75	297
2	87	76	207
3	83	77	84
4	64	64	352
5	122	129	214
6	-19*	11	337
7	42	42	338
8	100	97	335
9	69	65	22
10	79	88	148
11	73	71	329
12	61	53	57
13	44	39	181
14	67	64	250
15	40	41	238
16	67	71	355
5 K 4			
0	165	179	90
1	89	88	30
2	89	92	323
3	117	122	213
4	98	96	65
5	90	96	152
6	155	158	75
7	-24	31	173
8	139	130	257
9	67	57	338
10	131	122	138
11	45	36	359
12	91	79	83
13	37	35	112
14	55	56	185
15	31	33	335
6 K 4			
0	106	103	180
1	145	136	136
2	138	132	99
3	139	144	320
4	77	71	164
5	159	137	45
6	211	182	335
7	45	49	189
8	24	32	182
9	117	106	171
10	81	71	334
11	58	60	80
12	88	82	204
13	84	93	308
14	51	50	57
15	92	102	26
7 K 4			
0	84	80	270
1	77	77	300
2	82	85	167
3	72	68	78
4	72	76	119
5	45	39	357
6	65	67	256
7	69	56	254
8	45	35	132
9	86	75	122
10	-28	28	270
11	49	47	358
12	-24	10	17
13	46	41	267
14	63	63	352
8 K 4			
0	109	92	180
1	148	124	72
2	76	61	248
3	106	96	229
4	115	100	11
5	95	96	73
6	49	50	127
7	75	70	131
8	53	45	198
9	91	84	32
10	73	68	170
11	68	58	130
12	-22	9	139
13	32	34	313
14	36	40	239
9 K 4			
0	24	14	90
1	-30	21	335
2	74	62	204
3	-29	22	328
4	-29	34	317
5	54	49	293
6	88	81	110
7	-28	25	165
8	72	65	320
9	64	57	60
10	86	80	359
11	40	30	340
12	34	37	187
10 K 4			
0	23	25	0
1	-29	12	172
2	60	56	255
3	84	74	27
4	57	58	91
5	-28	15	121
6	-27	32	307
7	-26	5	43
8	-25	21	36
9	42	40	127
10	26	29	198
11	25	27	149
11 K 4			
0	53	53	90
1	-27	21	234
2	-27	19	113
3	-26	1	210
4	40	39	58
5	80	83	167
6	24	23	74
7	46	52	291
8	50	51	32
9	44	38	18
10	40	41	85
12 K 4			
0	-24	13	180
1	-24	19	158
2	-24	30	309
3	33	33	132
4	23	23	29
5	60	58	236
6	20	18	144
7	18	20	198
8	22	23	323
9	7*	26	281
13 K 4			
0	90	87	90
1	71	55	22
2	-19	16	32
3	-19	7	188
4	32	35	145
5	31	39	198
6	23	27	131
0 K 5			
0	117	108	180
1	51	35	270
2	60	50	180
3	-36	28	90
4	58	46	0
5	70	61	90
6	167	166	0
7	-35	3	270
8	-35	2	0
9	-34	8	270
10	-32	6	0
12	30*	9	0
1 K 5			
0	-26	14	270
1	25	16	295
2	44	46	209
3	60	57	252
4	88	90	223
5	72	65	268
6	112	107	211
7	53	46	109
8	75	72	352
9	-25	13	194
10	73	68	12
11	37	38	62
12	37	32	206
2 K 5			
0	22	19	180
1	64	62	322
2	60	60	20
3	53	47	148
4	-23	20	27
5	-23	28	10
6	82	78	228
7	42	45	230
8	-22	15	39
9	45	45	276
10	60	61	100
11	-18	4	0
12	21	18	121
3 K 5			
0	61	58	90
1	146	130	63
2	81	71	185
3	48	37	208
4	38	29	355
5	45	36	110
6	51	43	22
7	55	54	25
8	23	23	210
9	81	80	293
10	-20	12	296
11	44	40	269
12	12*	9	288
4 K 5			
0	130	126	0
1	51	48	231
2	108	95	126
3	120	117	276
4	-19	11	33
5	58	55	135
6	27	26	221
7	37	32	0
8	51	48	173
9	37	33	330
10	42	33	157
11	57	51	259
12	32*	37	345
5 K 5			
0	103	92	90
1	134	124	264
2	53	48	333
3	113	103	351
4	60	50	336
5	58	54	194
6	33	26	314
7	65	56	144
8	36	37	255
9	59	59	41
10	60	59	139
11	44	43	39
0 K 6			
0	45	45	180
1	-25	5	90
2	67	65	0
3	34	35	270
4	45	40	180
5	40	48	90
1 K 6			
0	-19	1	90
1	29	26	71
2	32	35	144
3	64	67	187
4	28	35	248
5	25	22	287
2 K 6			
0	-16	5	0
1	-15	10	193
2	45	48	180
3	39	40	133
4	44	46	345
5	16	18	26
3 K 6			
0	68	101	90
1	-15	19	230
2	60	68	184
3	40	45	355
4	35	44	13
4 K 6			
0	-11	11	0
1	32*	44	281
2	30	28	88
3	29	35	2

Each water molecule is surrounded by two diad-related oxygen atoms [O(5′)] and two diad-related nitrogen atoms [N(3)], all presumably participating in hydrogen bonding with the water molecule. The distance N(3)–O(1) of 3·019 Å is, however, rather long compared with similar nitrogen to water distances of 2·78 Å in calcium thymidylate (Trueblood *et al.*, 1961) and 2·91 Å in barium uridine-5′-phosphate (Shefter & True blood, 1965). Except for those of H(1) and H(13) it appears that none of the positions listed for the hydrogen atoms are significantly displaced from the donor-acceptor line. It is estimated that the standard devia-

Table 4. *The magnitudes and direction cosines (with respect to the crystallographic axes) of the principal axes of the thermal ellipsoids*

	Axis (i)	B_i	q_i^a	q_i^b	q_i^c
C(5)	1	3·10 Å^2	−0·432	−0·532	0·728
	2	2·35	0·663	0·360	0·656
	3	1·77	−0·611	0·766	0·197
C(6)	1	3·58	0·256	−0·377	0·890
	2	2·06	−0·943	0·104	0·315
	3	1·80	0·212	0·920	0·329
N(1)	1	2·83	−0·181	−0·223	0·958
	2	2·41	0·966	−0·223	0·131
	3	1·67	0·184	0·949	0·255
C(2)	1	3·54	−0·320	−0·167	0·933
	2	2·06	0·846	−0·493	0·203
	3	1·77	0·426	0·854	0·299
N(3)	1	4·22	−0·130	0·188	0·973
	2	2·46	0·960	−0·224	0·171
	3	1·84	−0·250	−0·956	0·152
C(4)	1	3·74	−0·198	0·179	0·964
	2	2·74	0·836	0·545	0·071
	3	1·67	0·512	−0·819	0·258
C(7)	1	4·52	−0·284	0·724	0·628
	2	3·43	−0·633	−0·634	0·445
	3	2·38	0·720	−0·271	0·638
O(1)	1	3·79	−0·011	−0·075	0·997
	2	3·70	−0·106	−0·994	0·000
	3	2·13	−0·994	0·106	0·000
O(2)	1	5·90	−0·303	0·268	0·914
	2	2·81	0·155	−0·933	0·325
	3	2·01	0·940	0·241	0·241
O(4)	1	7·14	−0·297	0·454	0·840
	2	3·90	0·936	0·315	0·160
	3	2·02	0·192	−0·834	0·518
C(1′)	1	3·01 Å^2	−0·635	−0·315	0·705
	2	2·33	−0·398	0·916	0·051
	3	1·60	0·662	0·248	0·707
C(2′)	1	2·97	−0·070	−0·318	0·946
	2	2·30	−0·935	0·351	0·049
	3	2·19	0·347	0·881	0·322
O(1′)	1	3·75	0·878	−0·054	0·475
	2	2·49	−0·342	−0·765	0·546
	3	1·57	−0·334	0·642	0·690
C(3′)	1	2·79	−0·733	−0·647	0·212
	2	2·02	0·680	−0·707	0·193
	3	1·89	0·025	0·286	0·958
C(4′)	1	3·32	−0·959	−0·060	0·278
	2	1·92	−0·019	0·989	0·145
	3	1·75	0·283	−0·133	0·950
O(2′)	1	5·89	−0·521	−0·259	0·813
	2	2·78	0·169	0·903	0·396
	3	1·73	0·837	−0·344	0·426
C(5′)	1	4·38	0·608	0·231	0·759
	2	3·12	−0·473	0·874	0·113
	3	2·32	−0·638	−0·427	0·641
O(5′)	1	4·17	−0·100	−0·526	0·845
	2	3·20	0·392	0·760	0·519
	3	2·05	−0·915	0·383	0·130
O(3′)	1	4·87	−0·153	−0·446	0·882
	2	2·75	0·984	0·016	0·179
	3	1·56	−0·093	0·895	0·436

Table 5. *Some close contacts less than* 3·5 Å *and hydrogen atom-donor-acceptor angles* (θ) *where appropriate*

Proposed hydrogen bonds are denoted by an asterisk. Other angles relevant to the hydrogen bonds are also shown.

Atom 1	Atom 2	Distance (Å)	θ°
O(1) (water)	N(3)	3·019*	17
O(2′) $(-x, -y+1, z)$	O(2′)	3·232	
O(2′) $(-x, -y+1, z)$	O(3′)	2·800*	4
C(5′) $(-x+\frac{1}{2}, y-\frac{1}{2}, -z+1)$	O(4)	3·305	
O(5′) $(-x+\frac{1}{2}, y-\frac{1}{2}, -z+1)$	O(1)	2·906*	7
O(5′) $(-x+\frac{1}{2}, y-\frac{1}{2}, -z+1)$	O(4)	3·354	
O(3′) $(-x+\frac{1}{2}, y-\frac{1}{2}, -z+1)$	C(7)	3·486	
O(3′) $(-x+\frac{1}{2}, y-\frac{1}{2}, -z+1)$	O(4)	2·735*	13
O(1′) $(x, y, z+1)$	C(5)	3·252	
O(1′) $(x, y, z+1)$	C(6)	3·195	
O(2′) $(x+\frac{1}{2}, -y+\frac{1}{2}, -z)$	C(7)	3·404	
O(2) $(x+\frac{1}{2}, -y+\frac{1}{2}, -z)$	O(5′)	2·742*	6
O(2) $(x+\frac{1}{2}, -y+\frac{1}{2}, -z+1)$	C(7)	3·476	

Other hydrogen-bond angles

O(5′) $(-x+\frac{1}{2}, y-\frac{1}{2}, -z+1)$–O(1)–O(5′) $(x-\frac{1}{2}, -y+\frac{1}{2}, -z+1)$	112·4°
C(5′)–O(5′)–O(2) $(x+\frac{1}{2}, -y+\frac{1}{2}, -z)$	112·5
C(2)–N(3)–O(1)	127·9
C(4)–N(3)–O(1)	104·9
C(3′)–O(3′)–O(4) $(-x+\frac{1}{2}, y+\frac{1}{2}, -z+1)$	102·2
C(2′)–O(2′)–O(3′) $(-x, -y+1, z)$	107·6

tions for the 'hydrogen-donor-acceptor' angles are of the order of 3·5°. The hydrogen atoms of the bonds N(3)–H(1)...O(1) and O(3')–H(13)...O(4) ($\frac{1}{2}-x, \frac{1}{2}+y, 1-z$) lie respectively 17° and 13° off the donor-acceptor line.

Because of the tilt (42·5°) of the base plane with respect to (001) there is little overlap between bases and stacking interactions will be small. The closest contact between parallel bases is 3·598 Å for C(2)–O(4) ($x, y, z-1$).

(*b*) *The pyrimidine ring*

Within the accuracy of this determination, the pyrimidine ring is planar but with C(1') significantly displaced (0·083 Å) to the same side of the plane as C(2'). The data for the least-squares plane are given in Table 6 and show that the other five exocyclic atoms are not significantly displaced from the plane. Fig. 6 provides a view of the C(7) hydrogen atoms looking down the bond C(7)–C(5). As might be expected, the conformation adopted is staggered with respect to O(4).

Table 6. *Deviations from the least-squares plane of the pyrimidine ring*

The equation of the least-squares plane is

$$-0{\cdot}3730X+0{\cdot}5629Y+0{\cdot}7375Z=1{\cdot}9841$$

where the coefficients are the direction cosines of the plane normal with respect to the crystallographic axes.

	Deviation (Å)		Deviation (Å)
N(1)	0·000	O(2)*	−0·001
C(2)	−0·008	O(4)*	0·012

Table 6 (*cont.*)

	Deviation (Å)		Deviation (Å)
N(3)	0·009	C(7)*	−0·013
C(4)	−0·001	C(1')*	0·083
C(5)	−0·006	H(1)*	−0·11
C(6)	0·007	H(2)*	0·02

* Atoms not included in the calculation of the plane.

The bond lengths in the pyrimidine base agree quite well with those of previous determinations of comparable accuracy (Harris & MacIntyre, 1964; Camerman

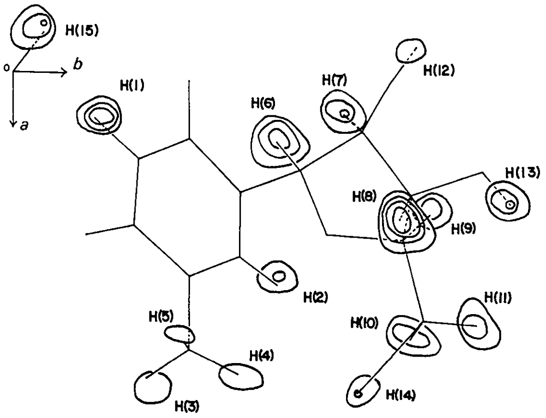

Fig. 4. The composite difference electron density map, for which the contributions of the hydrogen atoms were excluded from the calculated structure factors. Contours are drawn at intervals of 0·1 e.Å^{-3} starting at 0·3 e.Å^{-3}.

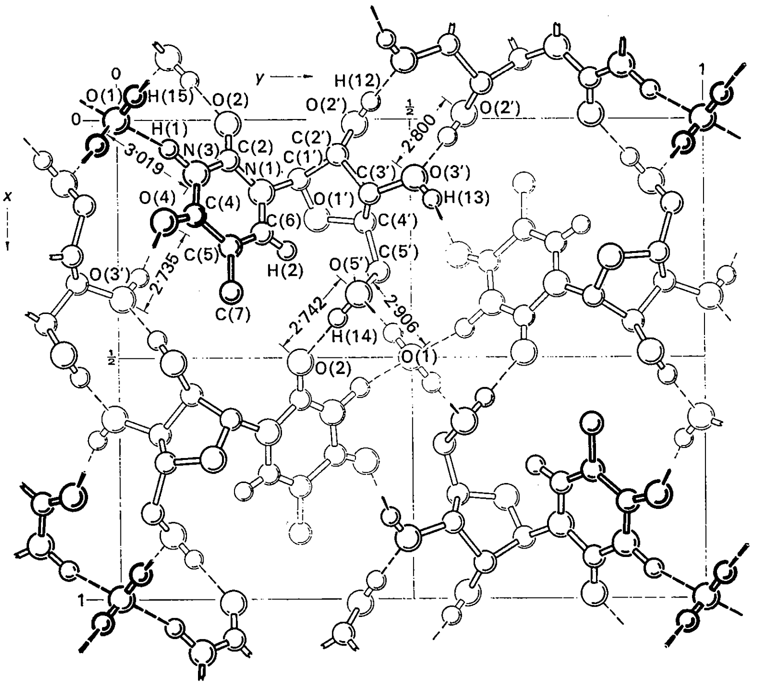

Fig. 5. The crystal structure viewed down the *c* axis. Not all the hydrogen atoms are shown.

& Trotter, 1965; Trueblood *et al.*, 1961; Shefter & Trueblood, 1965; Haschemeyer & Sobell, 1965; Iball *et al.*, 1966, 1968; Parry, 1954; Gerdil, 1961; Reeke & Marsh, 1966). The e.s.d.'s in bond lengths for 5-fluoro-2′-deoxyuridine (FUDR)* (Harris & MacIntyre, 1964) and thymine monohydrate (Gerdil, 1961) were quoted† as 0·008–0·011 and 0·006–0·009 Å respectively. There are no significant differences between the lengths of the bonds common to the pyrimidine bases of 5-methyluridine and FUDR. The agreement of thymine monohydrate with 5-methyluridine is similar except for the bond C(2)–O(2), which is 0·038 Å longer in the free base than in the nucleoside. The significance of this difference would probably be reduced if the effects of thermal motion were taken into account. For the bond angles, the agreement is nearly as good except around C(4), C(5) and C(6). The respective values of the internal angles at these atoms for 5-methyluridine and FUDR are 115·4, 117·6, 124·2, and 112·6(0·7), 122·7 (0·7), 120·3(0·7)°; the differences are not surprising in view of the different substituent at C(5) in the two compounds. The values for 5-methyluridine agree much better with those of thymine monohydrate (115·6, 118·2, 121·8°) where the calculated standard deviations of the bond angles vary from 0·3 to 0·5°.

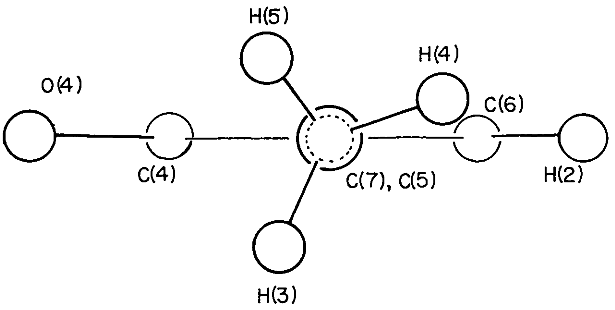

Fig. 6. The view of the C(7) hydrogen atoms looking down the bond C(7)–C(5).

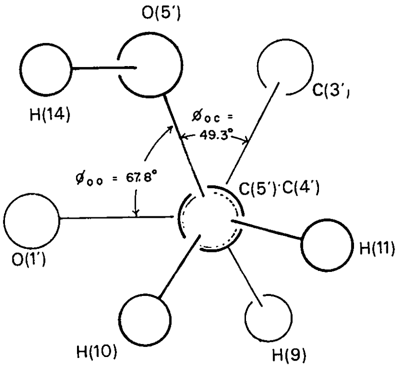

Fig. 7. The conformation of the bond C(5′)–C(4′).

(*c*) *The sugar residue*

The conformation adopted by the sugar ring can be described with respect to the plane through the atoms C(1′), C(2′), C(4′), and O(1′) (Table 7). C(3′) is displaced 0·605 Å out of this plane on the same side as C(5′), a conformation commonly observed amongst pyrididine nucleosides and referred to as C(3′) *endo*.

Table 7. *Deviations from a least-squares plane of the sugar residue*

The equation of the least-squares plane is

$$0.5055X - 0.2103Y + 0.8367Z = -0.5713.$$

	Deviation (Å)
C(1′)	0·012
C(2′)	−0·007
C(3′)	0·605*
C(4′)	0·008
C(5′)	0·741*
O(1′)	−0·013
O(2′)	−1·360*
O(3′)	0·283*
O(5′)	2·123*

* Atoms not included in the calculation of the plane.

The torsion angle (Sundaralingam & Jensen, 1965*a*) describing the relative orientation of the sugar and base residues with respect to the glycosidic bond [N(1)–C(1′)] is −29·4°, the *anti* conformation; it lies within the range of values previously observed for pyrimidine nucleosides (Haschemeyer & Rich, 1967). The dihedral angle between the least-squares plane through the six ring atoms of the base and that through the ring atoms of the sugar residue, excluding C(3′), is 71·9°.

For comparison purposes, the structure of the ribose in cytidine (Furberg, Peterson & Rømming, 1965) seems appropriate, and, in fact, the similarities in the geometries of the two residues are striking. Cytidine is also in the *anti* conformation with a torsion angle of −18·3° (our calculation); and C(3′) is displaced *endo* by 0·58 Å. The e.s.d.'s in bond lengths and angles are quoted as about 0·006 Å and 0·5° respectively. Only one of the ribose bond lengths in 5-methyluridine is significantly different from that in cytidine. The bond C(3′)–C(4′) is 0·030 Å shorter in 5-methyluridine (1·503 Å) than in cytidine (1·533 Å). The next largest difference is 0·019 Å for C(2′)–O(2′) which is 1·406 Å in 5-methyluridine and 1·425 Å in cytidine. This difference is probably not significant.

The internal ring angles of the ribose in 5-methyl uridine do not differ significantly from those determined for cytidine, but four of the external bond angles in the two nucleosides differ by approximately three e.s.d.'s. These angles are, for 5-methyluridine and cytidine in each case, N(1)–C(1′)–C(2′) (113·4, 111·4°), C(1′)–C(2′)–O(2′) (106·4, 108·9°), C(2′)–C(3′)–O(3′) (110·7,

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* The abbreviation of the original authors.

† For thymine monohydrate, the figures given here are confidence limits estimated by Gerdil and equal to twice the e.s.d.'s obtained from the least-squares method.

112·7°) and C(3′)–C(4′)–C(5′) (117·1, 114·8°). Presumably, these quite small differences arise from the different hydrogen-bonding and packing schemes in the crystals. That the modes of puckering of the five-membered rings of the two nucleosides are similar can be seen from a comparison of the torsion angles – the dihedral angle between the planes defined by a bond and its two adjacent ring bonds (Brown & Levy, 1963). These angles are given in Table 8.

Table 8. *Torsion angles in the ribose rings of* 5-*methyluridine* (MUR) *and cytidine*

Furberg *et al.* (1965) did not list signs for the torsion angles; we have allocated signs according to the definition of Brown & Levy (1963).

Bond	MUR	Cytidine
C(1′)→O(1′)	+2·5	+6·1
C(1′)→C(2′)	−25·5	−28·1
C(2′)→C(3′)	+38·5	+37·6
C(3′)→C(4′)	−38·2	−35·1
C(4′)→O(1′)	+22·5	+18·3

For 5-methyluridine the conformation about the bond C(4′)–C(5′) can be seen in Fig. 7, which also shows the two projected valency angles φ_{OO} and φ_{OC} (Shefter & Trueblood, 1965). For 5-methyluridine $\varphi_{OO}=67{\cdot}8°$ and $\varphi_{OC}=49{\cdot}3°$, the conformation is *gauche-gauche*, with C(5′)–O(5′) on the same side of the bonds O(1′)–C(4′) and C(3′)–C(4′). Sundaralingam & Jensen (1965*b*) and Shefter & Trueblood (1965) have noted that the *gauche-gauche* conformation occurs most frequently in nucleosides and nucleotides. For cytidine (Furberg *et al.* 1965) we have calculated $\varphi_{OO}=70{\cdot}4°$ and $\varphi_{OC}=47{\cdot}1°$ with the C(5′)–C(4′) bond also in the *gauche-gauche* conformation.

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