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A Refinement of the Crystal Structure of Cytidine

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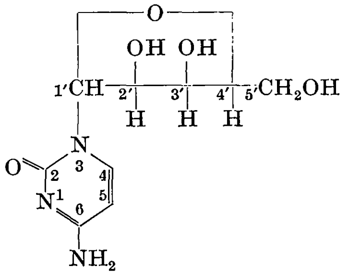
The crystal structure of cytidine has been refined by least-squares calculations to a final R index of 5·6%. Three-dimensional photographic data were used. The essential features of the original structure are confirmed, but the standard deviations in the bond lengths are reduced to about 0·006 Å and in the bond angles to less than 0·5°. The distances and angles in the cytosine part of the molecule are closely similar to those found in cytosine monohydrate. The two angles at each external bond to pyrimidine appear to differ significantly. The N-glycosidic linkage is found to be 1·497 Å. The two bonds to the furanose ring oxygen atom differ in length by 0·03 Å. The existence of an intramolecular C–H · · · O interaction at 3·23 Å is confirmed.

The crystal structure of cytidine, $C_9H_{13}O_5N_3$, was determined by one of us fifteen years ago from three electron density projections refined to $R = 0{\cdot}17 - 0{\cdot}19$ (Furberg, 1950). Pauling & Corey (1956), not considering this structure analysis reasonably reliable, deduced bond lengths and angles in the cytosine part of cytidine by considering the crystal structures of a number of other pyrimidines, mainly heavy atom derivatives. This work was later extended by Spencer (1959). Recently, an accurate crystal structure analysis of cytosine hydrate has been published (Jeffrey & Kinoshita, 1963), and precise information on the ribose residue has been obtained through three-dimensional analysis of calcium thymidylate (Trueblood, Horn & Luzzati, 1961) and adenosine 5-phosphate (Kraut & Jensen, 1963). However, cytidine is still the only unsubstituted nucleoside the structure of which has been solved. It contains no heavy atoms and its aromatic ring is not protonated. We have therefore refined the structure in order to obtain more exact information on this part of the nucleic acids.



Cytidine

Experimental

The unit-cell dimensions were redetermined by least-squares calculations based on measurements of 28 lines registered on a Guinier camera calibrated against KCl. The following values were found:

$$a = 13{\cdot}991 \pm 0{\cdot}002,\ b = 14{\cdot}786 \pm 0{\cdot}002\ ,$$
$$c = 5{\cdot}116 \pm 0{\cdot}001\ \text{Å}\ .$$

The uncertainties indicated are estimated standard deviations. The space group is $P2_12_12_1$ and there are four molecules in the unit cell.

Integrated equi-inclination Weissenberg photographs $hk0$–$hk2$, $h0l$–$h8l$ and $0kl$ were taken. Of the total of about 1450 unique reflexions within the Cu $K\alpha$ limit, 1195 were recorded. Of these 165 were too weak to measure. The intensities of each of the four equivalent reflexions were measured on a Hilger photometer and the mean value taken. The weakest reflexions were estimated visually. Crystals with cross-sections approximately $0{\cdot}1 \times 0{\cdot}1$ mm were used and no correction for absorption was applied. The various levels were placed on an approximately common absolute scale by means of calculated structure factors based on the two-dimensionally refined structure.

Refinement of the structure

The coordinates of the original structure were first refined by a number of difference syntheses in the a and c projections, including finally the thirteen hydrogen atoms. A common isotropic temperature factor was applied, and the values 0·12 and 0·10 were obtained for the R index. These coordinates were

Table 1. *Observed and calculated structure factors*

Within the groups of constant k the columns contain h, l, $10|F_o|$, $10|F_c|$ and the phase angle in degrees. The minimum observable $10|F_o|$ is listed for reflexions too weak to be observed; these are marked *u*

k = 0					
h	l				
2	0	920	862	0	
4	0	335	343	0	
6	0	118	109	180	
8	0	115	109	180	
10	0	72	70	0	
12	0	90	85	180	
14	0	243	230	180	
16	0	29	25	0	
1	1	79	74	270	
2	1	666	722	90	
3	1	479	528	90	
4	1	469	496	270	
5	1	28	22	90	
6	1	139	125	90	
7	1	47	39	270	
8	1	117	110	270	
9	1	157	154	90	
10	1	34	12	270	
11	1	125	120	90	
12	1	118	116	90	
13	1	285	276	90	
14	1	36	17	90	u
15	1	98	99	90	
16	1	72	68	270	
0	2	30	26	0	
1	2	88	92	0	
2	2	567	617	180	
3	2	243	245	0	
4	2	509	535	180	
5	2	408	402	180	
6	2	173	161	180	
7	2	53	56	180	
8	2	31	29	180	
9	2	275	272	180	
10	2	141	141	0	
11	2	22	22	180	
12	2	22	4	180	
13	2	100	92	0	
14	2	62	63	180	
15	2	57	54	0	
16	2	64	58	180	
17	2	34	35	0	
1	3	248	257	270	
2	3	384	403	90	
3	3	117	110	90	
4	3	153	147	90	
5	3	73	69	90	
6	3	79	78	90	
7	3	190	184	270	
8	3	104	110	270	
9	3	82	80	270	
10	3	45	23	270	u
11	3	89	86	270	
12	3	42	29	90	u
13	3	39	9	90	u
14	3	39	11	270	u
15	3	34	14	90	u
0	4	118	119	0	
1	4	95	95	0	
2	4	57	59	0	
3	4	88	92	0	
4	4	87	87	0	
5	4	148	132	180	
6	4	79	70	0	
7	4	83	80	180	
8	4	104	95	180	
9	4	44	25	180	u
10	4	61	62	0	
11	4	38	12	180	u
12	4	38	7	180	u
13	4	68	66	0	
14	4	25	16	0	u
1	5	45	19	270	u
2	5	53	44	90	
3	5	140	134	90	
4	5	43	5	270	u
5	5	77	72	90	
6	5	44	39	90	
7	5	39	11	270	u
8	5	49	45	270	
9	5	47	41	270	
10	5	33	15	90	u
11	5	117	116	270	
0	6	72	71	180	
1	6	66	68	0	
2	6	41	41	180	
3	6	36	27	0	
4	6	37	11	0	u
5	6	34	27	180	
6	6	31	23	180	
k = 1					
h	l				
1	0	396	400	90	
2	0	229	228	180	
3	0	27	24	90	
4	0	386	386	0	
5	0	78	77	270	
6	0	216	221	0	
7	0	80	80	90	
8	0	51	54	180	
9	0	32	24	90	u
10	0	80	77	0	
11	0	37	33	90	
12	0	223	221	180	
13	0	41	29	270	
14	0	44	44	180	
15	0	35	3	270	u
16	0	75	70	0	
17	0	59	56	270	
0	1	134	143	90	
1	1	702	728	15	
2	1	254	268	255	
3	1	519	537	144	
4	1	171	178	232	
5	1	186	187	187	
6	1	301	294	187	
7	1	308	301	129	
8	1	138	141	205	
9	1	78	80	306	
10	1	106	99	154	
11	1	178	177	93	
12	1	227	229	90	
13	1	109	105	124	
14	1	130	128	314	
15	1	83	81	134	
16	1	48	42	47	
17	1	32	19	103	u
0	2	305	324	270	
1	2	360	367	77	
2	2	206	201	220	
3	2	93	85	235	
4	2	164	166	166	
5	2	232	225	337	
6	2	226	224	293	
7	2	79	75	202	
8	2	196	194	246	
9	2	138	131	231	
10	2	104	98	3	
11	2	84	85	55	
12	2	78	72	145	
13	2	48	42	129	
14	2	66	68	109	
15	2	22	19	207	
16	2	59	53	321	
17	2	36	37	267	
0	3	172	160	270	
1	3	155	151	56	
2	3	220	231	8	
3	3	208	196	98	
4	3	203	200	286	
5	3	167	170	17	
6	3	100	95	89	
7	3	89	86	70	
8	3	43	37	65	
9	3	117	117	324	
10	3	104	109	135	
11	3	53	51	8	
12	3	44	42	50	
13	3	39	31	276	
14	3	42	30	306	
15	3	58	49	259	
0	4	36	32	270	
1	4	110	100	94	
2	4	102	94	91	
3	4	180	178	125	
4	4	200	197	71	
5	4	127	129	88	
6	4	92	89	29	
7	4	59	58	199	
8	4	53	52	21	
9	4	42	30	308	u
10	4	89	90	347	
11	4	54	56	22	
12	4	44	39	220	
13	4	39	33	34	
14	4	35	42	106	
0	5	141	140	90	
1	5	119	120	134	
2	5	117	112	90	
3	5	44	38	251	
4	5	72	63	175	
5	5	46	40	150	
6	5	124	124	161	
7	5	71	70	152	
8	5	37	36	228	
9	5	36	19	258	u
10	5	35	26	239	
11	5	28	11	109	u
0	6	32	28	270	
1	6	121	118	346	
2	6	37	40	257	
3	6	56	53	265	
4	6	32	21	125	u
5	6	35	33	315	
6	6	28	17	89	u
7	6	50	52	229	
k = 2					
h	l				
0	0	261	257	180	
1	0	592	552	270	
2	0	193	204	0	
3	0	220	219	90	
4	0	436	437	180	
5	0	349	344	270	
6	0	102	99	0	
7	0	46	50	270	
8	0	28	22	180	u
9	0	244	238	270	
10	0	197	203	0	
11	0	182	181	90	
12	0	158	157	0	
13	0	184	184	90	
14	0	177	180	0	
15	0	39	31	270	
16	0	31	33	0	
17	0	31	26	270	
0	1	237	230	180	
1	1	1009	953	69	
2	1	145	138	340	
3	1	541	546	101	
4	1	338	334	352	
5	1	261	258	26	
6	1	128	123	34	
7	1	90	93	236	
8	1	126	123	89	
9	1	128	124	221	
10	1	83	86	170	
11	1	42	42	282	
12	1	40	22	331	u
13	1	113	118	208	
14	1	39	21	355	u
15	1	35	23	350	u
16	1	36	36	184	
17	1	28	16	266	u
0	2	25	28	0	
1	2	315	296	353	
2	2	268	265	93	
3	2	86	88	24	
4	2	184	172	86	
5	2	247	229	19	
6	2	107	103	84	
7	2	160	159	90	
8	2	43	45	257	
9	2	57	49	327	
10	2	49	49	190	
11	2	179	190	122	
12	2	44	47	192	
13	2	26	25	76	
14	2	57	59	165	
15	2	39	42	168	
16	2	62	61	210	
17	2	18	11	128	u
0	3	146	138	180	
1	3	216	213	277	
2	3	297	306	178	
3	3	126	124	251	
4	3	39	38	353	
5	3	101	104	290	
6	3	50	57	276	
7	3	38	33	0	u
8	3	168	174	210	
9	3	126	130	44	
10	3	40	21	258	u
11	3	99	115	29	
12	3	38	24	8	u
13	3	35	33	150	
14	3	47	45	84	
15	3	30	24	325	u
0	4	41	38	0	
1	4	49	50	278	
2	4	56	65	91	
3	4	39	19	255	u
4	4	51	55	359	
5	4	110	111	263	
6	4	167	181	112	
7	4	54	61	49	
8	4	51	58	76	
9	4	47	48	315	
10	4	43	43	160	
11	4	76	84	292	
12	4	36	30	79	
13	4	87	82	286	
14	4	20	6	196	u
0	5	157	168	0	
1	5	41	44	18	
2	5	113	114	2	
3	5	39	24	160	u
4	5	67	85	23	
5	5	38	22	36	u
6	5	36	25	315	u
7	5	35	37	339	
8	5	39	44	251	
9	5	34	28	218	
10	5	31	21	295	u
11	5	24	18	265	u
0	6	19	7	180	u
1	6	84	88	104	
2	6	33	39	286	
3	6	39	46	75	
4	6	33	33	276	
5	6	29	10	151	u
6	6	26	4	282	u
k = 3					
h	l				
1	0	446	454	90	
2	0	487	489	0	
3	0	314	327	90	
4	0	425	440	0	
5	0	72	68	90	
6	0	112	118	0	
7	0	264	253	90	
8	0	183	173	180	
9	0	52	45	270	
10	0	198	193	0	
11	0	72	68	270	
12	0	96	92	0	
13	0	80	82	90	
14	0	142	138	0	
15	0	148	150	270	
16	0	91	85	0	
17	0	29	4	270	u
0	1	656	653	270	
1	1	547	549	125	
2	1	326	347	20	
3	1	248	268	174	
4	1	62	52	309	
5	1	317	320	99	
6	1	357	345	26	
7	1	86	99	39	
8	1	309	296	301	
9	1	78	72	179	
10	1	93	88	325	
11	1	262	256	346	
12	1	174	174	45	
13	1	80	84	35	
14	1	57	58	102	
15	1	34	21	193	u
16	1	43	33	222	
17	1	46	44	71	
0	2	42	51	90	
1	2	334	342	205	
2	2	222	229	47	
3	2	60	51	288	
4	2	53	63	191	
5	2	452	455	323	
6	2	125	126	247	
7	2	132	125	299	
8	2	225	221	146	
9	2	74	67	200	
10	2	80	79	252	
11	2	40	9	359	u
12	2	64	72	246	
13	2	167	169	72	
14	2	93	98	199	
15	2	47	43	93	
16	2	60	62	182	
0	3	56	41	270	
1	3	129	127	15	
2	3	182	177	17	
3	3	147	151	289	
4	3	189	167	67	
5	3	64	65	182	
6	3	155	146	38	
7	3	64	63	340	
8	3	92	89	82	
9	3	49	42	224	
10	3	108	108	252	
11	3	80	77	294	
12	3	37	31	230	
13	3	41	40	157	
14	3	41	39	243	
15	3	42	39	145	
0	4	132	135	270	
1	4	38	30	13	u
2	4	38	25	331	u
3	4	104	111	41	
4	4	43	42	241	
5	4	90	87	352	
6	4	40	28	19	u
7	4	54	52	172	
8	4	83	85	73	
9	4	135	132	220	
10	4	68	65	27	
11	4	62	63	224	
12	4	42	39	21	
13	4	29	1	351	u
0	5	41	38	270	
1	5	76	69	329	
2	5	67	63	299	
3	5	60	61	251	
4	5	79	78	218	
5	5	38	29	145	
6	5	39	41	346	
7	5	101	101	82	
8	5	57	54	101	
9	5	31	20	42	u
10	5	30	13	130	u
11	5	22	12	0	u
0	6	98	103	270	
1	6	44	44	186	
2	6	30	26	157	u
3	6	59	45	170	
4	6	30	21	166	
5	6	28	21	243	
6	6	23	19	10	u
k = 4					
h	l				
0	0	57	54	180	
1	0	341	351	90	
2	0	145	140	0	
3	0	172	164	270	
4	0	63	49	180	
5	0	323	306	90	
6	0	302	283	180	
7	0	99	105	270	
8	0	106	93	180	
9	0	199	180	270	
10	0	141	136	180	
11	0	148	145	90	
12	0	176	185	0	
13	0	36	10	90	u
14	0	149	141	180	
15	0	49	46	270	
16	0	44	38	180	
17	0	27	10	270	u
0	1	194	176	180	
1	1	322	346	226	
2	1	314	328	121	
3	1	488	506	303	
4	1	272	262	79	
5	1	228	225	296	
6	1	325	329	319	
7	1	303	293	191	
8	1	47	41	227	
9	1	36	16	282	u
10	1	107	112	280	
11	1	60	64	327	
12	1	161	155	92	
13	1	43	39	218	
14	1	86	89	108	
15	1	94	97	46	
16	1	45	48	267	
17	1	68	71	96	
0	2	24	16	180	u
1	2	232	225	84	
2	2	106	115	106	
3	2	183	182	16	
4	2	232	234	214	
5	2	234	216	105	
6	2	110	105	351	
7	2	68	70	331	
8	2	116	110	313	
9	2	181	187	257	
10	2	296	289	317	
11	2	126	126	345	
12	2	180	180	356	
13	2	49	44	280	
14	2	66	71	162	
15	2	26	21	277	
16	2	28	11	14	u
0	3	44	39	180	
1	3	57	56	154	
2	3	77	78	149	
3	3	183	180	99	
4	3	36	26	341	
5	3	39	38	15	
6	3	166	169	320	
7	3	225	230	101	
8	3	109	107	196	
9	3	112	113	47	
10	3	145	145	242	
11	3	65	75	298	
12	3	62	60	95	
13	3	32	24	268	u
14	3	59	57	188	
15	3	26	9	76	u
0	4	76	81	0	
1	4	237	234	308	
2	4	134	146	342	
3	4	151	150	286	
4	4	227	229	220	
5	4	50	53	12	
6	4	68	68	185	
7	4	43	37	32	
8	4	53	49	141	
9	4	86	89	176	
10	4	43	43	259	
11	4	32	19	16	u
12	4	57	54	135	
13	4	31	33	337	
0	5	65	68	0	
1	5	97	99	251	
2	5	39	26	133	u
3	5	57	60	178	
4	5	51	53	63	
5	5	63	66	216	
6	5	50	59	330	
7	5	43	51	193	
8	5	34	21	125	u
9	5	80	75	243	
10	5	52	44	31	
0	6	38	33	0	
1	6	63	64	42	
2	6	31	19	6	u
3	6	41	36	65	
4	6	33	33	250	
5	6	28	26	17	
6	6	40	43	59	
k = 5					
h	l				
1	0	143	154	270	
2	0	113	112	180	
3	0	400	394	90	
4	0	339	330	0	
5	0	122	126	270	
6	0	80	81	180	
7	0	391	384	270	
8	0	31	22	180	u
9	0	307	303	270	
10	0	53	49	180	
11	0	92	87	270	
12	0	38	20	180	u
13	0	35	31	90	
14	0	46	39	180	
15	0	35	34	90	
16	0	70	68	0	
17	0	26	9	270	u
0	1	884	905	90	
1	1	131	128	295	
2	1	286	276	204	
3	1	454	449	36	
4	1	322	323	180	
5	1	79	79	216	
6	1	197	199	206	
7	1	30	29	91	
8	1	122	126	230	
9	1	52	52	219	
10	1	73	72	345	
11	1	37	28	354	
12	1	109	116	337	
13	1	67	61	103	
14	1	133	132	305	
15	1	107	111	129	
16	1	45	48	322	
17	1	45	50	172	
0	2	175	175	270	
1	2	236	230	328	
2	2	309	318	189	
3	2	236	241	81	
4	2	182	184	316	
5	2	139	136	58	
6	2	250	249	129	
7	2	179	176	351	
8	2	136	135	67	
9	2	96	90	86	
10	2	88	33	31	
11	2	74	69	84	
12	2	50	47	235	
13	2	101	93	126	
14	2	55	52	25	
15	2	51	50	151	
16	2	70	74	63	
0	3	37	32	90	
1	3	71	66	144	
2	3	108	104	275	
3	3	68	68	197	
4	3	92	99	8	
5	3	324	326	219	
6	3	121	128	85	
7	3	42	34	228	u
8	3	90	88	203	
9	3	167	165	295	
10	3	102	104	105	
11	3	70	65	326	
12	3	51	49	172	
13	3	63	65	62	
14	3	42	36	128	
15	3	20	12	197	u
0	4	58	45	90	
1	4	61	55	181	
2	4	92	87	201	
3	4	187	184	174	
4	4	66	62	216	
5	4	70	65	162	
6	4	44	32	156	u
7	4	45	45	261	
8	4	52	54	277	
9	4	40	15	182	u
10	4	84	91	238	
11	4	88	85	335	
12	4	49	46	228	
13	4	26	14	282	u
0	5	60	65	270	
1	5	88	89	13	
2	5	41	7	268	u
3	5	40	5	33	u
4	5	41	35	24	
5	5	57	58	215	
6	5	36	10	250	u
7	5	35	34	339	
8	5	47	48	196	
9	5	43	39	23	
10	5	27	22	328	u
0	6	16	3	270	u
1	6	33	14	39	u
2	6	32	17	26	u
3	6	32	28	80	
4	6	44	48	79	
5	6	28	25	248	
k = 6					
h	l				
0	0	450	435	180	
1	0	636	673	90	
2	0	28	15	0	
3	0	159	145	270	
4	0	35	35	180	
5	0	191	187	90	
6	0	43	43	180	
7	0	29	11	270	
8	0	317	303	180	
9	0	156	161	90	
10	0	120	116	180	
11	0	37	14	270	u
12	0	183	180	180	
13	0	81	84	270	
14	0	33	11	180	u
15	0	32	13	90	u
16	0	43	37	180	
0	1	286	280	0	
1	1	240	232	218	
2	1	137	145	189	
3	1	156	147	288	
4	1	216	217	146	
5	1	160	129	267	
6	1	132	135	160	
7	1	91	88	196	
8	1	149	157	228	
9	1	91	94	285	
10	1	131	133	214	
11	1	134	134	236	
12	1	81	87	208	
13	1	77	76	305	
14	1	66	73	173	
15	1	39	37	190	
16	1	77	69	23	
0	2	129	132	0	
1	2	157	135	232	
2	2	244	245	5	
3	2	364	371	211	
4	2	229	235	349	
5	2	113	116	233	
6	2	91	87	311	
7	2	171	166	220	
8	2	98	95	355	
9	2	61	69	89	
10	2	66	72	297	
11	2	93	83	231	
12	2	38	18	52	u
13	2	36	33	53	
14	2	35	39	86	
15	2	29	9	227	u
16	2	60	60	182	
0	3	25	5	0	u
1	3	133	127	170	
2	3	35	23	254	u
3	3	102	92	249	
4	3	108	105	148	
5	3	136	135	144	
6	3	88	87	60	
7	3	111	115	60	
8	3	196	201	21	
9	3	83	76	109	
10	3	98	107	66	
11	3	37	27	123	
12	3	44	46	14	
13	3	43	48	336	
14	3	29	20	147	u
0	4	52	50	0	
1	4	66	68	184	
2	4	83	82	153	
3	4	62	61	96	
4	4	111	120	135	
5	4	111	114	76	
6	4	135	147	197	
7	4	40	38	237	
8	4	51	54	125	
9	4	36	13	145	u
10	4	33	15	117	u
11	4	33	28	245	
12	4	40	36	352	
13	4	28	27	3	
0	5	25	6	180	u
1	5	93	93	96	
2	5	50	50	40	
3	5	52	51	10	
4	5	41	38	186	
5	5	34	18	302	u
6	5	33	26	249	u
7	5	69	65	312	
8	5	30	17	228	u
9	5	65	59	307	
10	5	20	7	159	u
0	6	21	15	180	
1	6	28	20	236	
2	6	27	26	213	
3	6	29	32	55	
4	6	32	33	219	
k = 7					
h	l				
1	0	227	263	90	
2	0	20	15	0	u
3	0	254	240	90	
4	0	267	247	0	
5	0	507	486	90	
6	0	94	101	180	
7	0	72	73	90	
8	0	68	66	180	
9	0	36	9	90	u
10	0	37	9	180	u
11	0	58	58	270	
12	0	37	17	0	u
13	0	44	40	270	
14	0	33	7	180	u
15	0	26	20	90	
16	0	39	46	180	
0	1	81	70	270	
1	1	77	64	40	
2	1	394	349	234	
3	1	74	76	287	

Table 1 (*cont.*)

4	1	162	151	280	
5	1	187	157	357	
6	1	129	122	92	
7	1	37	35	228	
8	1	107	101	358	
9	1	80	73	353	
10	1	79	78	44	
11	1	121	121	241	
12	1	42	46	238	
13	1	56	60	232	
14	1	52	57	93	
15	1	48	55	260	
16	1	47	58	123	
0	2	106	114	90	
1	2	106	106	67	
2	2	87	83	231	
3	2	230	233	239	
4	2	214	221	62	
5	2	93	96	252	
6	2	161	158	139	
7	2	73	78	343	
8	2	42	42	165	
9	2	90	93	290	
10	2	48	52	258	
11	2	68	72	79	
12	2	48	48	327	
13	2	31	28	51	
14	2	45	41	337	
15	2	61	59	23	
0	3	130	127	90	
1	3	130	124	39	
2	3	101	96	180	
3	3	83	73	161	
4	3	197	197	114	
5	3	87	82	103	
6	3	68	71	108	
7	3	222	222	135	
8	3	41	24	179	u
9	3	145	150	112	
10	3	69	73	194	
11	3	37	34	89	
12	3	34	8	109	u
13	3	46	47	108	
14	3	27	3	139	u
0	4	41	34	270	
1	4	41	29	87	
2	4	57	61	329	
3	4	77	72	128	
4	4	83	92	341	
5	4	41	22	65	u
6	4	46	48	310	
7	4	44	41	55	
8	4	44	43	344	
9	4	57	63	165	
10	4	37	26	227	
11	4	31	39	156	
12	4	32	29	258	
0	5	57	61	270	
1	5	38	7	342	u
2	5	40	36	269	
3	5	37	12	124	u
4	5	40	42	77	
5	5	42	42	269	
6	5	53	54	223	
7	5	33	10	16	u
8	5	47	49	260	
9	5	52	52	327	
0	6	56	63	90	
1	6	24	22	73	u
2	6	32	36	65	
k = 8					
h	l				
0	0	314	315	0	
1	0	185	213	270	
2	0	150	160	180	
3	0	142	141	270	
4	0	61	58	180	
5	0	201	200	90	
6	0	215	206	180	
7	0	311	317	90	
8	0	173	178	0	
9	0	107	109	90	
10	0	94	93	0	
11	0	87	97	90	
12	0	105	110	180	
13	0	56	62	90	
14	0	44	22	180	
15	0	28	34	90	
0	1	173	164	180	
1	1	160	148	206	
2	1	224	203	123	
3	1	90	95	49	
4	1	296	281	144	
5	1	92	89	73	
6	1	105	100	142	
7	1	116	116	220	
8	1	176	183	247	
9	1	133	146	258	
10	1	98	91	101	
11	1	111	120	305	
12	1	37	7	91	u
13	1	35	20	160	u
14	1	47	48	37	
15	1	36	40	350	
16	1	22	11	50	
0	2	29	35	0	
1	2	113	111	199	
2	2	75	68	103	
3	2	209	198	305	
4	2	60	54	188	
5	2	87	74	7	
6	2	198	200	142	
7	2	136	134	339	
8	2	198	191	56	
9	2	183	178	301	
10	2	115	100	34	
11	2	85	86	350	
12	2	37	28	179	
13	2	46	34	275	
14	2	76	64	299	
15	2	42	38	85	
0	3	184	176	0	
1	3	101	103	305	
2	3	46	44	14	
3	3	122	121	342	
4	3	42	42	328	
5	3	259	273	55	
6	3	174	173	328	
7	3	37	42	161	
8	3	108	108	256	
9	3	76	69	297	
10	3	67	71	76	
0	4	148	146	0	
1	4	53	52	158	
2	4	109	109	29	
3	4	94	86	45	
4	4	85	91	300	
5	4	66	72	113	
6	4	36	40	127	
7	4	142	151	164	
0	5	33	30	0	
1	5	40	44	55	
2	5	43	54	138	
k = 9					
h	l				
1	0	37	26	270	
2	0	152	174	180	
3	0	276	277	90	
4	0	114	126	180	
5	0	113	106	90	
6	0	30	38	0	
7	0	153	165	270	
8	0	52	54	180	
9	0	33	7	270	u
10	0	40	18	180	
11	0	141	159	90	
12	0	91	97	180	
13	0	28	6	90	u
14	0	26	26	180	
15	0	108	131	90	
0	1	107	98	270	
1	1	98	93	70	
2	1	132	123	174	
3	1	83	70	113	
4	1	85	73	183	
5	1	204	220	175	
6	1	125	126	160	
7	1	139	139	89	
8	1	114	110	131	
9	1	218	249	3	
10	1	59	48	292	
11	1	72	60	105	
12	1	40	37	131	
13	1	58	56	277	
14	1	48	43	249	
15	1	60	70	297	
0	2	110	118	270	
1	2	134	126	7	
2	2	118	105	352	
3	2	57	50	81	
4	2	127	126	96	
5	2	29	25	35	
6	2	288	291	44	
7	2	209	212	255	
8	2	124	121	293	
9	2	28	29	224	
10	2	61	61	356	
11	2	58	49	80	
12	2	85	68	33	
13	2	40	28	253	
14	2	27	22	44	
0	3	82	71	270	
0	4	40	30	270	
0	5	22	7	270	u
k = 10					
h	l				
0	0	27	13	0	u
1	0	102	108	270	
2	0	157	179	0	
3	0	208	212	90	
4	0	73	80	180	
5	0	32	39	270	
6	0	80	84	180	
7	0	73	70	90	
8	0	53	57	0	
9	0	85	89	270	
10	0	31	11	180	u
11	0	80	80	270	
12	0	28	28	180	u
13	0	49	43	270	
14	0	44	48	0	
15	0	40	47	270	
0	1	189	194	0	
1	1	149	137	28	
2	1	160	157	342	
3	1	171	179	171	
4	1	114	111	4	
5	1	121	124	59	
6	1	189	183	234	
7	1	126	122	9	
8	1	95	92	241	
9	1	44	41	348	
10	1	56	60	178	
11	1	70	69	79	
12	1	33	26	354	u
13	1	28	16	93	u
14	1	23	19	2	u
0	2	135	131	180	
1	2	68	67	78	
2	2	129	124	210	
3	2	92	85	91	
4	2	133	136	228	
5	2	79	76	300	
6	2	108	110	270	
7	2	57	61	172	
8	2	86	82	310	
9	2	56	52	326	
10	2	104	94	38	
11	2	27	17	37	
12	2	21	17	26	u
13	2	25	19	194	
14	2	17	12	181	
0	3	30	19	0	
0	4	90	80	0	
0	5	42	36	180	
k = 11					
h	l				
1	0	29	12	90	u
2	0	104	112	0	
3	0	30	17	90	u
4	0	64	80	0	
5	0	31	18	90	u
6	0	99	118	0	
7	0	32	34	270	u
8	0	62	63	0	
9	0	64	68	90	
10	0	81	96	0	
11	0	28	6	270	u
12	0	26	8	0	u
13	0	27	23	90	
0	1	104	98	270	
1	1	49	41	290	
2	1	97	95	73	
3	1	166	156	310	
4	1	152	152	114	
5	1	98	98	243	
6	1	110	111	105	
7	1	101	92	292	
8	1	111	113	235	
9	1	59	52	220	
10	1	85	86	248	
11	1	44	41	266	
12	1	29	14	268	u
13	1	25	14	281	u
14	1	46	47	186	
0	2	32	35	90	
1	2	131	125	243	
2	2	54	47	238	
3	2	22	19	65	u
4	2	122	117	193	
5	2	75	74	273	
6	2	173	173	199	
7	2	137	144	225	
8	2	141	141	165	
9	2	80	74	179	
10	2	23	7	197	u
11	2	52	46	289	
12	2	87	78	83	
13	2	37	38	7	
0	3	30	34	270	
0	4	27	24	270	
0	5	44	37	90	
k = 12					
h	l				
0	0	33	17	180	u
1	0	237	278	270	
2	0	223	241	0	
3	0	54	64	270	
4	0	46	41	0	
5	0	95	100	90	
6	0	113	121	180	
7	0	71	83	270	
8	0	47	49	0	
9	0	79	95	270	
10	0	35	35	180	
11	0	60	69	270	
12	0	33	36	0	
0	1	97	90	0	
1	1	109	109	82	
2	1	144	146	351	
3	1	98	86	326	
4	1	185	179	56	
5	1	138	130	197	
6	1	137	137	310	
7	1	96	95	212	
8	1	44	37	192	
9	1	62	61	237	
10	1	73	75	42	
11	1	36	37	19	
12	1	38	34	7	
13	1	28	29	355	
0	2	40	50	180	
1	2	55	45	86	
2	2	127	110	305	
3	2	182	177	62	
4	2	57	56	192	
5	2	95	95	84	
6	2	102	89	142	
7	2	90	92	166	
8	2	73	61	76	
9	2	39	34	228	
10	2	35	27	171	
11	2	61	47	316	
12	2	18	16	184	
0	3	287	257	180	
0	4	23	8	180	u
0	5	25	16	180	
k = 13					
h	l				
1	0	52	47	270	
2	0	78	100	0	
3	0	127	147	90	
4	0	119	128	180	
5	0	34	9	90	u
6	0	108	116	180	
7	0	34	32	270	
8	0	31	24	180	u
9	0	29	9	270	u
10	0	113	122	180	
11	0	38	39	90	
0	1	95	93	270	
1	1	84	80	330	
2	1	42	33	337	
3	1	147	138	62	
4	1	90	95	58	
5	1	200	206	117	
6	1	64	54	238	
7	1	110	117	122	
8	1	89	86	116	
9	1	89	94	93	
10	1	33	36	58	
11	1	39	39	367	
12	1	32	29	323	
0	2	84	89	90	
1	2	112	116	171	
2	2	85	77	53	
3	2	87	80	84	
4	2	63	56	289	
5	2	120	95	160	
6	2	39	34	138	
7	2	32	26	259	
8	2	92	74	331	
9	2	38	28	342	
10	2	20	11	227	u
11	2	44	38	95	
0	3	39	32	270	
0	4	20	10	270	u
k = 14					
h	l				
0	0	52	65	0	
1	0	114	139	270	
2	0	50	59	180	
3	0	111	139	270	
4	0	38	38	180	
5	0	83	99	270	
6	0	61	64	180	
7	0	34	40	270	
8	0	120	142	180	
9	0	98	114	90	
10	0	37	37	0	
0	1	85	85	0	
1	1	66	54	51	
2	1	42	38	31	
3	1	42	38	29	
4	1	39	39	323	u
5	1	56	55	253	
6	1	73	66	98	
7	1	37	26	10	
8	1	52	62	13	
9	1	38	38	22	
10	1	26	26	342	u
11	1	42	48	216	
0	2	39	32	0	
1	2	79	72	80	
2	2	34	28	321	
3	2	74	68	81	
4	2	97	74	65	
5	2	32	30	79	
6	2	70	62	55	
7	2	29	24	233	
8	2	33	29	23	
9	2	85	74	133	
10	2	42	39	0	
0	3	26	19	180	
0	4	53	42	0	
k = 15					
h	l				
1	0	33	20	270	u
2	0	100	127	0	
3	0	102	123	270	
4	0	132	153	0	
5	0	30	41	90	u
6	0	65	67	0	
7	0	28	11	270	u
8	0	45	57	0	
0	1	38	25	90	u
1	1	38	29	185	
2	1	38	31	178	
3	1	40	28	207	
4	1	57	51	356	
5	1	36	18	325	u
6	1	34	24	336	u
7	1	64	56	218	
8	1	49	42	25	
9	1	78	74	282	
0	2	92	71	90	
1	2	46	37	46	
2	2	51	40	251	
3	2	48	33	275	
4	2	51	43	193	
5	2	46	31	117	
6	2	67	51	220	
7	2	59	52	88	
8	2	23	16	154	
9	2	14	5	297	u
0	3	88	69	270	
0	4	29	33	90	
k = 16					
h	l				
0	0	52	63	0	
1	0	33	35	90	
2	0	28	15	180	u
3	0	97	127	90	
4	0	27	16	180	u
5	0	26	11	270	u
6	0	24	14	180	u
7	0	40	46	90	
0	1	33	8	0	u
1	1	81	83	336	
2	1	74	78	346	
3	1	68	66	342	
4	1	27	31	287	u
5	1	30	18	295	u
6	1	29	30	118	
7	1	50	45	318	
8	1	39	34	241	
0	2	21	14	0	u
1	2	81	67	260	
2	2	54	44	229	
3	2	20	10	331	u
4	2	20	6	96	u
5	2	44	36	349	
6	2	46	38	228	
7	2	33	28	358	
0	3	42	39	180	
k = 17					
h	l				
1	0	46	52	90	
2	0	30	30	0	
3	0	26	23	90	u
4	0	25	25	180	u
5	0	40	45	270	
0	1	32	34	270	
1	1	68	68	32	
2	1	67	56	71	
3	1	82	82	154	
4	1	27	17	172	u
5	1	46	44	143	
6	1	25	25	332	
0	2	134	114	270	
1	2	47	39	117	
2	2	40	30	338	
3	2	27	14	17	u
4	2	55	45	175	
5	2	52	48	318	
k = 18					
h	l				
0	0	62	79	0	
0	1	73	70	180	
1	1	23	19	107	u
2	1	95	95	149	
3	1	77	74	144	

Table 2. *Positional parameters and estimated standard deviations* (Å) *of the non-hydrogen atoms*

Atom	x/a	y/b	z/c	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
C(6)	0·6795	0·2135	0·0400	0·0042	0·0047	0·0051
C(2)	0·5289	0·2461	0·2096	0·0041	0·0044	0·0051
C(4)	0·6180	0·1218	0·3820	0·0042	0·0046	0·0054
C(5)	0·6895	0·1390	0·2133	0·0043	0·0049	0·0062
C(1′)	0·4550	0·1563	0·5593	0·0041	0·0044	0·0045
C(4′)	0·4405	0·0132	0·7512	0·0041	0·0043	0·0045
C(3′)	0·4026	0·0079	0·4704	0·0039	0·0041	0·0041
C(2′)	0·3755	0·1049	0·4186	0·0039	0·0043	0·0045
C(5′)	0·5101	−0·0605	0·8247	0·0048	0·0047	0·0050
O(2)	0·4539	0·2916	0·2102	0·0028	0·0031	0·0037
O(1′)	0·4866	0·1005	0·7660	0·0031	0·0030	0·0033
O(5′)	0·5823	−0·0751	0·6389	0·0032	0·0032	0·0037
O(3′)	0·3252	−0·0522	0·4386	0·0031	0·0031	0·0040
O(2′)	0·2859	0·1298	0·5297	0·0029	0·0033	0·0037
N(1)	0·6026	0·2669	0·0466	0·0035	0·0035	0·0042
N(3)	0·5376	0·1745	0·3808	0·0034	0·0037	0·0040
N(6)	0·7471	0·2343	−0·1330	0·0041	0·0046	0·0050

used as a starting point for the three-dimensional refinement. First, the structure factors for all reflexions were calculated in order to obtain better scale factors for the different layers, giving $R=0\cdot133$ for all observed reflexions. Then three cycles of least-squares calculations were carried out on a FACIT computer with the program of Åsbrink & Bränden (I.U.Cr. *World List of Crystallographic Computer Programs*, 1962. No. 6023 FACIT). Coordinates, isotropic temperature factors and scale factors were refined to give $R=0\cdot10$. In the next step all thirteen hydrogen atoms were included in positions derived partly from the two-dimensional difference maps and partly by stereochemical considerations. A common isotropic B value of 3·0 Å^{-2} was used for these atoms throughout the calculations. Using the same program two more cycles of least-squares refinements were computed, the first one with fixed hydrogen positions, in the second varying also these. This gave $R=0\cdot085$, which was lowered to 0·080 by correcting 13 low order reflexions for secondary extinction (Pinnock, Taylor & Lipson, 1956). Finally, a least-squares refinement program prepared by Keilhau & Seip (Teknisk notat S 65, Math. Sec., Norwegian Defence Res. Establ., Kjeller, Norway) for the Ferranti MERCURY computer was utilized. This program, which is of the block diagonal approximation type, contains mainly the same features as the one described by Cruickshank for the PEGASUS at the Glasgow conference in 1960. The weighting is of the form $w=1/(a+|F_o|+c|F_o|^2)$ for the observed reflexions where a and c were given the values 4·0 and 0·02, respectively. A weight analysis gave average values of $w\Delta^2$ for eight groups of increasing $|F_o|$ near 1·0 with a root mean square deviation of 0·2. The non-observed reflexions which were included in the calculations with half the minimum observable $|F|$ value were given weights proportional to the inverse of the given $|F|$.

The atomic form factors were calculated from the analytical approximation

$$f(x)=A\exp(-ax^2)+B\exp(-bx^2)+C$$

with constants as given by Moore (1963).

Anisotropic temperature factors were introduced and refined together with the positional parameters for all the heavier atoms in five cycles; in two more cycles the positions of the hydrogen atoms were also refined. In the last cycle the mean shift in the positional parameters of carbon, oxygen and nitrogen atoms was of the order one fourth of the estimated standard deviations. The final R value is 0·056 for all observed reflexions. Observed and calculated structure factors are listed in Table 1 and final parameters in Tables 2–4. A difference Fourier synthesis was evaluated for the $hk0$ reflexions where the contribution from the non-hydrogen atoms was subtracted from the observed structure factors. Electron density peaks of appreciable values were found at the hydrogen positions only.

Table 3. *Positional parameters for the hydrogen atoms*

	x/a	y/b	z/c
H(1)	0·752	0·107	0·222
H(2)	0·618	0·072	0·509
H(3)	0·742	0·283	−0·260
H(4)	0·799	0·212	−0·118
H(5)	0·348	−0·114	0·423
H(6)	0·565	−0·117	0·512
H(7)	0·264	0·089	0·617
H(8)	0·471	−0·123	0·855
H(9)	0·553	−0·035	0·985
H(10)	0·374	0·122	0·211
H(11)	0·390	0·005	0·879
H(12)	0·457	−0·011	0·356
H(13)	0·427	0·217	0·628

The estimated standard deviations of the positional parameters of the hydrogen atoms were of the order 0·06 Å. Little significance can thus be attached to the bond lengths where hydrogen atoms are involved (Table 6).

Table 4. *Thermal parameters and* (*in parentheses*) *their estimated standard deviations*

$$T=\exp\{-10^{-4}(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl\}$$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(6)	31 (2)	39 (2)	299 (18)	−11 (3)	15 (11)	−9 (13)
C(2)	32 (2)	30 (2)	292 (17)	−11 (3)	−23 (11)	0 (11)
C(4)	30 (2)	32 (2)	379 (20)	−2 (3)	−6 (11)	27 (13)
C(5)	33 (2)	39 (2)	428 (23)	11 (4)	24 (13)	20 (15)
C(1′)	32 (2)	31 (2)	237 (15)	−12 (3)	9 (11)	12 (11)
C(4′)	34 (2)	29 (2)	195 (13)	−4 (3)	6 (10)	17 (11)
C(3′)	29 (2)	30 (2)	183 (13)	−2 (3)	5 (10)	−13 (10)
C(2′)	25 (2)	32 (2)	229 (14)	11 (3)	14 (9)	−1 (11)
C(5′)	42 (2)	35 (2)	281 (17)	1 (4)	−21 (13)	0 (12)
O(2)	32 (1)	34 (1)	336 (13)	9 (2)	21 (8)	40 (9)
O(1′)	43 (2)	30 (1)	221 (10)	−13 (2)	−6 (8)	13 (8)
O(5′)	37 (2)	44 (2)	376 (15)	18 (3)	−31 (9)	−56 (10)
O(3′)	36 (2)	32 (1)	438 (16)	−3 (2)	−50 (10)	−39 (10)
O(2′)	30 (1)	38 (1)	377 (14)	9 (3)	64 (9)	45 (10)
N(1)	31 (2)	28 (2)	319 (16)	−6 (3)	18 (10)	8 (10)
N(3)	29 (2)	31 (2)	279 (14)	−2 (3)	13 (9)	1 (10)
N(6)	34 (2)	47 (2)	462 (20)	3 (4)	80 (12)	41 (13)

The molecular structure

The bond lengths and bond angles (Fig. 1) calculated from the coordinates in Table 2 are listed in Table 5 together with their estimated standard deviations.

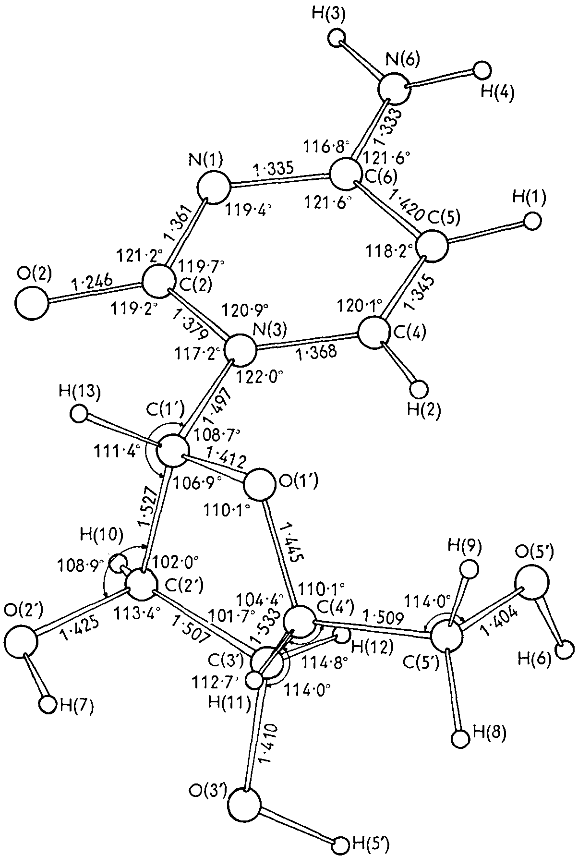


Fig. 1. Bond lengths and angles in cytidine.

The *cytosine* part of the molecule is essentially planar. The deviations from the least-squares plane through the six ring atoms (Plane I) are given in Table 8. Several atoms are slightly but significantly displaced from the plane. Among the ring atoms N(1) shows the greatest departure. The distances from another least-squares plane defined only by the five other ring atoms (Plane II) were therefore calculated. It is seen that these five atoms lie almost exactly in the plane, whereas N(1) deviates by 0·05 Å and the external atoms by 0·01–0·03 Å. The displacement of N(1) from the plane may be associated with the hydrogen bond N(1) · · · O(3′), which forms an angle of 47° with the plane and seems to be pulling N(1) out of it.

The results of the recent three-dimensional crystal structure analysis of Jeffrey & Kinoshita (1963) are also included in Table 5. The agreement between the two sets of parameters is remarkable, especially in view of the different surroundings of the cytosine

Table 5. *Intramolecular bond distances and angles not involving hydrogen atoms, and their estimated standard deviations*

Figures in parentheses are corresponding values in cytosine monohydrate by Jeffrey & Kinoshita (1963)

	Bond length		e.s.d.
C(6)–N(6)	1·333 Å	(1·332) Å	0·007 Å
C(6)–N(1)	1·335	(1·351)	0·006
N(1)–C(2)	1·361	(1·354)	0·006
C(2)–N(3)	1·379	(1·376)	0·006
N(3)–C(4)	1·368	(1·361)	0·006
C(4)–C(5)	1·345	(1·348)	0·007
C(5)–C(6)	1·420	(1·432)	0·007
C(2)–O(2)	1·246	(1·260)	0·005
C(1′)–N(3)	1·497		0·006
C(1′)–C(2′)	1·527		0·006
C(2′)–C(3′)	1·507		0·006
C(3′)–C(4′)	1·533		0·006
C(4′)–O(1′)	1·445		0·005
C(1′)–O(1′)	1·412		0·006
C(2′)–O(2′)	1·425		0·005
C(3′)–O(3′)	1·410		0·005
C(4′)–C(5′)	1·509		0·006
C(5′)–O(5′)	1·404		0·006

	Angle		e.s.d.
N(6)–C(6)–C(5)	121·6°	(120·2°)	0·5°
N(6)–C(6)–N(1)	116·8	(117·8)	0·4
N(1)–C(6)–C(5)	121·6	(122·0)	0·4
C(6)–N(1)–C(2)	119·4	(118·9)	0·4
N(1)–C(2)–N(3)	119·7	(120·1)	0·4
N(1)–C(2)–O(2)	121·2	(121·5)	0·4
O(2)–C(2)–N(3)	119·2	(118·4)	0·4
C(2)–N(3)–C(4)	120·9	(121·3)	0·4
N(3)–C(4)–C(5)	120·1	(120·6)	0·4
C(4)–C(5)–C(6)	118·2	(117·1)	0·5
C(4)–N(3)–C(1′)	122·0		0·4
C(2)–N(3)–C(1′)	117·2		0·4
N(3)–C(1′)–C(2′)	111·4		0·4
N(3)–C(1′)–O(1′)	108·7		0·4
C(2′)–C(1′)–O(1′)	106·9		0·4
C(1′)–C(2′)–O(2′)	108·9		0·4
C(1′)–C(2′)–C(3′)	102·0		0·4
O(2′)–C(2′)–C(3′)	113·4		0·4
C(2′)–C(3′)–O(3′)	112·7		0·4
C(2′)–C(3′)–C(4′)	101·7		0·4
O(3′)–C(3′)–C(4′)	114·0		0·4
C(3′)–C(4′)–O(1′)	104·4		0·3
C(3′)–C(4′)–C(5′)	114·8		0·4
O(1′)–C(4′)–C(5′)	110·1		0·4
C(1′)–O(1′)–C(4′)	110·1		0·3
C(4′)–C(5′)–O(5′)	114·0		0·4

Table 6. *Intramolecular bond distances involving hydrogen*

C(4)–H(2)	0·98 Å
C(5)–H(1)	1·00
C(1′)–H(13)	1·04
C(2′)–H(10)	1·09
C(3′)–H(12)	1·00
C(4′)–H(11)	0·97
C(5′)–H(8)	1·08
C(5′)–H(9)	1·08
N(6)–H(3)	0·97
N(6)–H(4)	0·80
O(2′)–H(7)	0·82
O(3′)–H(5)	0·98
O(5′)–H(6)	0·93

molecule in the two structures. The r.m.s. deviation among corresponding interatomic distances is 0·009 Å, or about 50% above the standard deviation. A similar assertion may be made about the bond angles, in which the r.m.s. deviation is 0·74°. The only noteworthy difference between the two analyses occurs in the bonds from N(1), which were found to be equal in cytosine monohydrate, but differ by 0·026 Å, about four times the e.s.d., in the present structure. The crystals of cytidine probably represent a better approximation to the environments in the nucleic acids than cytosine monohydrate and it seems reasonable to assume that the parameters derived in the present work are those of the corresponding part of the nucleic acids to within about 0·01 Å in the bond lengths and 1° in the bond angles. A remarkable feature of the structure is the shortness of the bond C(4)–C(5), which is found to be only 1·345 Å, not far from the normal double bond length. The high degree of double-bond character in this bond may provide a clue to the understanding of some of the reactions of cytosine and the other pyrimidines of the nucleic acids. The bond C(2)–O(2) is found to be 1·246 Å, probably significantly longer than the normal double bond distance. The corresponding bond in cytosine hydrate is as long as 1·260 Å, but this was thought to be partly due to the exceptionally strong hydrogen bonding of O(2) in that structure.

The bond angles are close to 120° and only those at C(5) and C(6) deviate significantly from this value. The external angles at C(2) and C(6) are not equal, but differ by about 2° at C(2) and 5° at C(6). A similar effect was observed in cytosine hydrate and may be a feature of the free molecule.

Although the hydrogen coordinates are inaccurate, it is of interest to note that the positions derived for H(3) and H(4) correspond to an approximately planar arrangement of the three bonds from N(6), the sum of the angles at N(6) being 359°. The NH_2 group is twisted about 7° in relation to the ring plane (Plane II). H(3) and H(4) lie in the plane of the hydrogen bonds from N(6), but not on the lines N(6) · · · O, the angle between these being only 79°.

The observed bond lengths may be satisfactorily accounted for in terms of the principal resonance structures, as shown by Gerdil (1961) in the similar case of thymine.

The predicted molecular parameters of Pauling & Corey (1956) and Spencer (1959) differ in important respects from those derived in the present work and do not appear to represent a significantly better approximation to the structure than the original two-dimensional analysis. The most serious discrepancy lies in the C–C bonds, which were thought to be equal, but differ by as much as 0·08 Å.

The *glycosidic* linkage N(3)–C(1′) is found to be 1·497 Å, in agreement with the value of 1·492 Å found in adenosine-5-phosphate. It forms an angle of only 0·7° with the least-squares pyrimidine plane. Its angles with the adjacent pyrimidine bonds differ by 5°. The angles at the ribose are close to the tetrahedral value (109° and 111°). The relative orientation of pyrimidine and ribose corresponds to an *anti* conformation of the nucleoside. The angle between the plane N(3)C(1′)O(1′) and the pyrimidine plane is 18°. The factors governing the relative orientation of base and sugar have earlier been discussed by one of us (Furberg, 1952). It was concluded that there is a range of favourable orientations of about 90°, which is likely to be preferred by nucleosides and nucleotides. This has been verified by all subsequent structure analyses, the angle defined above being 6° in cytidylic acid (Alver & Furberg, 1959), 48° in calcium thymidylate and 18° in adenosine-5-phosphate. These values all lie well within the 90° range.

The *ribose*: Least-squares planes were calculated for all combinations of four ring atoms. The one defined by C(1′), O(1′), C(2′) and C(4′) is much better than the others, the deviations lying in the range 0·018–0·033 Å. The fifth atom (C(3′)) is away from this plane by as much as 0·58 Å. Alternatively the ring may be described by means of a series of 'torsion' angles between planes defined by a particular bond and the adjacent ring bonds. These angles are 6·1° for C(1′)–O(1′), 28·1° for C(1′)–C(2′), 37·6° for C(2′)–C(3′), 35·1° for C(3′)–C(4′) and 18·3° for C(4′)–O(1′).

The C–C bonds vary between 1·507 and 1·533 Å, the mean value being 1·519 Å, and some of the deviations may be significant. The two bonds to the ring oxygen atom differ by 0·033 Å, about six times the e.s.d., and we believe that this effect is real. The carbon ring angles vary between 102° and 107°, whereas the angle at the oxygen atom is somewhat greater (110·1°), as found in a number of other investigations of furanose and pyranose sugars (Trueblood *et al.*, 1961; Ferrier, 1963). The external angles lie in the range 109–115°, the mean value being 112·0°. These bond lengths and angles are similar to those reported for calcium thymidylate and adenosine-5-phosphate. Thus, the r.m.s. deviation among corresponding distances in cytidine and adenosine-5-phosphate is 0·022 Å, the largest deviation, 0·037 Å, occurring in the bond C(2)–C(3). The ring bond angles show interesting and parallel variations between 100° and 110° in the three compounds, the angles increasing in the order C(3′) (101·5°), C(2′) (102·1°), C(4′) (105·0°), C(1′) (106·9°) and O(1′) (109·0°). The values quoted are mean values for the three compounds. Their r.m.s. deviation from those found in the present work is only 0·6°. As for the external angles, they vary between 107° and 119° in the same compounds and again the angular variations run parallel, although the deviations, as is to be expected, are greater than for the ring angles. This constancy of the molecular parameters in different compounds indicates that this conformation of ribose is a stable and precisely defined one.

On the basis of proton magnetic resonance studies

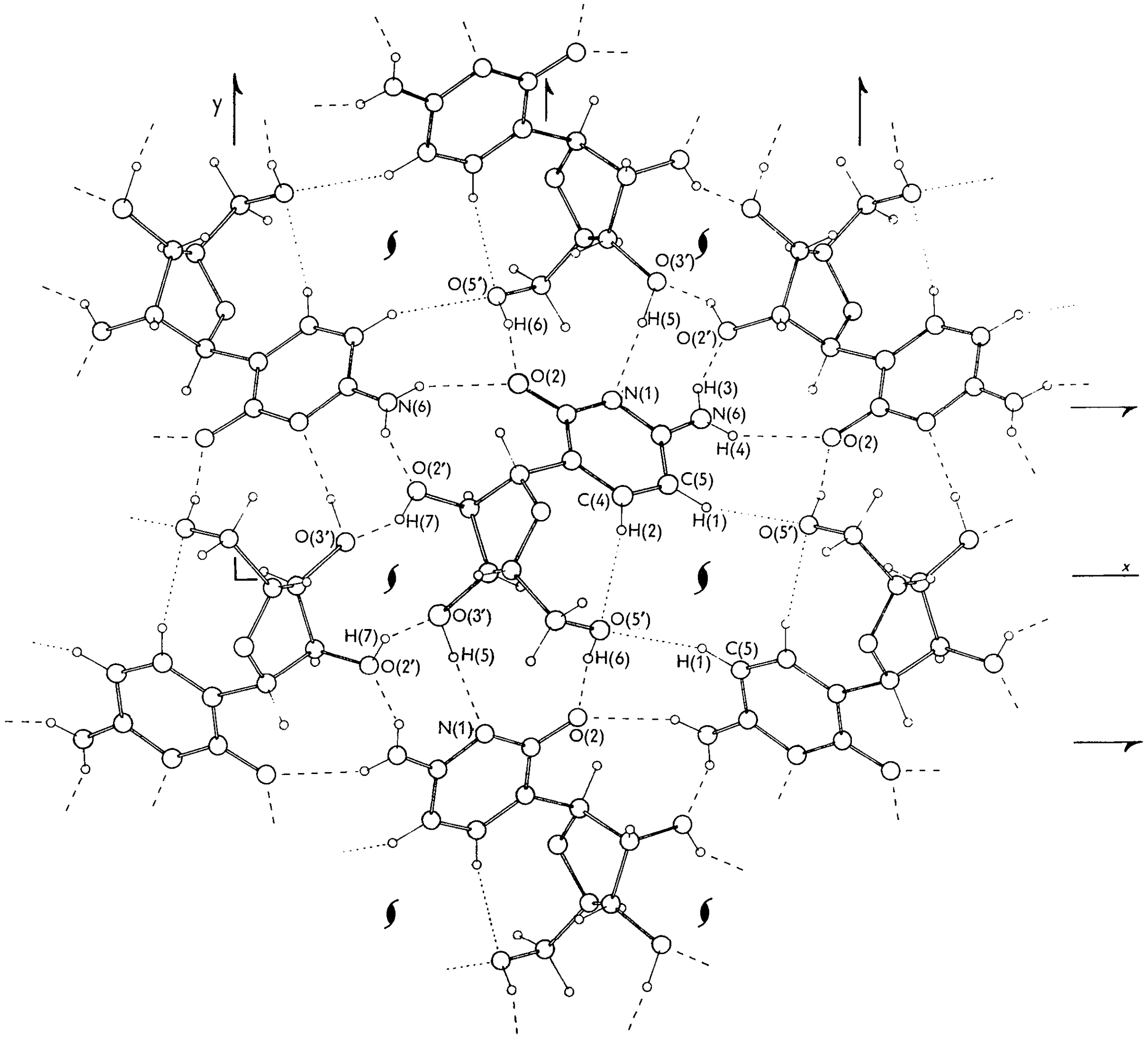


Fig. 2. The *c* projection of the structure of cytidine. Broken lines indicate hydrogen bonds, dotted lines C–H · · · O interactions.

it has been suggested (Jardetzky, 1960) that the ribose conformation in pyrimidine nucleosides is different from that in purine nucleosides. As shown above, this is not necessarily so, at least not in the solid state.

Table 7. *Intermolecular contacts and their estimated standard deviations*

	Length	e.s.d.
N(6)–O(2)	2·945 Å	0·005 Å
N(6)–O(2′)	2·907	0·006
N(1)–O(3′)	2·860	0·005
O(2)–O(5′)	2·708	0·005
O(2′)–O(3′)	2·848	0·005

		Length	e.s.d.
C(4)–O(5′)	(Intramolecular)	3·233 Å	0·006 Å
C(5)–O(5′)	(Intermolecular)	3·351	0·005

Intermolecular forces

The hydrogen bond system described in the original paper is confirmed (Fig. 2). The five hydrogen atoms attached to nitrogen and oxygen all form hydrogen bonds, whose lengths are given in Table 7. In addition, the atom O(5′) is at a relatively short distance from C(4) in the same molecule (3·233 Å) and C(5) in a neighbouring molecule (3·351 Å). In both cases the C–H bond points approximately in the C · · · O direction, making the separations smaller than corresponding to normal van der Waals radii. It appears reasonable to describe these interactions,

Table 8. *Departures from the least-squares planes*

Atom	Plane I	Plane II
N(1)	0·027 Å	−0·053 Å
C(2)	−0·019	0·002
N(3)	0·000	0·000
C(4)	−0·013	−0·004
C(5)	0·006	0·006
C(6)	0·013	−0·004
N(6)	0·040	0·013
O(2)	0·057	0·033
C(1′)	0·021	0·030

especially the intramolecular one, as weak C–H · · · O hydrogen bonds. It is interesting that the four interaction directions from O(5′) are roughly tetrahedrally distributed.

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The Crystal Structures of $NbAs_2$ and $NbSb_2$

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The compounds $NbAs_2$ and $NbSb_2$ are isostructural. The monoclinic unit cells have the dimensions:

$NbAs_2$: $a=9.357$, $b=3.3823$, $c=7.792$ Å; $\beta=119.46°$.
$NbSb_2$: $a=10.239$, $b=3.6319$, $c=8.333$ Å; $\beta=120.07°$.

The space group is $C2$ (C_2^3) and the unit cell contains 4 Nb and 2 × 4 As (Sb) in general positions 4(c). The parameters were found to be Nb: $x_1=0.3444$, $y_1=\frac{1}{2}$, $z_1=0.3044$; As_I: $x_2=0.0948$, $y_2=0.488$, $z_2=0.3928$; As_{II}: $x_3=0.1399$, $y_3=0.067$, $z_3=0.0257$ for $NbAs_2$ and Nb: $x_1=0.343$, $y_1=\frac{1}{2}$, $z_1=0.304$; Sb_I: $x_2=0.096$, $y_2=0.5$, $z_2=0.393$; Sb_{II}: $x_3=0.142$, $y_3=0.0$, $z_3=0.027$ for $NbSb_2$.

The relationships to the $CuAl_2$, $PbCl_2$, MoP_2 and marcasite types of structure are discussed in terms of the general (8–N) rule.

Introduction

In a recent study of niobium arsenides and antimonides (Furuseth & Kjekshus, 1964) the existence of phases with compositions $NbAs_2$ and $NbSb_2$ was established.* No extended ranges of homogeneity of these phases exist, and the compositions were unequivocally determined to be $NbAs_2$ and $NbSb_2$. According to Furuseth & Kjekshus (1964) the unit cells are monoclinic with the following dimensions:

$NbAs_2$:
$a=9.357$, $b=3.3823$, $c=7.792$ Å; $\beta=119.46°$.
$NbSb_2$:
$a=10.239$, $b=3.6319$, $c=8.333$ Å; $\beta=120.07°$.

On the basis of the observed densities, 7·41 g.cm^{-3} ($NbAs_2$) and 8·22 g.cm^{-3} ($NbSb_2$), the unit cells contain 4 NbX_2-groups ($Z_c=3.96$ for $NbAs_2$ and $Z_c=3.98$ for $NbSb_2$).

All reflexions hkl with $h+k=2n+1$ are missing and the possible space groups are accordingly $C2$ (C_2^3), Cm (C_s^3) and $C2/m$ (C_{2h}^3).

Needle-shaped single crystals of $NbSb_2$ grew from the vapour phase. Single crystals of $NbAs_2$ (with a similar shape, but of somewhat smaller dimensions) were obtained by means of a transport reaction using traces of iodine as a transport agent.

Interest in the nature of the chemical bonding in these compounds and their properties as possible semiconducting materials has prompted the investigation of their crystal structures.

Structure determination of $NbAs_2$

A single crystal with a parallelogram-formed (almost square) cross-section was investigated in an integrating Weissenberg camera with Cu K radiation. Intensity measurements of the $h0l$ and $h1l$ reflexions were carried out microphotometrically by the multiple-film technique. The intensities were corrected for the combined Lorentz and polarization factor and for absorption ($\mu R=1.5$). For the calculation of F_c values the atomic scattering factors were taken from Forsyth

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* *Note added in proof.* — The paper by Saini, Calvert & Taylor (1964) which independently confirms the existence of $NbAs_2$ has come to hand since this article was accepted for publication. Their lattice dimensions and observed density are: $a=9.354\pm0.002$, $b=3.381\pm0.002$, $c=7.799\pm0.002$ Å; $\beta=119.43\pm0.08°$; $d_{obs}=7.01$ g.cm^{-3}.