se font vis-à-vis, d'autre part les distances d'une molécule aux trois molécules qui l'encadrent dans la chaîne lui faisant face dans la couche voisine sont toutes supérieures à 4 Å (C_1–C_1=4,03, 4,58 et 4,85 Å; C_2–C_2=4,28 et 4,55 Å). Cependant il s'établit une liaison de van der Waals (C_2–C_2=3,90 Å) avec une quatrième molécule de cette couche située au même niveau dans la chaîne suivante le long de l'axe *a*.

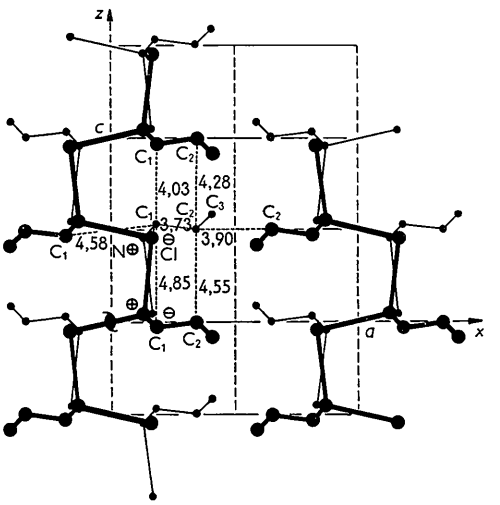

Fig. 8. Dispositions relatives des molécules des couches $y=\frac{1}{4}$ (traits fins) et $y=\frac{3}{4}$ (traits épais).

Après l'opération des axes binaires hélicoïdaux, les ions N^+ et Cl^- viennent se superposer les uns aux autres. Les molécules de pipéridine d'une couche correspondent alors aux intervalles laissés libres entre les molécules de l'autre.

La disposition est donc telle qu'une molécule de pipéridine vient se loger dans une cavité située entre quatre molécules et au fond de laquelle se trouve un atome de chlore.

Comme on le voit encore dans la Fig. 8, on peut également considérer que les chaînes forment des couches perpendiculaires à l'axe *a* en se juxtaposant dans le plan $x=0$. Dans ce cas la disposition des molécules de pipéridine est analogue: elles font saillie de part et d'autre de ces couches et chacune vient se loger dans une cavité de la couche suivante comprise entre quatre molécules et au fond de laquelle se trouve un atome de chlore. Ces saillies et ces cavités sont plus marquées que les précédentes ce qui explique que le cristal s'allonge davantage dans la direction de l'axe *a* que dans celle de l'axe *b*.

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The Crystal and Molecular Structure of Pyrimidine

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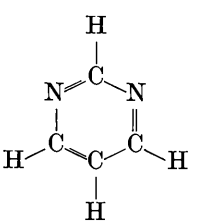
The crystal structure of pyrimidine has been determined by a three-dimensional least-squares analysis. The crystals are orthorhombic, $Pna2_1$, with four molecules in the unit cell. No molecular symmetry is required, and there seems to be a slight but significant departure from the expected *mm* symmetry. The bond lengths obtained from the X-ray analysis have been corrected for the effects of thermal motion which is very important in these simple cyclic molecules. The final *R* factor is 8·7%.

Experimental

Pyrimidine (I) melts at 21 °C. and, like the other azines, volatilizes very readily. The crystals used to obtain the X-ray diffraction intensities were grown from the melt in thin-walled Lindemann-glass capillaries of 0·25 mm. internal diameter. The exposures were taken at −2 °C. in a cold-room. Cu $K\alpha$ radiation and the multiple-film technique were used to take Weissenberg photographs round [100] and [101]. Three layer lines were recorded round [100] and seven round [101]. The relative intensities of the reflexions were estimated visually by comparison with a standard scale obtained from the same crystals, and the different sets of photographs



(I)

Table 1. *Observed and calculated structure factors*

Values are 100 times absolute scale.

h	k	l	F_o	F_c	A_c	B_c
2	0	0	701	1057	+1057	0
4			1093	1600	+1600	0
6			517	546	+ 546	0
8			1077	1052	+1052	0
10			996	1045	+1045	0
12			709	679	− 679	0
14			153	165	+ 165	0
1	1	0	105	98	− 98	0
3			607	587	+ 587	0
4			235	170	+ 170	0
6			1252	1511	−1511	0
7			561	483	− 483	0
8			296	298	+ 298	0
10			1175	1236	−1236	0
11			627	621	+ 621	0
12			399	382	− 382	0
14			96	61	− 61	0
1	2	0	550	557	+ 557	0
2			433	398	− 398	0
4			2636	3113	−3113	0
5			577	528	− 528	0
6			871	889	− 889	0
7			137	133	+ 133	0
9			770	787	+ 787	0
10			527	470	− 470	0
12			231	163	+ 163	0
13			352	369	− 369	0
14			164	172	− 172	0
1	3	0	354	290	− 290	0
2			1906	2163	−2163	0
3			288	282	− 282	0
4			619	556	− 556	0
5			418	391	− 391	0
6			1824	1893	+1893	0
7			257	224	− 224	0
8			283	241	+ 241	0
9			414	416	− 416	0
11			750	663	− 663	0
13			100	94	− 94	0
14			166	171	+ 171	0
0	4	0	2753	3581	−3581	0
1			316	269	+ 269	0
3			150	153	+ 153	0
4			1217	1213	+1213	0
5			853	798	− 798	0
6			361	357	+ 357	0
8			452	377	− 377	0
10			239	235	− 235	0
12			351	357	+ 357	0
1	5	0	955	905	+ 905	0
2			1252	1275	+1275	0
5			503	484	+ 484	0
6			785	779	+ 779	0
7			1008	900	+ 900	0
9			275	254	+ 254	0
10			466	449	+ 449	0
12			216	208	+ 208	0
0	6	0	1452	1545	+1545	0
2			110	16	− 16	0
4			1682	1830	+1830	0
5			361	326	+ 326	0
6			459	392	+ 392	0
7			302	307	− 307	0
8			474	396	− 396	0
9			309	306	− 306	0
10			279	319	+ 319	0
12			220	215	− 215	0
13			120	127	+ 127	0
1	7	0	152	143	− 143	0
2			396	316	+ 316	0
3			626	581	− 581	0
4			420	452	+ 452	0
5			542	525	− 525	0
6			1589	1816	−1816	0
7			198	166	− 166	0
8			139	99	− 99	0
10			375	318	− 318	0
0	8	0	659	749	− 749	0
1			244	220	− 220	0
4			1315	1386	−1386	0
5			515	481	+ 481	0
6			536	509	− 509	0
7			219	228	+ 228	0
10			168	171	− 171	0
1	9	0	211	199	+ 199	0
2			233	291	+ 291	0
3			392	355	+ 355	0
4			179	190	− 190	0
6			418	104	+ 407	0
7			236	222	− 222	0
10			419	339	+ 339	0
0	10	0	715	770	+ 770	0
1			146	127	− 127	0
3			278	256	− 256	0
5			219	172	− 172	0
8			454	357	+ 357	0
1	11	0	135	147	− 147	0
2			288	296	− 296	0
3			93	143	+ 143	0
5			178	205	+ 205	0
6			345	347	− 347	0
4	0	1	1098	1041	− 985	+ 337
6			883	820	+ 511	+ 642
8			1004	1053	−1048	− 102
10			1134	1180	+ 45	+1179
12			382	360	+ 14	+ 360
14			89	69	− 48	+ 49
1	1	1	1067	1241	+ 969	− 776
2			2222	2734	−2423	+1267
3			759	749	+ 749	+ 22
4			1367	1343	+ 136	+1336
5			459	438	− 22	+ 438
6			887	821	− 494	+ 656
7			231	244	− 243	− 8
8			721	748	− 295	+ 687
9			472	472	− 67	− 467
10			741	745	+ 480	+ 570
11			151	176	+ 174	− 28
12			395	376	− 187	− 327
13			221	255	+ 30	+ 253
14			181	185	+ 154	+ 103
1	2	1	1314	1533	+1520	− 200
2			404	396	− 274	− 286
3			975	946	− 366	+ 872

Table 1 (*cont.*)

h k l	F_o	F_c	A_c	B_c	*h k l*	F_o	F_c	A_c	B_c
4	1084	1006	+ 999	+ 117	6	484	442	+ 19	+ 442
5	354	407	− 325	+ 246	7	286	288	− 94	− 273
6	1428	1438	− 379	−1387	8	366	310	− 161	− 265
7	228	233	− 119	− 200	10	105	192	+ 125	+ 146
8	561	482	+ 463	− 133					
9	278	262	+ 184	+ 186	2 8 1	204	131	+ 15	+ 131
10	533	425	− 97	− 414	3	504	495	+ 65	− 491
11	723	689	+ 37	+ 688	4	544	562	+ 536	+ 170
12	173	176	+ 82	− 156	5	258	273	+ 128	− 241
13	232	229	− 189	+ 129	6	923	885	− 293	− 835
14	148	156	+ 72	− 139	7	126	143	− 34	+ 139
					8	296	254	+ 208	− 146
0 3 1	1161	1166	+ 191	+1150	10	279	250	− 64	− 241
1	894	883	− 691	+ 549					
2	451	443	+ 352	− 269	0 9 1	612	679	− 19	− 679
3	381	382	− 344	− 166	2	374	350	+ 312	− 157
4	2217	2095	− 185	−2087	3	155	180	+ 159	+ 86
5	200	205	− 90	+ 185	4	416	394	− 138	− 369
6	589	544	− 153	− 522	5	397	356	− 93	+ 344
8	356	333	+ 50	+ 329	6	268	240	+ 21	− 239
9	281	280	+ 52	+ 275	7	275	240	− 233	+ 55
11	277	234	− 228	− 50	8	238	234	+ 100	− 212
12	195	224	+ 110	− 196					
13	185	218	− 2	− 218	1 10 1	154	179	+ 50	+ 172
14	122	128	− 80	− 99	2	371	376	+ 19	+ 375
					5	228	231	− 213	− 91
1 4 1	750	712	− 182	− 688	6	175	181	+ 65	− 168
2	1725	1790	− 316	−1762	7	135	123	+ 37	− 118
3	151	134	+ 5	− 134					
4	225	191	− 43	− 186	0 11 1	251	297	+ 44	+ 294
5	441	410	− 374	− 168	1	153	184	+ 48	− 177
6	619	509	+ 17	+ 508	2	103	148	+ 81	+ 124
7	822	778	+ 162	− 761	4	198	214	− 47	− 208
8	311	321	+ 271	+ 171					
9	283	283	+ 73	− 274	0 0 2	2192	2498	+2262	−1061
10	323	356	+ 35	− 354	2	2017	2180	+1313	+1740
11	259	242	+ 35	− 240	4	190	165	+ 108	+ 125
12	122	155	− 101	− 117	6	769	799	+ 510	+ 614
13	106	127	+ 111	− 62	8	671	785	+ 713	+ 329
					10	590	564	+ 84	− 557
0 5 1	1785	1899	− 638	−1789					
1	218	218	− 106	+ 191	1 1 2	950	959	− 298	− 911
2	885	781	+ 765	− 156	2	1002	992	− 817	+ 563
3	413	408	− 310	− 266	3	731	670	+ 624	+ 242
4	438	431	+ 18	− 431	4	1023	963	− 459	− 847
5	680	660	+ 206	− 627	5	419	427	+ 187	+ 384
6	232	197	+ 180	− 79	6	393	327	− 80	+ 317
7	467	442	+ 440	+ 46	8	946	930	− 345	− 863
9	359	344	− 9	+ 344	10	466	419	− 419	+ 9
10	344	356	− 289	− 207	11	236	251	+ 248	− 34
12	217	239	+ 99	+ 218	13	135	164	+ 85	+ 141
1 6 1	574	535	− 311	+ 436	0 2 2	613	636	− 565	+ 291
2	300	289	+ 285	− 46	1	1133	1118	+ 910	+ 650
3	340	305	+ 108	+ 286	2	1294	1307	− 777	−1051
4	663	675	− 669	− 94	3	947	928	+ 64	+ 925
5	521	517	+ 385	+ 345	4	794	773	− 770	+ 64
6	1322	1288	+ 260	+1262	5	253	193	− 185	− 55
7	627	589	− 69	+ 585	6	459	365	− 337	− 139
8	498	432	− 424	+ 84	8	175	224	− 167	− 149
9	146	152	− 141	+ 57	10	234	185	− 75	+ 169
10	288	259	+ 79	+ 247	11	315	281	+ 117	+ 255
12	138	145	− 60	+ 132	13	209	241	− 232	+ 66
0 7 1	535	558	+ 237	+ 505	1 3 2	787	715	− 78	+ 711
1	221	202	+ 91	+ 180	2	803	749	− 738	+ 128
2	776	778	− 761	+ 164	3	688	656	− 652	− 73
3	198	186	+ 173	+ 68	4	489	428	+ 89	+ 419
4	1540	1552	+ 180	+1541	5	159	113	− 30	− 109
5	162	177	− 2	− 177	6	537	485	+ 473	− 107

Table 1 (*cont.*)

h	*k*	*l*	F_o	F_c	A_c	B_c
7			385	355	− 353	− 44
8			133	109	+ 107	− 20
11			221	288	− 287	+ 10
13			218	229	− 108	− 202
0	4	2	801	765	− 667	+ 374
1			255	214	− 212	+ 30
2			289	261	− 84	− 247
3			441	412	+ 407	− 64
4			575	518	+ 500	− 133
5			384	347	− 302	− 171
6			177	228	+ 40	− 225
7			483	375	− 65	− 369
8			177	202	− 191	− 67
9			215	232	+ 229	− 33
10			219	276	+ 41	+ 273
1	5	2	567	545	+ 478	+ 263
2			391	363	+ 176	− 317
3			344	326	+ 183	− 270
4			476	455	+ 188	+ 415
5			475	420	− 101	− 408
6			242	186	+ 157	− 98
7			663	649	+ 647	+ 45
8			488	490	+ 150	+ 466
9			296	286	+ 105	+ 266
0	6	2	302	327	+ 57	− 322
1			271	275	+ 3	− 275
2			946	917	+ 405	+ 823
3			440	420	− 270	− 321
4			650	626	+ 626	− 19
5			213	176	+ 138	+ 110
6			196	202	+ 184	+ 84
7			381	345	− 36	+ 343
8			209	140	− 49	+ 132
9			222	236	− 233	+ 39
1	7	2	281	264	− 185	− 188
2			349	334	+ 331	+ 44
3			258	301	− 298	+ 43
4			665	704	− 187	− 679
5			162	158	− 137	+ 78
6			499	493	− 435	+ 231
8			400	368	− 211	− 301
0	8	2	205	163	− 162	− 18
2			665	669	− 327	− 583
4			430	440	− 410	+ 160
5			262	255	+ 233	+ 103
6			194	180	− 179	+ 15
7			207	222	+ 141	+ 171
2	9	2	141	136	+ 126	+ 51
3			205	204	+ 137	+ 151
4			220	250	+ 91	+ 233
5			279	306	+ 148	+ 268
7			134	164	− 162	− 28
0	10	2	279	317	+ 317	− 5
1			141	124	− 102	+ 71
2			124	138	+ 129	+ 49
5			105	107	− 72	− 80
4	0	3	493	451	− 305	+ 332
6			290	268	+ 1	− 268
8			561	627	− 480	+ 403
1	1	3	386	370	+ 155	− 336
2			864	790	− 530	+ 587
3			451	445	+ 419	− 151
4			152	145	− 139	+ 41
6			347	350	− 252	+ 243
8			235	330	− 112	+ 310
1	2	3	542	508	+ 474	− 183
2			154	148	− 12	+ 148
3			542	505	− 24	+ 505
4			384	349	+ 246	− 247
5			416	370	− 334	+ 159
8			235	251	+ 181	− 174
1	3	3	382	326	+ 9	+ 326
2			204	254	+ 159	− 199
3			529	445	− 441	− 60
4			244	232	+ 3	− 232
9			152	149	− 97	− 112
1	4	3	235	221	− 19	− 220
2			177	124	− 90	− 85
3			371	347	− 240	− 250
7			338	333	− 84	− 323
8			207	235	+ 198	− 127
9			159	197	+ 193	− 39
1	5	3	332	313	− 312	+ 25
2			381	365	+ 285	− 228
3			338	327	− 63	+ 321
7			267	269	+ 260	− 69
9			173	208	+ 73	+ 195
1	6	3	370	351	− 211	+ 281
2			223	233	+ 28	− 232
3			220	206	+ 182	+ 97
4			380	374	− 290	+ 235
7			200	205	+ 50	+ 199
8			208	255	− 189	+ 171
9			116	187	− 169	+ 81
2	7	3	496	500	− 373	+ 334
4			162	140	− 32	+ 137
3	8	3	143	150	+ 101	− 111
4			234	276	+ 202	− 188
1	9	3	125	119	+ 119	− 15
2			173	200	+ 120	− 160
3			115	140	+ 70	− 121
0	0	4	299	268	− 120	+ 240
2			213	214	+ 182	+ 112
2	1	4	135	124	+ 89	− 86
3			152	137	+ 136	− 14
5			203	162	+ 108	+ 121
0	2	4	173	134	+ 62	− 119
2			155	153	− 133	− 74
3			267	264	+ 107	+ 242
3	3	4	223	200	− 164	+ 115
5			203	176	− 103	− 143
1	4	4	169	175	+ 31	+ 173
3			136	133	+ 75	− 110
1	5	4	160	215	+ 196	+ 88
3			135	153	+ 27	− 150

were placed on the same relative scale by correlation through common reflexions. A total of 374 independent reflexions was observed. The systematic absences of $h0l$ with h odd and $0kl$ with $k+l$ odd are consistent with two possible space groups, $Pna2_1$ and $Pnam$. The length of c was found from rotation photographs, and the lengths of a and b were determined by the Straumanis method. The results were:

$a = 11{\cdot}698 \pm 0{\cdot}003, b = 9{\cdot}493 \pm 0{\cdot}003, c = 3{\cdot}806 \pm 0{\cdot}005$ Å.

It will be observed that these values are very close to the values $2c; a; b$ (11·822; 9·318; 3·815 Å) found in the isomeric pyrazine (Wheatley, 1957). The cell constants of pyrimidine give a calculated density of 1·259 g.cm.$^{-3}$ on the assumption that there are four molecules in the unit cell. This calculated density is close to the values found for the other azines.

The X-ray structure analysis

The similarity in the cell dimensions of pyrimidine and pyrazine suggests similar crystal structures. It is improbable that the planes of the pyrimidine molecules can be perpendicular to the shortest axis, and it is unlikely, therefore, that $Pnam$ is the correct space group. Consequently the alternative, non-centrosymmetric space group $Pna2_1$ was selected, and this choice was justified by the results of the analysis.

The positions of the molecules were obtained by packing considerations and by trial-and-error calculations of the $hk0$ reflexions. An $hk0$ Fourier synthesis permitted allocation of the peaks to carbon and nitrogen atoms because of the different peak heights. The z coordinates of the atoms were obtained from the known x and y coordinates and from a knowledge of standard bond lengths. Since these trial coordinates gave satisfactory agreement between the observed and calculated $hk0$ and $h0l$ structure factors, immediate refinement was carried out in three dimensions by least-squares methods on the Pegasus computer at the University of Leeds. The programmes were devised by Dr D. W. J. Cruickshank and Miss D. E. Pilling. The weighting scheme of Hughes (1941) was employed.

Seven refinement cycles were performed. After the fifth cycle the reflexions 210, 020, 201, and 011 were omitted, as they seemed to be badly affected by extinction, and hydrogen atoms were introduced. The coordinates obtained from the fifth cycle showed that the molecule was planar, but was not a regular hexagon. However, the principal diagonals of the hexagon intersected at a point and it was assumed that the hydrogen atoms lay on the extensions of these diagonals at a distance of 1·03 Å from the carbon atoms. Thermal parameters were assumed for the hydrogen atoms, and neither the thermal nor the positional parameters of the hydrogen atoms were refined in the last two cycles. The scattering factors employed were those of Berghuis *et al.* (1955). The final R factor, omitting unobserved reflexions and the four reflexions affected by extinction, was 8·7%. A list of F_o, F_c, A_c, and B_c is given in Table 1.

The final atomic coordinates are shown in Table 2, and the final thermal parameters in Table 3. The thermal parameters are defined by the expression

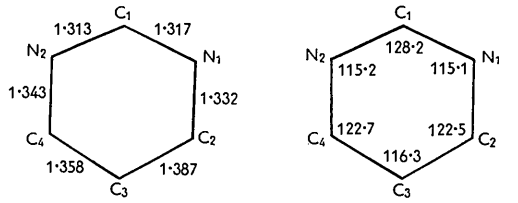


Fig. 1. Molecular dimensions before correction for the effect of thermal motion has been made.

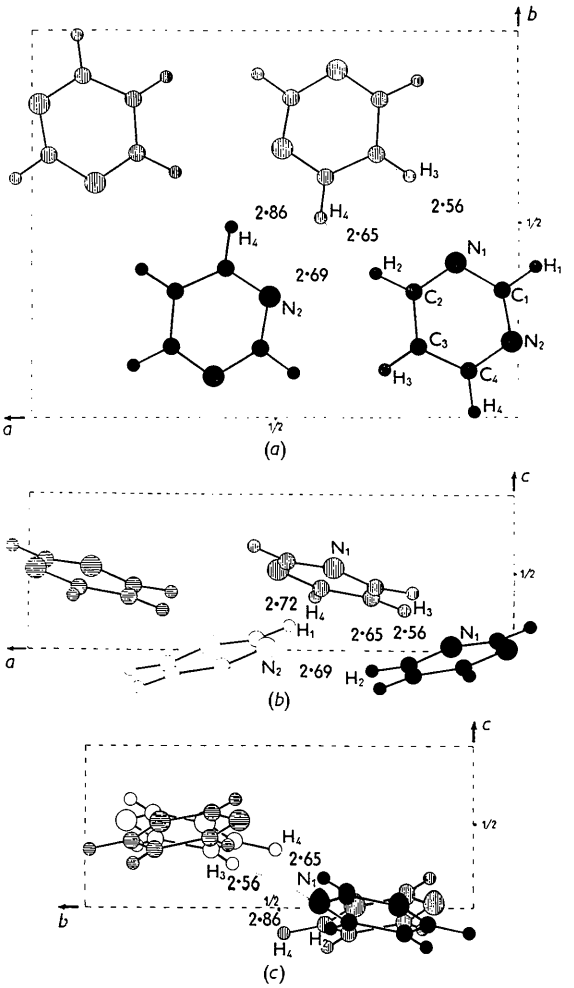


Fig. 2. Pyrimidine: Projections of the structure (*a*) on (001), (*b*) on (010), (*c*) on (100). In each diagram a right-handed set of axes has been chosen, so that the positive direction of the axis normal to the plane of the paper is upwards in (*b*) and downwards in (*a*) and (*c*). If u is the coordinate, in a direction normal to the plane of the paper, of an atom, the full circles lie at $+u$, the empty circles at $\frac{1}{2}-u$, the horizontally cross-hatched circles at $-u$, and the vertically cross-hatched circles at $\frac{1}{2}+u$.

$$f_i = f_0 \left[\exp -(B_{11}h^2 + 2B_{12}hk + 2B_{13}hl + B_{22}k^2 + 2B_{23}kl + B_{33}l^2\right] .$$

The molecular dimensions, before application of any correction for thermal motion (Cruickshank, 1956), are shown in Fig. 1. Projections of the structure down

Table 2. *Final atomic coordinates in* Å

Atom	x	y	z
N_1	1·4946	3·7650	0·1104
N_2	0·1431	1·8245	0·0598
C_1	0·3725	3·0979	0·2818
C_2	2·5174	3·0442	−0·3461
C_3	2·4129	1·6862	−0·6099
C_4	1·1959	1·1234	−0·3930
H_1	−0·422	3·662	0·610
H_2	3·420	3·492	−0·498
H_3	3·220	1·144	−0·936
H_4	1·060	0·114	−0·592

Table 3. *Final thermal parameters in* Å²

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{23}	B_{13}
N_1	4·043	2·929	6·269	−0·174	−0·375	−0·502
N_2	3·600	3·545	6·688	−0·521	−0·494	−0·245
C_1	3·948	3·679	5·740	0·450	−0·806	−0·273
C_2	3·845	3·940	6·372	−0·521	−0·573	−0·170
C_3	3·545	3·664	5·077	0·427	−0·892	−0·454
C_4	4·256	2·937	5·953	0·241	−0·853	−0·482
H	4·3	4·0	7·0	0	−0·6	−0·2

the three principal axes, and also the shortest intermolecular contacts, are shown in Fig. 2. As in pyrazine, the short contacts are between hydrogen and hydrogen atoms, and between hydrogen and nitrogen atoms. The standard deviations of the x, y, and z coordinates average 0·004, 0·005, and 0·008 Å respectively. The standard deviations of the bond lengths are 0·007 Å and of the angles 0·5°.

Discussion of the results

The similarity in the structure of pyrazine and pyrimidine is reflected in the similarity of the intermolecular contacts and of the thermal motions in the two crystals. The thermal parameters show that there is considerable motion round the axis normal to the molecular plane, and even more motion normal to the plane of the molecule. The mean temperature factor for pyrimidine is a little higher than that of pyrazine, no doubt because the analysis of pyrimidine was carried out a little nearer to the melting point. By analogy with pyrazine, it is to be expected that the bond lengths are about 0·020 Å shorter, as a result of the thermal motion, than they should be. The best estimates of the bond lengths are given in Table 4. The figures in this table have a standard deviation of 0·007 Å, and an uncertainty of ±0·015 Å due to the thermal motion. The values of the angles will be virtually unaffected by the thermal motion. The molecule appears to depart from the expected *mm* symmetry, but only in the lengths of the two chemically similar bonds C_2C_3 and C_3C_4. The angles at the nitrogen atoms are less than 120° by about 5°, in agreement with values found previously in similar heterocyclic systems. The angles at the carbon atoms are all greater than 120°—that at C_1 considerably so—with the exception of that at C_3, which is only 116·3°. This low value is unexpected.

Table 4. *Best estimates of the bond lengths after correction for thermal motion*

Bond	Length
C_1N_1	1·34 Å
C_1N_2	1·33
C_2N_1	1·35
C_4N_2	1·36
C_2C_3	1·41
C_3C_4	1·38

The analysis of pyrimidine again emphasizes how imperative it is to eliminate as far as possible the effects of thermal motion by working at low temperatures, if accurate bond lengths are required from X-ray structure determinations.

It had been hoped to complete this study of the known azines by the elucidation of the crystal structure of pyridazine, a sample of which has kindly been furnished by Dr K. Eichenberger of Ciba Ltd. This will not now be possible.

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