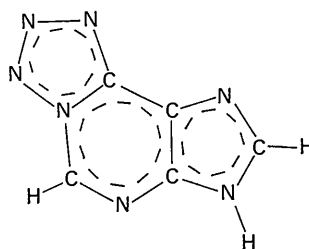# The Molecular Structure of an Azidopurine*

By Jenny Pickworth Glusker, Dick van der Helm,† Warner E. Love,‡ Jean A. Minkin
and A. L. Patterson§

The Institute for Cancer Research, Fox Chase, Philadelphia, Pa. 19111, *U.S.A.*

An azidopurine, prepared from 6-hydrazinopurine by treatment with nitrous acid, is shown to have the chemical structure

The crystals are orthorhombic, space group $Pna2_1$, with cell dimensions $a=11\cdot73$, $b=17\cdot01$, $c=3\cdot78$ Å. One formula unit, $C_5H_3N_7$, and one molecule of water are in the asymmetric unit. The structure was solved by using Harker–Kasper inequalities and refined by the block-diagonal least-squares method to a final R value of 9·6%. The molecule is planar within experimental error and packs with other molecules in planes 3·35 Å apart. There is a close intermolecular C–H⋯N packing distance between molecules in which the H⋯N distance is 2·19 Å. The water molecule is involved in three hydrogen bonds with nitrogen atoms of three molecules at distances 2·70, 2·88 and 3·02 Å.

Introduction

Crystals of an azidopurine of unknown chemical structure were given to us by Dr Aaron Bendich of the Sloan-Kettering Institute, New York. They were obtained in the course of a study of various 6-substituted purines as potential anticancer agents. The compound was prepared as a white precipitate by the action of sodium nitrite on an acidified solution of 6-hydrazinopurine (Bendich, Giner-Sorolla & Fox, 1957). After repeated recrystallizations from ethanol, the material obtained gave analysis figures corresponding to the empirical formula $C_5H_3N_7$, melted with decomposition at 193–195°C, and gave adenine on catalytic reduction with hydrogen. The crystals used for this study were fine clear needles (the c axis was the needle axis) grown from an aqueous solution. They deteriorated with time and tended to become opaque and to split into very thin laths.

Experimental

Unit-cell dimensions were measured on a General Electric XRD-5 diffractometer and a precession camera with Cu $K\alpha$ radiation ($\lambda=1\cdot5418$ Å). The parameters are $a=11\cdot73$, $b=17\cdot01$, and $c=3\cdot78$ Å (estimated standard deviation of 0·13%). The systematic absences ($h0l$, h odd; $0kl$, $(k+l)$ odd) are consistent with either of the space groups $Pnam$ or $Pna2_1$. Subsequently the structure determination showed the latter to be correct. There are four molecules in the unit cell. The measured density of 1·60 g.cm^{-3} indicated the presence of one molecule of water per $C_5H_3N_7$ unit (calculated density: 1·58 g.cm^{-3}).

All data were collected with use of Cu $K\alpha$ radiation. Of the 947 independent reflections measured, 189 were below the threshold of measurement. The $hk0$ data were estimated visually from non-integrated Weissenberg photographs of a needle of cross section $0\cdot6\times0\cdot3$ mm^2. For the remaining data another needle of cross section $0\cdot3\times0\cdot03$ mm^2 was used. Some intensities were measured from integrated Weissenberg photographs with a Nonius densitometer. All Weissenberg data were collected about the c axis. Further data were collected with a General Electric XRD-5 diffractometer equipped with a scintillation counter and a pulse height analyzer. A moving-crystal moving-counter ($\theta/2\theta$) scan technique was used. The intensities were corrected for the

Lorentz and polarization factors and a Wilson plot calculated to give an approximate absolute scale.

Structure determination and refinement

A sharpened two-dimensional $|F|^2$ map was computed from the $hk0$ reflections in the hope that the short c axis would permit the solution of the structure in projection. However attempts to interpret this map by looking for sixfold vectors between pyrimidine rings were not successful, as none of the trial structures could be refined.

Harker–Kasper inequalities were used on the $hk0$ reflections to solve the structure. First packing considerations for the space groups $Pnam$ and $Pna2_1$ showed that the longest direction in the molecule should be approximately parallel to the b axis and that the ring system of the molecule should not pass near the planes $y=0$ or $y=\frac{1}{2}$. Therefore negative signs were assigned to both the 020 and 040 reflections. Two other terms were given arbitrary signs. Then more signs were determined from Harker–Kasper inequalities and Fourier syntheses calculated on 11 and on 21 signs. The maps are very similar and that for 11 terms is shown in Fig. 1. The indicated position of the purine ring system was consistent with the $|F|^2$ map. A Fourier map, calculated on signs from such a trial structure, was much cleaner in appearance than its predecessors.

A linear azide group was tried in two possible positions where it would pack well. One of these trial structures is shown in Fig. 2 ($R=55\%$). This indicates clearly that the correct structure involves a cyclic instead of a linear azide group. One high extra peak was assumed to be due to water of crystallization. The trial structure with a cyclic azide group gave an R value of 40% which dropped to 27% after one stage of refinement using Fourier and difference Fourier maps. The identity of each atom was determined from temperature factor shifts as described later. The final R value for the observed $hk0$ data was 11·5%. In the original maps 2 out of the 11 and 4 out of the 21 signs used were incorrect. It was obvious from the bond lengths that the molecule did not lie in a plane perpendicular to the c axis, therefore excluding the space group $Pnam$.

The z coordinates of the atoms were determined from the three-dimensional $|F|^2$ map. Refinement of the structure was carried out with block-diagonal least-squares matrices*, first isotropically, then anisotropically. Hydrogen atom positions were located from a difference Fourier map. The z parameter of the hydrogen atom H(15) was difficult to establish exactly from this map but the value used refined satisfactorily. The

---

* The weights assigned were $1/|F_o|^2$ for observed reflections and $1/|3F_{min}|^2$ for unobserved reflections. The program omits reflections for which both $|F_o|$ and $|F_c|$ are small, and near-in reflections with large negative values of $|F_o|-|F_c|$. The quantity minimized is $\Sigma w(|kF_o|-|F_c|)^2$.
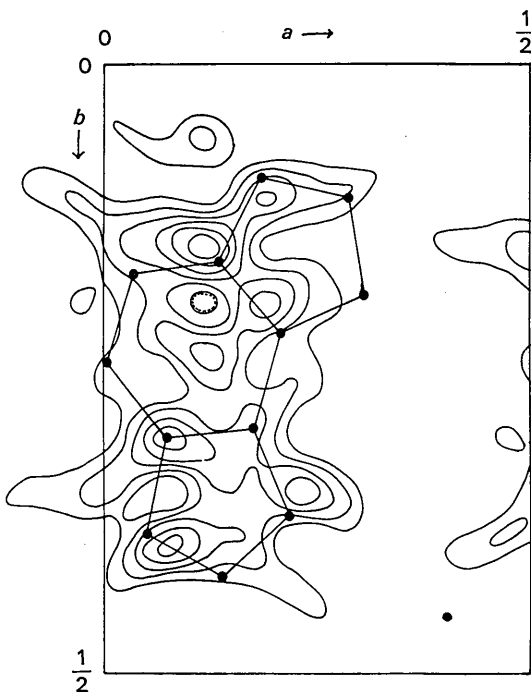

Fig. 1. Trial electron density projection on (001) using 11 terms. Final atomic positions are indicated by filled circles. The contour interval is arbitrary.
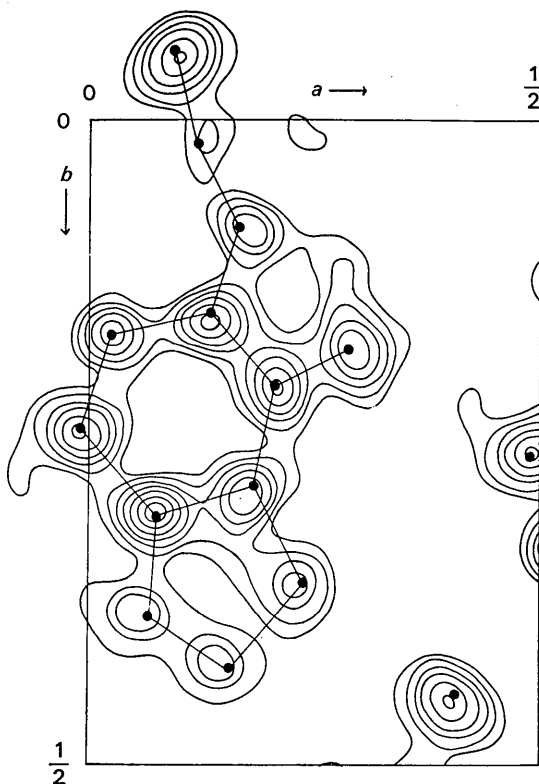

Fig. 2. Trial structure with a linear azide group, indicating a second five-membered ring.

final R values for all the data are 9·6% for the anisotropic refinement (hydrogen atoms isotropic), and 11·6% for the isotropic refinement. The data were corrected for absorption ($\mu = 10·6$ cm^{-1}), and were used for an additional refinement which yielded no significant improvement of the R value. No extinction correction was applied. The final parameters with their estimated standard deviations from the inverted block-diagonal matrices are given in Table 1. A list of observed and calculated structure factors (for data without an absorption correction) is given in Table 2. A final electron density Fourier projection down the c axis is shown in Fig. 3.

One important aspect of the structure determination is the differentiation between carbon and nitrogen atoms in the ring system. The numbering used is given in Fig.4(a). The assignment was made by considering values of ΔB obtained from a least-squares calculation using carbon scattering factors and the same temperature factor for all the atoms in the structure. This was done for both two- and three-dimensional data and results from the latter are presented in Table 3. The identities of atoms 5, 6, 7, 9, 10 and 12 are the same regardless of which five-membered ring represents the azide group. A consideration of the relative values of $(\Delta B - \overline{\Delta B})$ for each atom in the three rings separately established the identification of atoms 1, 3 and 11 as nitrogen and 2, 4 and 8 as carbon. Also atom 3 must be a nitrogen atom because it accepts a hydrogen bond from the water molecule. The hydrogen atoms attached to atoms 2 and 8 (carbon atoms) were located from the difference Fourier synthesis. In addition the alternative assignment of atomic types (1, 3, 11 as carbon and 2, 4, 8 nitrogen) was used but this trial structure did not refine below $R = 12·9\%$ whereas the preferred structure refined to $R = 9·0\%$. In both these cases the refinement was anisotropic, hydrogen atoms were omitted and only the observed data were used.

Table 1. *Atomic parameters and their estimated standard deviations*

Positional parameters are expressed as fractions of cell edges. Anisotropic temperature factors are expressed as:

$$\exp\{-(h^2 b_{11} + k^2 b_{22} + l^2 b_{33} + 2hk b_{12} + 2hl b_{13} + 2kl b_{23})\}.$$

Isotropic temperature factors are of the form $\exp(-B \sin^2 \theta/\lambda^2)$. Standard errors, determined from the inverted block-diagonal matrices, are listed for the last decimal place given.

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
N(1)	0·0731	0·3048	0·1416	0·0060	0·0032	0·066	0·0001	−0·0011	0·0000
	4	3	16	3	2	4	2	10	7
C(2)	0·0036	0·2427	0·0407	0·0047	0·0044	0·065	−0·0002	−0·0011	0·0001
	4	4	20	3	2	4	2	10	9
N(3)	0·0332	0·1711	0·1057	0·0059	0·0031	0·060	−0·0008	−0·0003	−0·0014
	4	3	15	3	2	4	2	9	6
C(4)	0·1339	0·1618	0·2763	0·0050	0·0035	0·058	0·0001	0·0012	−0·0001
	5	3	16	4	2	4	2	10	7
C(5)	0·2102	0·2205	0·3817	0·0047	0·0032	0·054	−0·0002	0·0002	0·0002
	4	3	17	3	2	3	2	9	7
C(6)	0·1755	0·2979	0·3082	0·0063	0·0030	0·062	−0·0002	0·0009	0·0005
	5	3	16	4	2	5	2	10	7
N(7)	0·3049	0·1885	0·5370	0·0059	0·0035	0·063	0·0002	−0·0036	0·0006
	4	3	16	4	2	4	2	10	6
C(8)	0·2845	0·1115	0·5232	0·0066	0·0035	0·064	0·0006	−0·0004	0·0017
	5	4	19	4	2	4	2	12	9
N(9)	0·1853	0·0931	0·3717	0·0073	0·0028	0·065	0·0003	0·0012	0·0018
	4	3	15	4	2	3	2	11	6
N(10)	0·2187	0·3694	0·3572	0·0083	0·0027	0·096	−0·0008	−0·0021	−0·0005
	5	3	20	4	2	5	2	13	7
N(11)	0·1389	0·4190	0·2159	0·0101	0·0030	0·105	0·0000	−0·0001	0·0023
	6	3	20	5	2	6	2	15	9
N(12)	0·0526	0·3835	0·0846	0·0086	0·0033	0·100	0·0007	−0·0011	0·0012
	5	3	20	5	2	6	2	13	9
O(W)	0·4038	0·4520	0·6948	0·0083	0·0031	0·106	0·0007	−0·0066	−0·0011
	4	3	18	4	2	4	2	10	7

	x	y	z	B
H(13)	−0·069	0·260	−0·105	4·6 Å^2
	6	5	21	1·6
H(14)	0·335	0·070	0·594	4·6
	7	4	24	1·6
H(15)	0·152	0·038	0·385	5·8
	7	5	28	1·9
H(17)	0·462	0·409	0·792	5·4
	7	5	28	1·9
H(18)	0·348	0·419	0·616	4·7
	7	4	24	1·6

Table 2. *Values of observed and calculated structure factors*

The table lists $|F_o|$, $|F_c|$ and α. An asterisk indicates that the reflection was too weak for measurement. The value listed for such a reflection corresponds to an intensity of half the threshold intensity for the technique of measurement used.

K	OBS	CALC	ALPHA
(0,K,0)			
2	68.2	66.0	180
4	54.5	54.6	180
6	18.6	20.5	180
8	25.0	24.5	180
10	17.6	19.0	0
12	7.4	6.6	180
14	10.7	10.6	180
16	4.2	4.0	0
18	1.4*	0.5	180
20	2.1	0.8	0
22	1.0*	0.8	0
(1,K,0)			
1	6.0	5.0	0
2	8.8	8.7	180
3	28.1	23.4	0
4	29.7	28.2	0
5	15.5	14.2	0
6	2.4	1.3	0
7	9.0	7.7	0
8	11.5	9.5	0
9	30.4	31.9	180
10	9.6	8.0	0
11	11.5	9.9	180
12	9.8	9.3	180
13	14.4	13.8	0
14	16.2	16.9	180
15	16.3	17.2	180
16	10.3	10.4	0
17	4.4	3.7	0
18	3.9	2.2	0
19	4.8	4.1	0
20	4.0	3.0	180
21	1.4*	1.0	0
22	0.9*	1.2	180
(2,K,0)			
0	33.4	25.5	180
1	89.3	75.9	180
2	6.1	6.1	180
3	6.3	6.2	180
4	27.8	29.1	0
5	30.6	31.1	0
6	11.7	10.0	0
7	14.1	14.7	0
8	2.5	3.0	180
9	21.0	21.7	0
10	20.9	20.8	180
11	3.4	3.6	180
12	10.8	9.9	0
13	7.1	6.3	180
14	11.1	10.8	180
15	12.5	12.2	180
16	5.8	4.2	0
17	1.9	2.3	180
18	3.1	2.9	0
19	1.7*	0.2	0
20	1.4*	0.7	0
21	1.4*	0.7	0
(3,K,0)			
1	9.1	10.2	0
2	39.8	46.9	0
3	27.2	31.0	180
4	25.2	27.4	0
5	20.5	19.9	0
6	30.2	30.5	0
7	6.9	6.8	180
8	26.7	30.3	180
9	4.7	4.5	180
10	4.8	3.9	0
11	8.6	8.3	0
12	11.6	12.1	0
13	5.9	5.1	180
14	10.5	9.8	180
15	8.6	8.1	0
16	3.3	3.6	180
17	3.2	2.2	0
18	3.3	3.1	180
19	7.4	6.4	180
20	1.4*	0.7	180
21	1.0*	0.3	180
(4,K,0)			
0	3.9	4.3	0
1	8.3	8.9	180
2	34.6	39.5	180
3	14.9	15.2	0
4	3.3	2.3	180
5	34.7	38.0	0
6	1.0*	1.2	0
7	13.2	13.4	180
8	29.8	32.5	0
9	8.6	8.3	0
10	3.3	2.9	0
11	6.9	6.2	180
12	4.2	4.0	180
13	9.7	9.3	180
14	6.3	5.4	180
15	13.8	13.8	0
16	6.4	5.2	0
17	5.3	4.7	180
18	2.0*	1.8	0
19	4.9	4.4	180
20	4.1	3.6	180
21	2.7	2.8	180
(5,K,0)			
1	17.3	18.0	180
2	14.5	14.6	0
3	3.0	2.4	0
4	2.9	1.8	180
5	2.6	0.0	0
6	14.8	14.6	180
7	15.5	16.1	180
8	7.3	7.5	0
9	11.9	12.5	0
10	9.6	9.8	0
11	10.4	10.6	0
12	4.1	3.4	0
13	14.8	15.7	0
14	7.2	6.8	180
15	7.4	8.2	180
16	2.0*	0.9	180
17	4.4	3.2	180
18	1.7*	0.2	180
19	2.9	1.9	180
20	1.4*	1.1	180
(6,K,0)			
0	13.0	12.3	0
1	18.6	19.8	180
2	15.3	16.2	180
3	2.9	0.7	0
4	2.9	3.0	0
5	2.9	1.1	180
6	9.0	8.6	180
7	6.0	5.0	0
8	2.4	2.3	180
9	7.3	7.5	180
10	5.3	5.8	0
11	8.5	7.5	180
12	14.5	15.9	0
13	8.6	8.0	0
14	2.4*	0.5	0
15	2.0*	1.8	0
16	3.3	2.7	180
17	2.9	2.2	0
18	3.9	3.4	180
19	2.1	1.9	180
20	1.1*	0.6	180
(7,K,0)			
1	10.8	11.2	0
2	16.4	16.4	180
3	28.1	32.6	180
4	6.7	5.2	180
5	5.8	4.3	0
6	10.5	11.0	180
7	15.6	15.9	0
8	5.2	4.9	180
9	3.5	3.3	0
10	10.3	10.0	0
11	2.1	2.9	0
12	2.4	0.2	180
13	11.1	11.1	180
14	4.0	3.5	180
15	2.6	2.4	0
16	4.5	4.3	0
17	6.4	5.8	0
18	1.4*	0.3	180
19	1.0*	1.5	180
(8,K,0)			
0	18.2	19.0	180
1	12.2	11.8	180
2	9.0	7.9	0
3	14.2	14.3	180
4	7.9	6.4	0
5	2.0*	0.6	180
6	3.8	3.3	180
7	8.4	9.2	180
8	8.1	6.9	0
9	3.9	3.7	0
10	17.7	20.2	180
11	4.4	3.8	0
12	10.9	11.1	0
13	7.5	7.5	180
14	2.9	2.3	0
15	6.2	6.1	0
16	2.0*	1.6	180
17	4.0	3.5	0
18	1.0*	1.2	0
(9,K,0)			
1	1.7*	1.6	0
2	2.0*	1.2	180
3	1.8	2.4	180
4	11.6	11.0	0
5	13.6	15.0	0
6	6.8	6.3	180
7	11.5	12.4	180
8	14.2	15.5	180
9	1.7*	1.2	0
10	2.7	2.0	0
11	4.2	4.4	180
12	2.0*	1.3	0
13	2.0*	1.3	180
14	3.0	2.6	0
15	1.7*	1.3	0
16	3.5	3.4	0
17	1.0*	0.4	0
(10,K,0)			
0	10.8	11.5	0
1	3.9	3.8	180
2	5.8	5.6	180
3	2.4*	0.9	0
4	9.3	10.0	0
5	4.4	3.9	0
6	6.9	6.9	180
7	4.8	4.9	180
8	2.4*	0.6	0
9	9.9	9.0	180
10	1.7*	0.2	180
11	9.6	9.7	0
12	4.8	4.6	180
13	1.7*	0.3	180
14	2.8	2.0	180
15	1.4*	1.3	180
16	1.0*	1.6	0
(11,K,0)			
1	7.3	7.4	0
2	10.3	11.8	180
3	8.7	9.2	0
4	11.3	12.6	0
5	15.2	17.9	180
6	6.8	6.7	180
7	2.0*	0.5	180
8	6.2	6.0	0
9	6.5	6.7	0
10	1.4*	1.1	0
11	4.5	4.0	180
12	5.4	5.1	180
13	1.4*	0.9	180
14	1.4*	0.0	0
15	1.0*	0.9	180
(12,K,0)			
0	6.7	6.7	180
1	3.6	3.9	0
2	1.7*	0.9	0
3	2.6	1.7	0
4	9.2	10.0	0
5	2.5	1.6	180
6	4.2	4.2	180
7	4.9	5.1	0
8	2.9	2.2	180
9	2.1	0.6	0
10	1.7*	0.5	180
11	1.2*	0.0	0
12	1.0*	0.6	180
13	2.9	3.5	180
(13,K,0)			
1	3.2	3.4	180
2	1.4*	1.6	0
3	2.8	2.8	0
4	4.5	4.1	180
5	1.9	2.0	0
6	6.9	5.6	0
7	1.7*	0.2	180
8	1.7*	1.0	0
9	1.8	1.1	0
10	1.7	1.6	0
11	1.1*	2.3	180
(14,K,0)			
0	6.0	5.8	0
1	1.7*	0.1	180
2	3.8	4.5	180
3	4.2	4.6	0
4	1.2*	1.2	0
5	1.7	0.9	0
6	2.7	2.7	180
7	2.3	2.2	180
8	3.0	4.3	0
(15,K,0)			
1	2.0	1.8	180
2	1.1*	0.1	0
3	0.9*	0.1	0
(0,K,1)			
1	89.2	92.0	188
3	33.0	30.2	268
5	37.7	32.7	357
7	11.7	11.3	351
9	23.1	22.2	24
11	14.4	13.5	249
13	10.7	9.3	123
15	9.4	10.0	199
17	4.8	4.6	143
19	1.4*	2.0	93
21	1.8	1.8	42
(1,K,1)			
1	31.2	28.5	271
2	29.6	21.5	331
3	30.2	31.9	127
4	32.0	34.4	319
5	2.0	2.4	135
6	17.8	15.9	14
7	20.6	19.3	89
8	17.3	14.9	140
9	18.1	17.4	309
10	9.8	9.5	307
11	14.8	14.0	285
12	3.6	3.6	316
13	9.0	8.7	149
14	15.7	14.6	160
15	15.8	14.7	20
16	4.6	4.7	335
17	2.0*	1.8	196
18	3.1	3.2	104
19	6.0	5.7	192
20	2.4	2.1	191
21	1.0*	0.9	228
(2,K,1)			
0	104.3	120.1	166
1	84.4	91.1	86
2	3.8	3.3	320
3	14.5	15.3	0
4	19.4	19.0	333
5	20.0	18.5	317
6	12.3	11.2	49
7	17.8	16.9	235
8	29.6	26.6	335
9	5.8	5.9	298
10	7.0	5.8	136
11	11.5	10.6	75
12	3.1	3.1	289
13	10.0	9.6	147
14	6.5	6.8	269
15	10.8	9.9	311
16	6.0	6.3	72
17	2.0*	1.9	129
18	2.0*	1.1	27
19	2.3	2.7	144
20	2.1	1.8	177
(3,K,1)			
1	14.0	15.7	159
2	7.8	6.6	254
3	4.5	4.0	148
4	10.2	9.9	104
5	8.6	8.2	95
6	12.0	12.1	139
7	18.7	19.6	211
8	2.3	2.3	250
9	20.9	19.5	345
10	7.9	7.3	294
11	13.5	11.8	12
12	10.6	8.4	289
13	2.5	3.2	49
14	18.7	16.4	70
15	8.7	7.9	283
16	4.7	5.3	216
17	3.6	3.7	153
18	3.0	3.2	223
19	3.1	3.0	129
20	2.3	1.4	325
(4,K,1)			
0	35.2	37.7	73
1	5.3	5.2	89
2	9.1	8.7	218
3	10.3	9.8	283
4	10.7	10.1	230
5	11.3	11.9	130
6	12.5	12.1	194
7	5.9	5.7	91
8	6.3	5.9	33
9	17.1	15.7	159
10	9.4	9.5	8
11	11.1	10.7	252
12	4.7	4.7	294
13	8.1	8.6	39
14	7.9	7.6	8
15	6.9	7.5	337
16	4.9	4.9	216
17	2.0*	1.7	346
18	3.8	3.3	155
19	1.7*	1.4	188
20	1.4*	1.1	201
(5,K,1)			
1	5.0	3.4	26
2	29.9	30.8	119
3	12.8	10.4	171
4	20.9	21.1	232
5	6.0	5.3	140
6	20.0	20.1	165
7	13.7	13.6	14
8	9.1	8.8	38
9	14.5	13.7	283
10	4.6	4.9	346
11	3.6	3.6	142
12	7.4	7.1	103
13	7.3	8.1	120
14	9.2	9.3	261
15	3.4	3.4	267
16	4.6	5.1	331
17	2.0*	2.0	336
18	4.4	4.3	67
19	1.7	2.0	294
(6,K,1)			
0	19.0	22.3	137
1	9.4	10.4	206
2	15.8	18.8	336
3	10.2	10.4	200
4	11.5	11.4	294
5	14.4	15.1	150
6	14.4	13.6	98
7	7.3	6.3	239
8	8.8	8.9	208
9	15.3	14.1	82
10	11.9	11.2	158
11	9.2	9.1	297
12	8.7	7.9	9
13	2.4*	2.1	166
14	9.6	9.9	278
15	2.0*	1.5	238
16	5.7	5.6	112
17	3.0	3.6	341
18	2.4	2.3	90
19	2.1	2.2	43
(7,K,1)			
1	8.3	9.1	291
2	8.6	9.4	201
3	3.0	2.5	346
4	9.1	9.6	340
5	10.3	11.0	46
6	13.5	12.4	77
7	5.0	4.4	137
8	15.8	14.0	204
9	5.3	5.5	246
10	3.8	3.9	126
11	7.1	6.7	176
12	3.0	3.3	247
13	7.7	7.0	151
14	7.2	5.5	21
15	5.4*	4.1	343
16	2.5	2.7	334
17	1.7*	0.6	42
18	1.4*	1.0	258
(8,K,1)			
0	4.8	4.3	46
1	9.6	11.4	64
2	7.2	6.4	290
3	5.7	5.6	260
4	6.0	5.3	352
5	8.2	8.2	72
6	2.4*	2.8	155
7	3.3	3.1	239
8	5.0	4.9	54
9	7.0	6.9	204
10	7.5	5.2	233
11	10.1	9.8	353
12	10.3	10.0	167
13	4.8	4.5	135
14	6.1	3.1	77
15	1.7*	1.3	189
16	1.9	2.1	335
17	1.4*	1.1	95
(9,K,1)			
1	6.9	5.7	62
2	9.0	9.4	95
3	20.4	18.8	318
4	11.7	11.5	18
5	12.9	14.0	148
6	9.2	10.9	242
7	8.1	7.9	217
8	7.5	7.3	330
9	4.7	4.4	13
10	4.3	4.5	108
11	7.2	7.2	118
12	3.9	4.6	127
13	2.9	2.9	322
14	1.7*	2.0	282
15	1.7*	1.7	242
16	1.4*	1.9	235
(10,K,1)			
0	3.3	2.4	161
1	8.7	9.5	287
2	2.3	2.0	90
3	7.9	9.5	314
4	7.7	6.6	330
5	6.2	5.7	106
6	4.7	4.1	223
7	7.6	7.1	346
8	3.8	4.2	161
9	5.0	4.8	96
10	7.7	7.1	9
11	6.3	6.5	249
12	5.7	5.9	133
13	1.7*	1.1	69
14	2.8	3.0	253
15	2.6	2.7	146
(11,K,1)			
1	4.3	3.8	185
2	2.9	2.7	304
3	4.6	4.7	1
4	10.6	8.9	209
5	5.8	5.2	184
6	8.8	8.4	25
7	5.8	5.7	26
8	5.7	5.9	340
9	3.4	2.9	274
10	2.0	2.0	127
11	1.4*	1.0	134
12	1.7*	1.3	237
13	1.4*	0.0	90
14	1.0*	1.1	220
(12,K,1)			
0	1.4*	0.7	45
1	7.6	7.0	70
2	3.5	3.8	245
3	6.8	6.1	305
4	4.5	3.9	134
6	5.3	4.6	71
7	3.8	3.9	274
8	3.6	3.0	316
9	3.6	3.7	12
10	3.5	3.5	265
11	3.5	3.2	150
12	0.6*	0.9	56
(13,K,1)			
1	6.7	4.5	132
2	5.0	4.3	340
3	6.3	5.1	233
4	7.1	5.8	114
5	6.4	5.7	24
6	4.1	4.3	279
7	1.9	2.4	272
8	2.1	2.5	272
9	2.4	2.3	77
(14,K,1)			
0	1.8	1.5	4
1	2.9	2.6	182
2	1.0*	1.2	161
3	2.9	2.5	282
4	2.7	2.4	135
5	4.2	3.8	107
6	2.1	2.1	273
(0,K,2)			
0	13.0	15.3	103
2	16.4	16.3	183
4	3.4*	0.8	51
6	11.2	10.6	101
8	21.3	21.1	319
10	7.3	6.6	341
12	2.6	2.5	102
14	12.9	14.2	230
16	9.8	9.1	55
18	1.0*	2.0	37
(1,K,2)			
1	10.0	9.8	209
2	21.8	22.7	166
3	7.0	6.6	89
4	12.9	13.1	95
5	8.9	9.2	66
6	14.4	13.7	137
7	9.8	9.1	143
8	15.0	15.7	311
9	14.4	13.4	291
10	2.4	2.2	270
11	6.4	6.4	303
12	5.6	6.1	187
13	5.2	5.4	35
14	10.7	10.9	51
15	3.0	3.4	298
16	2.0*	2.6	245
17	3.0	3.4	126
18	4.1	4.2	262
19	3.2	2.9	174
(2,K,2)			
0	44.9	47.1	89
1	18.1	20.2	17
2	15.9	17.2	243
3	6.8	8.0	105
4	8.8	9.1	308
5	13.1	13.2	140
6	4.4	4.1	184
7	2.4	2.2	357
8	5.4	6.7	16
9	11.5	12.2	171
10	6.5	6.2	156
11	1.7*	0.9	132
12	5.0	4.9	316
13	4.8	4.7	319
14	2.4*	2.5	339
15	4.0	4.5	24
16	2.0*	0.7	287
17	3.9	4.0	316
18	1.7*	1.7	152
19	1.4*	1.6	240
(3,K,2)			
1	10.5	10.2	69
2	21.7	19.9	68
3	14.2	11.9	215
4	11.4	12.1	174
5	10.3	8.3	10
6	12.8	12.4	184
7	9.4	9.2	304
8	2.6	3.1	133
9	6.4	6.6	207
10	5.6	6.0	47
11	7.3	7.8	84
12	6.2	6.2	43
13	7.5	7.5	101
14	10.9	11.3	268
15	10.4	10.2	241
16	2.0*	2.1	128
17	4.2	4.3	0
18	1.4*	1.5	53
19	1.0*	0.4	346
(4,K,2)			
0	28.8	31.8	358
1	44.0	45.6	242
2	8.9	8.6	243
3	8.4	8.2	181
4	1.4*	1.4	192
5	10.4	11.2	78
6	7.9	8.1	126
7	4.0	4.5	134
8	6.0*	3.8	178
9	11.5	12.5	65
10	3.8	3.5	190
11	10.4	11.4	245
12	7.4	7.7	56
13	2.4*	1.1	265
14	6.3	6.1	241
15	4.3	4.3	8
16	1.7*	0.7	107
17	1.7*	1.2	240
18	1.3	1.8	56
(5,K,2)			
1	4.9	4.7	285
2	5.2	4.9	256
3	10.9	10.6	295
4	6.9	7.2	327
5	7.4	7.0	51
6	8.6	8.8	69
7	8.4	8.7	93
8	9.3	9.3	223
9	4.1	4.9	218
10	6.7	7.1	89
11	7.8	8.2	170
12	2.4*	2.4	78
13	3.3	3.5	327
14	7.8	7.9	250
15	4.6	4.8	108
16	3.8	3.9	19
17	1.4*	0.8	90
18	1.0*	0.9	312
(6,K,2)			
0	10.9	11.7	290
1	4.0	4.0	78
2	2.0*	3.6	230
3	1.7*	1.0	174
4	7.5	7.8	12
5	5.4	5.9	68
6	1.7*	0.9	154
7	8.4	9.2	278
8	6.8	7.1	88
9	3.0	2.8	230
10	8.8	9.3	231
11	8.3	9.3	34
12	4.5	4.7	94
13	4.6	5.0	235
14	3.2	3.4	159
15	6.9	6.4	139
16	3.3	3.0	24
17	1.7	1.6	47
(7,K,2)			
1	6.2	6.8	5
2	1.7*	3.1	209
3	8.7	9.0	272
4	15.1	16.5	36
5	10.3	10.3	118
6	10.5	10.8	249
7	11.9	11.2	238
8	7.1	7.1	280
9	8.4	8.8	55
10	5.0	5.7	58
11	3.3	3.8	68
12	2.7	3.1	180
13	4.1	4.1	223
14	2.5	2.2	98
15	1.7*	0.5	282
16	1.4*	1.7	144
(8,K,2)			
0	6.3*	6.6	170
1	11.5	10.3	262
2	3.8	3.4	168
3	3.2	3.7	279
4	10.0	9.5	20
5	5.4	5.6	20
6	11.2	10.9	242
7	5.3	5.3	7
8	4.5	4.6	33
9	4.2	4.2	256
10	2.7*	0.7	98
11	3.3	3.3	180
12	3.2	3.3	113
13	2.1	2.4	147
14	1.7*	1.5	66
15	3.0	3.0	30
(9,K,2)			
1	7.3	5.7	132
2	8.7	7.5	321
3	1.7*	1.7	14
4	7.1	6.9	168
5	6.8	6.9	259
6	6.7	5.6	300
7	2.9	3.0	62
8	3.0	3.4	26
10	1.4*	1.2	90
11	1.4*	1.2	190
12	2.0	2.1	343
13	3.2	3.3	318
14	2.6	3.1	218
(10,K,2)			
0	8.9	7.9	328
1	1.7*	1.7	114
2	6.7	6.5	173
3	7.4	7.1	335
4	4.5	4.1	86
5	4.0	3.3	211
6	2.0*	1.9	102
7	3.5	3.2	208
8	2.0*	2.0	279
9	3.9	3.7	40
10	4.1	3.8	292
11	3.1	3.1	123
12	3.5	3.2	7
13	3.8	4.1	291
(11,K,2)			
1	1.4*	0.2	120
2	7.4	5.5	236
3	4.1	4.1	159
4	3.9	3.1	97
5	2.0*	1.6	79
6	1.7*	1.0	73
7	2.8	3.0	312
8	1.8*	2.7	283
9	3.2	2.8	32
10	2.7	2.7	105
11	3.8	3.3	260
(12,K,2)			
0	3.1	2.6	115
1	3.0	2.0	123
2	2.5	2.3	247
3	3.6	3.2	152
4	5.3	4.4	67
5	2.8	2.6	110
6	2.9	2.7	246
7	1.4*	1.0	314
8	2.0	2.2	40
9	4.4	4.2	246
(13,K,2)			
1	1.4*	1.3	27
2	1.0*	1.6	227
3	2.5	2.4	132
4	5.4	5.1	62
5	3.0	3.5	297
(0,K,3)			
1	9.0	9.0	156
3	1.7*	0.4	14
5	8.6	8.8	112
7	7.2	6.6	323
9	8.6	8.6	117
11	2.9	3.2	229
13	4.5	4.3	263
15	4.1	3.8	51
17	1.9	2.8	320
(1,K,3)			
1	3.8	3.8	295
2	8.2	7.7	111
3	6.4	6.1	132
4	2.4*	1.7	107
5	2.4*	1.9	302
6	9.0	8.7	140
7	9.4	8.8	334
8	3.1	3.3	293
9	4.6	4.4	183
10	3.8	3.6	352
11	2.6	2.7	109
12	2.0*	1.4	45
13	9.1	8.7	121
14	2.4	2.5	187
15	3.8	3.9	291
16	1.4*	1.2	324
(2,K,3)			
0	6.3	6.7	130
1	12.2	10.8	205
2	7.8	7.5	306
3	2.4*	1.2	25
4	4.4	4.0	297
5	6.5	5.7	47
6	1.7*	1.3	38
7	4.5	4.8	209
8	4.6	4.5	136
9	4.3	3.7	66
10	7.0	6.8	146
11	2.0	2.0	282
12	2.6	2.4	0
13	4.3	4.5	138
14	1.7*	2.6	7
15	6.8	6.6	323
16	1.4*	1.5	212
(3,K,3)			
1	1.7*	3.5	47
2	10.2	9.8	299
3	10.0	9.6	267
4	5.6	6.1	334
5	2.4*	1.0	6
6	3.5	3.4	26
7	3.1	3.4	148
8	10.5	10.3	174
9	4.8	4.3	49
10	5.3	5.6	62
11	4.4	4.6	126
12	3.5	3.5	323
13	3.4	3.5	263
14	5.4	5.2	236
15	2.8	3.2	103
16	3.4	3.3	42
(4,K,3)			
0	24.7	23.9	271
1	10.2	10.2	174
2	8.3	7.5	75
3	5.3	5.5	297
4	3.4	3.3	63
5	3.9	3.8	55
6	5.6	5.3	202
7	5.8	5.4	276
8	5.7	6.1	77
9	2.4*	1.6	116
10	7.4	7.2	277
11	4.4	4.6	355
12	4.8	4.6	105
13	3.0	2.6	205
14	1.7*	1.8	60
15	2.4	2.5	143
(5,K,3)			
1	5.3	5.4	302
2	9.3	8.6	180
3	2.4*	2.1	346
4	10.0	9.7	4
5	11.1	10.7	129
6	5.2	4.8	214
7	6.0	5.9	235
8	7.9	8.1	307
9	7.4	7.0	16
10	2.0*	1.1	50
11	1.4*	0.3	0
12	3.3	3.4	154
13	4.7	4.4	221
14	4.4	4.4	117
15	4.1	4.5	45
(6,K,3)			
0	22.6	22.9	154
1	6.5	6.2	74
2	5.6	5.2	357
3	5.2	4.9	284
4	7.0	6.4	341
5	2.4*	1.8	150
6	6.7	6.3	252
7	6.0	5.4	328
8	2.9	2.8	47
9	4.5	4.5	233
10	4.2	4.1	104
11	3.0	2.9	41
12	3.5	3.4	253
13	3.6	3.6	154
14	1.4*	1.7	7
(7,K,3)			
1	3.4	3.9	157
2	3.4	3.3	289
3	7.1	6.5	62
4	4.4	4.5	143
5	7.5	7.1	250
6	6.2	5.5	283
7	1.4*	1.3	351
8	3.9	3.7	76
9	2.9	3.1	25
10	1.4*	0.4	270
11	2.4	2.5	241
12	4.1	3.9	263
13	1.8	2.1	128
(8,K,3)			
0	1.7*	4.0	327
1	3.6	4.0	58
2	3.5	3.3	164
3	7.1	6.7	322
4	4.8	4.3	82
5	4.2	4.2	210
6	2.0*	1.6	146
7	3.1	3.1	115
8	4.2	4.1	276
9	1.4*	2.0	127
10	2.8	3.0	336
11	1.0*	1.3	266
12	1.7	2.0	186
(9,K,3)			
1	1.4*	2.1	115
2	1.4*	1.5	138
3	1.4*	1.1	350
4	2.0*	1.1	230
5	1.7*	2.0	177
6	3.3	2.6	120
7	1.7*	1.0	37
8	2.4	3.0	328
9	2.5	2.6	327
10	3.2	3.3	151
(10,K,3)			
0	1.4*	2.1	98
1	2.8	2.8	133
2	1.4*	2.4	340
3	1.7*	1.1	333
4	1.7*	1.5	195
5	6.8	5.9	135
6	1.7*	0.6	170
7	2.5	2.8	313
8	2.5	2.5	111
(11,K,3)			
1	2.6	1.8	280
2	2.7	2.3	157
3	2.6	2.6	65
4	1.4*	1.0	73
5	1.0*	1.2	180
(0,K,4)			
0	11.2	9.9	116
2	7.6	7.2	288
4	4.1	2.4	282
6	2.5	2.6	88
8	1.8	1.3	63
10	3.4	3.2	150
12	0.6*	1.5	12
(1,K,4)			
1	1.4*	1.1	220
2	4.4	3.7	235
3	1.4*	1.6	266
4	3.8	3.2	349
5	2.0*	1.6	51
6	3.4	3.2	358
7	1.7*	0.5	24
8	4.9	4.3	200
9	1.7*	1.7	294
10	2.8	2.8	88
11	2.4	2.3	134
(2,K,4)			
0	1.4*	2.1	242
1	6.1	5.0	339
2	1.4*	0.5	282
3	2.0*	0.5	294
4	4.9	3.4	28
6	5.6	5.7	204
7	2.5	2.0	243
8	4.9	4.7	40
9	3.2	3.0	210
10	3.1	3.1	241
11	3.4	3.6	8
(3,K,4)			
1	1.4*	2.6	25
2	2.0*	1.2	275
3	6.2	3.6	268
4	2.0*	2.6	11
5	5.7	5.2	109
6	4.6	4.4	220
7	6.9	6.4	206
8	3.4	2.8	328
9	5.7	5.8	14
10	1.0*	1.2	120
11	0.6*	1.0	6
(4,K,4)			
0	6.3	5.5	132
1	1.4*	2.3	211
2	1.4*	1.1	355
3	3.3	2.9	234
4	1.4*	1.8	16
5	2.5	2.0	171
6	4.4	4.0	222
7	5.0	4.6	6
8	1.4*	2.1	28
9	1.4*	1.1	265
10	1.0*	1.7	343
(5,K,4)			
1	4.0	4.5	195
2	1.7*	1.2	144
3	8.3	7.0	41
4	4.5	4.4	150
5	3.2	3.1	218
6	3.2	3.0	287
7	2.0	3.4	8
8	3.3	3.6	8
9	1.0*	1.9	235
(6,K,4)			
0	6.7	6.0	64
1	9.1	9.1	8
2	5.5	5.1	227
3	1.0*	1.6	334
4	4.2	3.8	58
5	3.6	3.2	189
6	1.0*	1.6	14
7	1.4*	1.6	94
8	1.0*	2.7	251
(7,K,4)			
1	3.4	3.4	73
2	2.6	2.7	13
3	3.0	2.8	245
4	1.4*	1.1	130
5	1.4*	0.8	292
6	1.0*	1.0	134
(8,K,4)			
0	5.6	5.4	353
1	4.6	4.7	227
2	0.6*	1.4	215
3	1.0*	1.7	73

Table 3. *Assignment of atom type in the ring system*

Atom number (see Fig. 4)	Input	$\overline{AB} - \overline{AB}$	Suggested new input
1	C	−0·50	N
2	C	+0·37	C
3	C	−0·61	N
4	C	−0·04	C
5	C	−0·14	C*
6	C	+0·16	C*
7	C	−0·41	N*
8	C	+0·50	C
9	C	−0·38	N*
10	C	−0·18	N*
11	C	−0·48	N
12	C	−0·45	N*

Regardless of which five-membered ring represents the azide group the atomic type of those atoms marked with * is constant.

Description and discussion of the structure

This investigation has shown that when 6-hydrazino-purine reacts with nitrous acid the resulting azide group is not linear but is in a cyclic form resulting in the formation of a tetrazolopurine. This had been guessed but not proved from ultraviolet and infrared spectra (Johnson, Thomas & Schaeffer, 1958). Since the compound is of interest as a purine derivative, the numbering of atoms has been chosen for this paper to conform to the numbering for adenine. This numbering and the numbering used in *The Ring Index* (1963), which designates the compound as 7*H*-tetrazolo[5,1-*i*]-purine, are shown in Fig. 4.
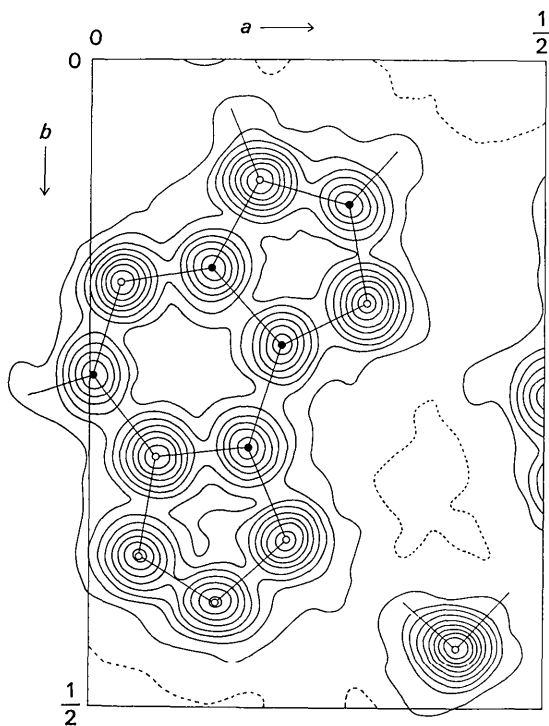
Fig. 3. Electron density projection on (001). Contour interval 1 e.Å⁻². Zero contour dotted. Open circles: nitrogen, oxygen. Closed circles: carbon.

The intramolecular bond lengths and angles are shown in Fig. 5. The estimated standard deviations are 0·009 Å for all bonds not including hydrogen, 0·09 Å for distances involving one hydrogen and 0·13 Å for distances between hydrogen atoms. For angles the estimated standard deviations are 0·6° unless a hydrogen atom is involved, in which case they range from 5 to 8°.

The bond lengths in purine and its derivatives have been reported (see for example, Hoogsteen, 1963; Kraut & Jensen, 1963; Watson, Sweet & Marsh, 1965; Watson, Sutor & Tollin, 1965; Sundaralingam, 1966). In the tetrazolopurine the distances N(1)–C(2), N(3)–C(4) and N(7)–C(8) appear slightly longer and C(8)–N(9) slightly shorter than expected from the literature.

At first the equivalence of the bonds C(8)–N(7) and C(8)–N(9) (1·33 Å) was surprising. However in purine (Watson, Sweet & Marsh, 1965) and imidazole (Martinez-Carrera, 1966) the differences in these N–C bond lengths are small, of the order of 0·02 Å. Also in guanine hydrochloride dihydrate (Iball & Wilson, 1965) these distances are 1·322 and 1·335 Å but each nitrogen atom is attached to a hydrogen atom and both form short hydrogen bonds (2·656 and 2·702 Å) to water molecules. In crystals of the tetrazolopurine the hydrogen atom H(15) attached to N(9) is not very well defined in the difference map. However the hydrogen atoms [H(17) and H(18)] attached to the water molecule are clearly seen in this map and neither of these hydrogen atoms is involved in the short (2·70 Å) hydrogen bond from the water molecule to N(9) (see Fig. 6). This would suggest that the short hydrogen bond must involve H(15). The environment of N(7) (with C(2) of two other molecules at 3·21 Å and 3·23 Å) indicates that N(7) probably does not have an attached
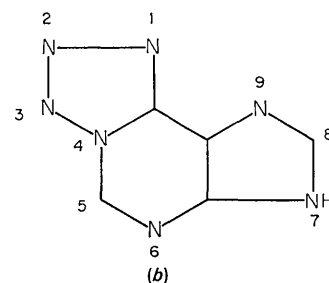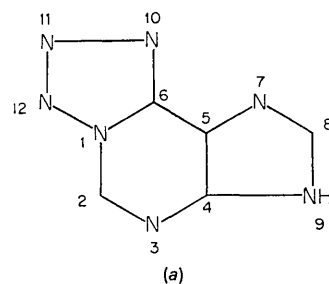
Fig. 4. (a) Atomic numbering used in this paper. (b) Atomic numbering in *The Ring Index* (1963).

hydrogen atom because if it did it would be expected to form a hydrogen bond to a nitrogen or oxygen atom.

From a consideration of the bond lengths and the packing of the molecule it would seem that there exists a large amount of resonance in the ring system with the result that N(9) has an excess positive charge and N(7) and N(10) a slight negative charge. The bond N(11)–N(12) is shorter than the value measured for the anions in the hydrazine salt of 5-aminotetrazole (Bryden, 1958) or in sodium tetrazolate (Palenik, 1963). Presumably resonance forms with a charge on N(11) or N(12) make less of a contribution to the structure of the tetrazolopurine than do analogous forms in these anions.

The molecule is planar within experimental error. The deviations from this plane are given in Table 4 and none is considered significant.

Table 4. Deviations of atoms from the least-squares plane through the molecule

The planes through the molecule have the following equations where X, Y, Z are in Å.

Least-squares plane:
$$-0.45889X + 0.04830Y + 0.88718Z = 0.32345 \text{ Å}$$

Intermediate-squares plane:
$$0.83750X + 0.35693Y + 0.41376Z = 3.42056 \text{ Å}$$

Most-squares plane:
$$0.29667X - 0.93288Y + 0.20424Z = -3.17788 \text{ Å}$$

|	
N(1)	0.008 Å
C(2)	−0.007
N(3)	−0.007
C(4)	0.015
C(5)	0.006
C(6)	0.010
N(7)	−0.009
C(8)	−0.009
N(9)	0.002
N(10)	0.001
N(11)	−0.003
N(12)	−0.008
H(13)	−0.09
H(14)	−0.09
H(15)	0.18

The magnitudes of the principal axes of the thermal ellipsoids are listed in Table 5. The estimated standard deviation of a temperature factor for nitrogen, carbon and oxygen is approximately 0.24 Å². Those atoms having temperature factors which are not significantly anisotropic (all values less than 2σ from the mean value) are marked with an asterisk. (Anisotropic temperature factor parameters for all atoms are included in Table 1 because these values were used in the preparation of Table 2.) The direction cosines of the principal axes with respect to the three orthogonal axes perpendicular to the least, intermediate and most squares planes are listed. The values show that the motions of the atoms N(10), N(11) and N(12) are principally in planes perpendicular to the long axis of the molecule, showing that the azide group can easily bend out of the molecular plane. The motions of the other atoms are prin-

cipally in the plane of the molecule. N(1) and C(6) are probably isotropic and C(2) and N(3) move principally in the direction of the long axis of the molecule.

Table 5. Principal axes of thermal ellipsoids

B_i are the magnitudes along the principal axes of the thermal ellipsoids and are given in Å². Atoms marked with * are probably isotropic (less than 2σ from the mean B_i). Direction cosines (l_1, l_2, l_3) are listed with respect to the three axes perpendicular to the least-, intermediate- and most-squares planes through the molecule (see Table 4).

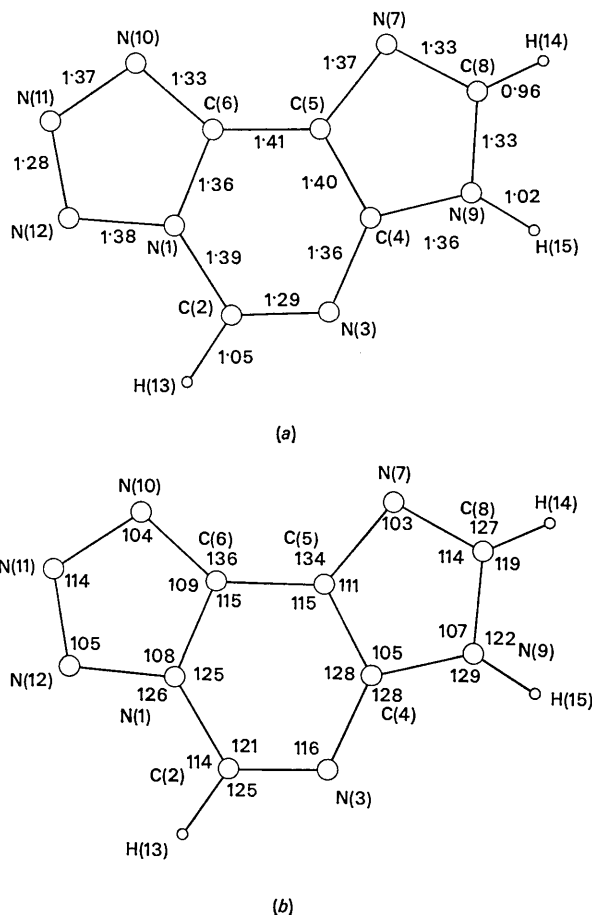|	B_i	l_1	l_2	l_3
N(1)*	3.9	0.983	0.078	0.166
	3.6	−0.111	−0.464	0.879
	3.2	0.146	−0.882	−0.448
C(2)	5.1	−0.107	−0.299	0.948
	3.7	0.946	0.263	0.190
	2.6	−0.306	0.917	0.254
N(3)	4.2	0.053	0.283	0.958
	3.4	0.999	−0.010	−0.053
	2.7	−0.005	0.959	−0.283
C(4)	4.0	−0.001	0.421	−0.907
	3.4	0.692	0.655	0.304
	2.7	0.722	−0.627	−0.292

(a)

(b)

Fig. 5. (a) Intramolecular bond lengths (Å). (b) Intramolecular angles (°).

Table 5 (cont.)

	B_t	l_1	l_2	l_3
C(5)	3·7	−0·165	−0·240	0·957
	3·1	0·844	0·468	0·263
	2·5	−0·511	0·850	0·126
C(6)*	3·7	0·420	0·838	0·349
	3·6	−0·612	−0·023	0·791
	3·2	0·671	−0·545	0·503
N(7)	4·1	−0·637	−0·280	0·718
	4·0	0·751	−0·435	0·497
	2·7	0·173	0·856	0·487
C(8)	4·5	−0·179	−0·817	0·548
	3·7	0·966	−0·252	−0·060
	3·1	0·187	0·519	0·834
N(9)	4·3	−0·167	−0·986	0·027
	3·7	0·903	−0·164	−0·398
	3·0	0·396	−0·043	0·917
N(10)	5·6	0·993	0·107	0·059
	4·7	−0·119	0·737	0·665
	2·8	0·028	−0·667	0·744
N(11)	6·2	0·885	0·465	−0·006
	5·5	−0·445	0·851	0·280
	3·3	0·136	−0·245	0·960
N(12)	5·8	0·934	0·351	0·063
	5·0	−0·342	0·932	−0·120
	3·5	−0·100	0·090	0·991
O(W)	6·8	0·976	−0·116	0·184
	4·1	0·144	0·978	−0·150
	3·3	−0·163	0·173	0·970

There are three hydrogen bonds per molecule each involving the water molecule and using all hydrogen atoms available for such bonding. These bonds are shown in Figs.6 and 7. The water molecule donates hydrogen atoms to N(3) (distance 3·02 Å) and to N(10) (distance 2·88 Å) and accepts one from N(9) (distance 2·70 Å). The last is a short hydrogen bond. There is also a close C–H---N contact involving C(2) and N(7) of another molecule, with an H---N distance of 2·19 Å. Distances and angles involved in the environments of the hydrogen atoms are listed in Table 6. The oxygen of the water molecule lies 0·66 Å from the plane through N(3), N(9) and N(10). It also packs against other water molecules along the screw axis parallel to c at a distance of 3·37 Å.

Molecules related by a c translation pack in parallel planes approximately 3·35 Å apart. Molecules related by the a-glide operation are approximately parallel as the molecular plane is almost perpendicular to the (010) plane and N(7) of one molecule makes close contacts (3·21 Å and 3·23 Å) with C(2) of two other molecules which are separated from each other by a c translation. As seen in Table 6 one of these close contacts involves an almost linear C–H---N contact and the two molecules involved are almost in the same plane.

Computations

All computations except the first $|F|^2$ projection map (which was calculated on the LGP 30 computer at The Johns Hopkins University) were performed on an IBM 1620 computer installed in this laboratory. In an earlier contribution from this laboratory (Johnson, 1965) a listing of programs is given. In addition a program (ICR no.7) was used to solve the sums from
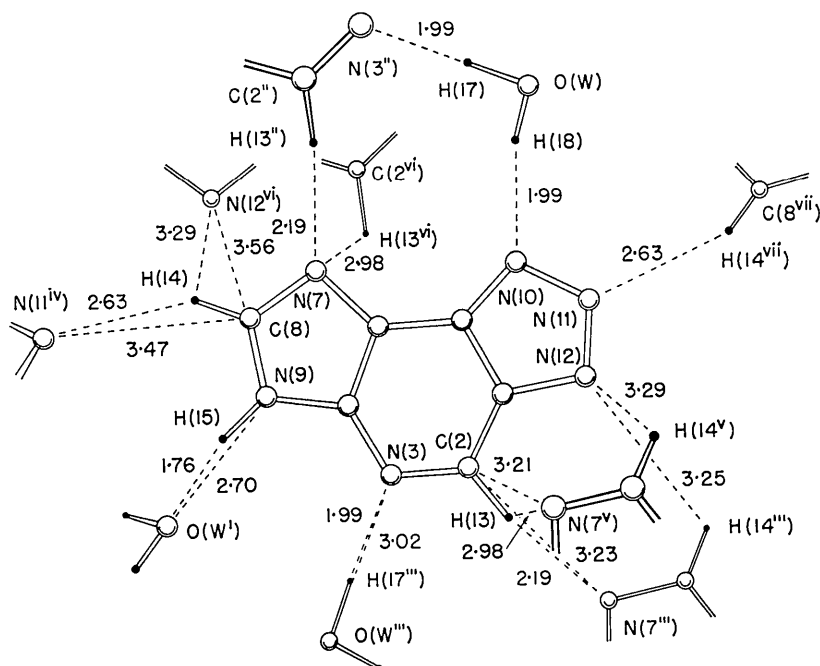

Fig. 6. Lateral packing and hydrogen bonding of the molecule. This diagram is drawn with respect to the plane of the molecule.

Table 6. *Environments of hydrogen atoms*

Atoms			Distances (Å)			Angles (°)	
X	H	Y	X–Y	X–H	H···Y	X–H···Y	H–X···Y
N(9)—H(15)···O(W')			2·70	1·02	1·76	153	17
O(W)–H(17)···N(3")			3·02	1·06	1·99	161	13
O(W)–H(18)···N(10)			2·88	0·92	1·99	165	10
C(2)—H(13)···N(7''')			3·23	1·05	2·19	168	8
C(8)—H(14)···N(11iv)			3·48	0·96	2·63	147	24
C(2)—H(13)···N(7v)			3·21	1·05	2·98	93	68

The following superscripts are used for this Table and for Fig. 6.

$$' \quad \tfrac{1}{2}-x, \ y-\tfrac{1}{2}, \ z-\tfrac{1}{2}$$
$$'' \quad \tfrac{1}{2}+x, \ \tfrac{1}{2}-y, \ z+1$$
$$''' \quad x-\tfrac{1}{2}, \ \tfrac{1}{2}-y, \ z-1$$
$$\text{iv} \quad \tfrac{1}{2}-x, \ y-\tfrac{1}{2}, \ z+\tfrac{1}{2}$$
$$\text{v} \quad x-\tfrac{1}{2}, \ \tfrac{1}{2}-y, \ z$$
$$\text{vi} \quad x+\tfrac{1}{2}, \ \tfrac{1}{2}-y, \ z$$
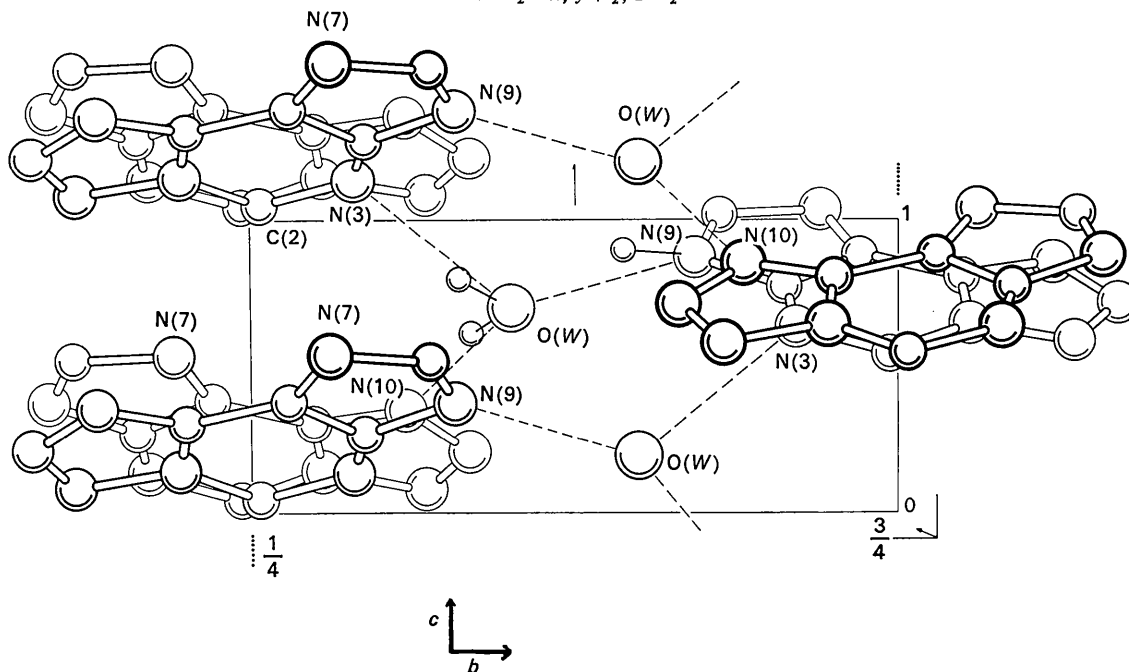$$\text{vii} \quad \tfrac{1}{2}-x, \ y+\tfrac{1}{2}, \ z-\tfrac{1}{2}$$

Fig. 7. Projection of the structure on (100) showing the packing around water molecules.

ICR no. 4 and to determine the standard deviations. The atomic scattering factors for carbon, nitrogen and oxygen were taken from *International Tables for X-ray Crystallography* (1962). Those for hydrogen were the values given by Stewart, Davidson & Simpson (1965).

References

BENDICH, A., GINER-SOROLLA, A. & FOX, J. J. (1957). In *Ciba Foundation Symposium on the Chemistry and Biology of Purines*. WOLSTENHOLME, G. E. W. & O'CONNOR, C. M., editors, p. 3. London: J. and A. Churchill.
BRYDEN, J. H. (1958). *Acta Cryst.* 11, 31.
HOOGSTEEN, K. (1963). *Acta Cryst.* 16, 907.

IBALL, J. & WILSON, H. R. (1965). *Proc. Roy. Soc.* A 288, 418.
International Tables for X-ray Crystallography (1962). Vol. III, p. 202. Birmingham: Kynoch Press.
JOHNSON, C. K. (1965). *Acta Cryst.* 18, 1004.
JOHNSON, J. A., JR., THOMAS, H. J. & SCHAEFFER, H. J. (1958). *J. Amer. Chem. Soc.* 80, 699.
KRAUT, J. & JENSEN, L. H. (1963). *Acta Cryst.* 16, 79.
MARTINEZ-CARRERA, S. (1966). *Acta Cryst.* 20, 783.
PALENIK, G. J. (1963). *Acta Cryst.* 16, 596.
The Ring Index, Supplement I (1957–1959) (1963). p. 77. (no. 8279). American Chemical Society.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175.
SUNDARALINGAM, M. (1966). *Acta Cryst.* 21, 495.
WATSON, D. G., SUTOR, D. J. & TOLLIN, P. (1965). *Acta Cryst.* 19, 111.
WATSON, D. G., SWEET, R. M. & MARSH, R. E. (1965). *Acta Cryst.* 19, 573.