From these data it has proved possible to determine its crystal structure.

The table displays the spacings, d_0, and intensities, I_0, given by Shore and Parry in comparison with those calculated from the proposed structure, d_c and I_c. The indices *hkl* relate to a tetragonal cell with $a = 5.255$ Å. and $c = 5.048$ Å., derived from a least squares fitting of the cell to the data after indices had been derived by trial-and-error. The indices indicate body-centering. With two molecules per cell the calculated density is 0.740 g./cm.^{3}, a reasonable value when compared with the density of solid ethane.

TABLE I

hkl	d_0, Å.	d_c, Å.	I_0	I_c
110	3.72	3.716	VVS	12,200
101	3.65	3.641	VS	10,100
200	2.63	2.628	S	2,930
002	2.52	2.524	MS	473
211	2.13	2.131	S	2,150
112	2.09	2.088	MS	758
220	1.86	1.858	W	548
202	1.82	1.820	W	341
310	1.66	1.662	MW	594
301	...	1.655	...	248
103	1.60	1.603	MS	782
222	...	1.496	...	85
321	1.40	1.400	VW	163
312	...	1.388	...	104
213	1.37	1.368	W	554
400	...	1.314	...	63

With two molecules in a body-centered cell, the molecule is the asymmetric unit and must lie on the four-fold axis and the molecule must either rotate about the axis or orient at random with regard to rotation about it in order to conform statistically to the four-fold symmetry. The structure is built up from one such molecule by the translations of the body-centered tetragonal lattice. The space group is I4mm. Another structure with more disorder with the B–N bonds pointing up and down the *c* axis at random has the space group I4/mmm. It appears improbable because the molecule is expected to have a considerable dipole moment, which would favor the polar arrangement. Moreover, the intensities, although similar, are slightly different and calculations favor the polar form.

The nitrogen atom is at 0,0,0 and the boron at 0,0,z with z selected to give the best agreement between calculated and observed intensities. Special consideration was given to those reflections not observed although permitted by the cell. The scattering factors are those of McWeeny[2] with the scattering power of two hydrogens added to each to allow roughly for the hydrogens of the molecule. The multiplicity, Lorentz and polarization factors are included, assuming that Cu Kα rays were used by Shore and Parry. An asymmetric temperature factor of the form exp $[-\alpha (h^2 + k^2) - \beta l^2]$ was used, with $\alpha = 0.08$ and $\beta = 0.03$, for direct application to intensities.

The agreement is as good as the qualitative nature of the data will permit. The parameter z is 0.31 ± 0.01, and the B–N distance is 1.56 ± 0.05Å. Each boron has as nearest neighbors on other molecules one nitrogen at 3.49 Å. along the *c* axis and four nitrogens at 3.84 Å. on molecules produced by the body-centering. Each nitrogen has a similar set of borons as nearest neighbors. As one expects the borons and nitrogens to carry opposite formal charges, this arrangement leads to charge compensation between neighbors.

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(2) R. McWeeny, *Acta Cryst.*, **4**, 513 (1951).

THE STRUCTURE OF H_3NBH_3

Sir:

The preparation, chemical properties and approximate molecular weight of H_3NBH_3 have been determined by Shore and Parry,[1] who also indicated its crystalline nature. We have determined the crystal structure from a sample supplied to us by Professor R. W. Parry, and have strong supporting evidence that the monomeric formula H_3NBH_3 is correct.

Powder diffraction patterns, taken both on film in a cylindrical camera and on the General Electric XRD-3 unit, were readily indexed on a tetragonal unit cell with dimensions $a = 5.234$ and $c = 5.027$ Å. ($\lambda = 1.542$ Å.). Observation of reflections only when $h + k + l$ is even indicated a body centered unit cell, thus requiring an even number of molecules. An experimental observation that the density of the material is less than that of water then indicated only two molecules in the unit cell and thus led to a calculated density of 0.74 g. cm.$^{-3}$. Normal decline of the *hk*0 intensities, and a positive pyroelectric test then led to a structure in the polar space group C_{4v}^9—I4mm., in which all B–N bonds have the same orientation along the *c* axis. Consequently, the atomic positions are $0,0,z_B$; $\frac{1}{2}, \frac{1}{2}, \frac{1}{2} + z_B$; $0,0,z_N$; $\frac{1}{2}, \frac{1}{2}, \frac{1}{2} + z_N$. The only short B–N distance, obtained from $z_B - z_N = 0.32$, is 1.6 Å. Hydrogen atoms were not located, but were introduced into the calculated structure factors (Table I) as axial rotators about the B–N axis, since orientational disorder or rotation is indicated by the four-fold axis along the B–N axis.

The crystal structure is very similar to that[2] of HCN, and certain aspects of that determination apply here. For example, the thermal motion is anisotropic, thus requiring the temperature factor $\exp[-0.023 (h^2 + k^2) - 0.013 l^2]$, which has been multiplied into the $F_{\text{calcd.}}$ (Table I). Also a transition has been observed at $-43 \pm 5°$ which leaves the major features of the structure unchanged, and causes the appearance of only very faint additional lines in the powder pattern. As in the HCN, there is a strong interaction of the scale and temperature factors with the distance parameter which causes the probable error of the B–N distance to be very large, about 0.2 Å. Finally, the non-polar structure based on the space group D_{2h}^7—$P_{4/n}$ mm was

(1) S. G. Shore and R. W. Parry, THIS JOURNAL, **77**, 6084 (1955).

(2) W. J. Dulmage and W. N. Lipscomb, *Acta Cryst.*, **4**, 330 (1951).

also eliminated on the basis of detailed comparison of $F_{\text{calcd.}}$ with the lower limit of observation on our films for those reflections for which $h + k + l$ is odd.

TABLE I

SPACING AND INTENSITY DATA FOR H_3NBH_3

hkl	$\sin^2\theta$ obs.	$\sin^2\theta$ calcd.	*F* obs.	*F* calcd.
110	0.0439	0.0434	7.66	7.02
101	unresolved	.0452	3.24	3.79
200	0.0868	.0868	4.56	4.56
002	.0941	.0941	4.18	4.31
121	.1317	.1320	2.34	2.30
112	.1377	.1374	3.67	2.39
220	.1732	.1735	2.57	2.76
202	.1809	.1808	2.72	1.54
130	.2172	.2169	2.73	2.50
301		.2188		2.35
103	.2334	.2333	2.96	3.19
222	.2671	.2676	0.69	1.00
231	.3045	.3055	1.17	1.29
132		.3110		1.16
123	.3198	.3201	1.82	2.37

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FORMYLGLYCINAMIDINE RIBOTIDE AND 5-AMINOIMIDAZOLE RIBOTIDE—INTERMEDIATES IN THE BIOSYNTHESIS OF INOSINIC ACID *DE NOVO*[1]

Sir:

In preliminary experiments from this Laboratory,[2] it has been reported that (α-N-formyl)-glycinamide ribotide (FGAR) reacted with glutamine in ethanol-precipitated extracts of pigeon liver to yield a new arylamine ribotide. Upon incubation of this arylamine with aspartic acid, 5-amino-4-imidazolecarboxamide ribotide was formed. We wish to report further studies upon the characterization of the enzymatic systems involved in the above process, as well as the identification by chemical analysis of the arylamine as 5-aminoimidazole ribotide (AIR). Furthermore, by fractionation of the enzymatic components, a second compound, which is an intermediate in the conversion of FGAR to AIR, has been isolated, purified and identified as (α-N-formyl)-glycinamidine ribotide (FGAM).

The enzymatic system responsible for the conversion of FGAR to AIR was found in a fraction of pigeon liver extract precipitating between 13 and 33% ethanol concentration. Reaction took place only upon the addition of glutamine and adenosine triphosphate (ATP). The conversion was conveniently followed by measurement of the formation of a chromophore with an absorption maximum at 500 mμ when the arylamine was treated with the Bratton-Marshall reagents.[3]

AIR behaves as a typical mononucleotide in regard to its properties on ion exchange resins. It has been isolated from deproteinized incubation mixtures by chromatography on Dowex 1 acetate with 0.04 *M* ammonium acetate buffer, *p*H 5.3. The compound was precipitated from 85% ethanol in crude form as the barium salt. To remove certain impurities, chiefly glutamine, a solution of this water-soluble barium salt was acidified (*p*H 2) and passed through a Dowex 50 sodium column. The eluate was then rechromatographed on Dowex 1 acetate. A barium salt was again formed, reprecipitated from water–ethanol and dried *in vacuo* over phosphorus pentoxide. All of the above chromatographic operations were performed at 3° because of the considerable instability of the arylamine.

Analysis of the compound by methods previously described[4] is shown in Table I.

TABLE I

ANALYSES OF 5-AMINOIMIDAZOLE RIBOTIDE AND (α-N-FORMYL)-GLYCINAMIDINE RIBOTIDE

	Molar ratio (glycine = 1.00)		
	AIR	$FGAM_1$	$FGAM_2$
Glycine	1.00	1.00	1.00
Formic acid	1.10	0.93	1.03
Acid-labile N	2.27	1.73	1.80
Total N	3.28	...	2.74
Pentose	1.00	0.99	0.90
Organic P	1.26	0.99	0.93

The results show that glycine, formic acid, acid-labile N, total N, pentose and organic phosphorus were liberated from AIR in the approximate molar ratios of 1:1:2:3:1:1. It is known that, upon hydrolysis, glycine, formic acid and ammonia are produced from the free base, 5-aminoimidazole, in the ratios of 1:1:2.[5]

The chemical constitution of the heterocyclic ring system of AIR was established by conversion of AIR-2-C^{14} to the ureido derivative, followed by hydrolysis to the free base and admixture with unlabeled 5-ureidoimidazole[6] as carrier. From the mixture, a picrate of 5-ureidoimidazole was isolated, which could be recrystallized to constant specific activity. The structure of the new arylamine ribotide, which is consistent with all of the above data, is presented below.[7] On the basis of the molecular weight calculated for this structure (430), the sample obtained for the above analysis was approximately 60 per cent. pure.

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[1] This work was supported by grants-in-aid from the National Cancer Institute, National Institutes of Health, United States Public Health Service, the Damon Runyon Memorial Fund for Cancer Research, Inc., and the National Science Foundation.

[2] B. Levenberg, S. C. Hartman and J. M. Buchanan, *Federation Proc.*, **14**, 243 (1955).

[3] A. C. Bratton and E. K. Marshall, Jr., *J. Biol. Chem.*, **128**, 537 (1939).

[4] S. C. Hartman, B. Levenberg and J. M. Buchanan, THIS JOURNAL, **77**, 501 (1955).

[5] G. Hunter and J. A. Nelson, *Canadian J. Research*, **19B**, 296 (1941).

[6] G. Hunter and I. Hlynka, *Biochem. J.*, **31**, 488 (1937).

[7] The compound isolated from bacterial sources by S. H. Love and J. S. Gots, *J. Biol. Chem.*, **212**, 647 (1955), has recently been identified in this laboratory, by similar procedures, as 5-aminoimidazole riboside (S. H. Love and B. Levenberg unpublished data). It is of interest that the heterocyclic ring structure of 5-aminoimidazole represents an aglycone common to the pathways of purine *de novo* synthesis in avian and bacterial systems as well as to the reactions of purine catabolism in microorganisms (J. C. Rabinowitz and W. E. Pricer, Jr., *Federation Proc.*, **14**, 266 (1955)).