in a series of exposures between each of which the crystal was turned 10′. The total errors in crystal positions lead to $\Delta\theta = 5'$. No significant errors were to be found in the eccentricity and graduation of the circular scale. The difference between $\theta_{15,0,\overline{15}}$ and $\theta_{\overline{15},0,15}$ was only 2′.

The following direct lattice elements were obtained:

$$a = 14{\cdot}95 \pm 0{\cdot}02,\quad b = 7{\cdot}54 \pm 0{\cdot}03,\quad c = 15{\cdot}80 \pm 0{\cdot}02\ \text{Å};\quad \beta = 102^\circ\ 13' \pm 7'.$$

They were measured with both Cu $K\alpha_1$, and Cu $K\alpha_2$ radiations. The agreement is very good: the differences are smaller than the theoretical errors obtained with $\Delta\theta = 5'$.

The equatorial vectors chosen have approximately the directions of a, c and the normal to the bisector of β^*. Owing to this, the errors in the direct cell constants are of the same order of magnitude as the measured reciprocal vectors.

Systematic extinctions were found for $h0l$ reflexions with $h+l$ odd and for $0k0$ reflexions with k odd. The space group is thus $P2_1/n$, in agreement with the absence of a piezoelectric effect. (We are indebted to Mr J. Baecklund for the use of his apparatus.)

The density was determined by the flotation and the pycnometric methods, both of which gave 1·89±0·02 g.cm.$^{-3}$. On the basis of eight formula units per unit cell the calculated density is 1·89 g.cm.$^{-3}$. The number of formula units in the unit cell indicates that there are either two non-equivalent molecules or a dimeric molecule in the crystalline state. A complete crystal-structure determination has been started at this Institute.

We wish to thank Prof. G. Hägg for all the facilities placed at our disposal and Prof. A. Tiselius for his great interest. Thanks are also due to Dr I. Lindqvist, Mr T. Löfgren and A. Rosenberg for valuable discussions. These investigations were made possible by a grant from The Royal Swedish Academy of Sciences (Wallmarkska fonden) for which we express our thanks.

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Unit cell and space group for sodium tetraborate, $Na_2O.4B_2O_3$.

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Morey & Merwin (1936) have reported two modifications of sodium tetraborate. The stable modification at high temperatures separates at 816° C. from a melt of the stoichiometric composition $Na_2O.4B_2O_3$. Single crystals suitable for X-ray work were grown in a platinum crucible at 700° C. from a melt containing an excess of boron oxide. X-ray powder patterns established these crystals as identical with the phase crystallizing at 816° C. from the melt of stoichiometric composition.

Oscillation and Weissenberg exposures around three axes revealed a monoclinic symmetry. The following dimensions for the unit cell were found:

$$a = 6{\cdot}50,\quad b = 17{\cdot}80,\quad c = 8{\cdot}37\ \text{Å},\quad \beta = 96{\cdot}5^\circ.$$

These figures are estimated to be accurate within 0·5%. With four $Na_2O.4B_2O_3$ in the unit cell, the calculated density is 2·35 g.cm.$^{-3}$. A density of 2·32 g.cm.$^{-3}$ was observed by the flotation method.

Systematic extinctions were observed only in the $0k0$ reflections, the extinctions occurring for $k = 2n+1$. This is compatible with the space group $P2_1$ or $P2_1/m$.

Reference

Morey, G. W. & Merwin, H. E. (1936). *J. Amer. Chem. Soc.* **58**, 2248.

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The crystal structure of urea phosphate.

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A preliminary analysis, in this laboratory, of the crystal structure of urea phosphate, $OC(NH_2)_2.H_3PO_4$, has been reported by Sundera-Rao & Pepinsky (1954). That analysis was based upon (010) and (001) projections. Indications of a very interesting hydrogen-bond system prompted a complete three-dimensional study. Some results of the complete analysis are reported here.

The crystals are orthorhombic with

$$a = 17{\cdot}68,\quad b = 7{\cdot}48,\quad c = 9{\cdot}06\ \text{Å};$$

the observed density is 1·770 g.cm.$^{-3}$. With $Z = 8$ molecules per unit cell, the asymmetric unit contains one $OC(NH_2)_2.H_3PO_4$. Systematic extinctions indicate space group *Pbca*.

The structure was solved (Sundera-Rao & Pepinsky, 1954) first in the (010) projection by means of the linear inequalities of Okaya & Nitta (1952), and then in the (001) projection using the isomorphous arsenate. Refinement of the phosphate was accomplished using $F_o - F_c$ difference maps on both projections.

Three-dimensional data were taken with Cu $K\alpha$ radiation, using the multiple-film technique (ten DuPont Type 608 films) and visual comparison of intensities. Structure-factor calculations and least-squares refinement cycles were done for ten iterations, using a modification of Sayre's (1956) program on the IBM 704 calculator. During the process the disagreement factor, $R_1 = \Sigma||F_o|-|F_c|| \div \Sigma|F_o|$ (calculated for $F_o \neq 0$ only) dropped from 34·5 to 15·8%. Table 1 lists final coordinates and temperature factors.

Table 1. *Final coordinates and temperature factors for urea phosphate*

	x/a	y/b	z/c	B (Å^2)
P	0·3111	0·2755	0·3092	1·378
O_1	0·3393	0·0905	0·3618	2·366
O_2	0·2776	0·3847	0·4351	2·130
O_3	0·2464	0·2498	0·1931	2·439
O_4	0·3795	0·3662	0·2392	2·704
O_u	0·4474	0·6327	0·3102	3·168
N_1	0·5085	0·7800	0·4895	2·695
N_2	0·3958	0·6378	0·5431	3·101
C	0·4503	0·6814	0·4466	2·263

All intramolecular bond distances and angles, shown in Table 2, appear normal. The phosphate group forms a distorted tetrahedron, while the planar urea molecule shows slightly shortened C–N distances and a slightly lengthened C–O distance.

Table 2. *Bond distances and angles in urea phosphate*

$P-O_1$	1·546 Å	O_1-P-O	112·0°
$P-O_2$	1·523	O_1-P-O_3	109·5
$P-O_3$	1·565	O_1-P-O_4	105·8
$P-O_4$	1·523	O_2-P-O_3	106·6
		O_2-P-O_4	109·4
$C-N_1$	1·323	O_3-P-O_4	110·7
$C-N_2$	1·340		
$C-O_u$	1·290	N_1-C-O_u	118·0
		N_2-C-O_u	121·9
		N_1-C-N_2	120·2

The hydrogen-bond network, which is responsible for the close packing in this structure, is very involved. From the list of short intermolecular distances shown in Table 3, it is seen that the seven available hydrogen atoms must be distributed to allow nine close approaches (each asymmetric unit composed of one urea molecule plus the closest phosphate group is identified by the number in parentheses). The urea oxygen forms a very short O–H · · · O bond, of length 2·41 Å, with one oxygen of the nearest phosphate group, and also forms two O · · · H–N bonds with the nitrogens of another urea molecule. Three of the phosphate oxygens form one O–H · · · O bond and one O · · · H–N bond each, while the fourth oxygen is involved in two O–H · · · O bonds and one O · · · H–N bond. Each nitrogen forms three N–H · · · O bonds.

Table 3. *Short intermolecular distances in urea phosphate*

$O_u(5)-O_4(5)$	2·414 Å	$O_4(5)-N_1(7)$	2·936 Å
$O_u(5)-N_2(3)$	3·103	$O_4(5)-O_u(5)$	2·414
$O_u(5)-N_1(3)$	3·167		
		$N_1(5)-O_u(3)$	3·167
$O_1(5)-N_1(1)$	3·159	$N_1(5)-O_1(1)$	3·159
$O_1(5)-O_2(2)$	2·659	$N_1(5)-O_4(7)$	2·936
$O_2(5)-O_1(2)$	2·659	$N_2(5)-O_u(3)$	3·103
$O_2(5)-O_3(3)$	2·603	$N_2(5)-O_2(5)$	2·983
$O_2(5)-N_2(5)$	2·983	$N_2(5)-O_3(8)$	2·976
$O_3(5)-O_2(3)$	2·603		
$O_3(5)-N_2(8)$	2·976		

Figures in parentheses identify which molecule of 8 in unit cell is involved.

The structure is comprised of alternate layers of phosphate tetrahedra and of urea molecules, with the layers parallel to (100). Four of the O–H · · · O bonds are in the phosphate layer, four N–H · · · O bonds are in the urea layer, and the remaining ten hydrogen bonds (two O–H · · · O and eight N–H · · · O) occur between the layers. The packing is very close, as a result, and the high density is explained.

Further three-dimensional refinement, which includes effects of hydrogen scattering, is in progress, and detailed results will be published at a later date.

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