Table 2 reports the main characteristics of the NH_4 groups and details of the hydrogen bonds involving hydrogen atoms of these groups.

Details of the hydrogen-bond scheme involving water molecules are given in Fig. 2, which shows a projection along the *b* axis of a row of NaO_6 octahedra. Numerical data corresponding to these hydrogen bridges are given in Table 2. It should be noticed that one oxygen atom [O(*E*22)] acts as an acceptor for three bonds.

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Structure of Pentarubidium Heptahydrogentetrakis(phosphate)

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Abstract. $Rb_5H_7(PO_4)_4$, $M_r = 814{\cdot}29$, orthorhombic, *Pnma*, $a = 28{\cdot}57$ (6), $b = 10{\cdot}277$ (5), $c = 6{\cdot}090$ (3) Å, $V = 1788{\cdot}3$ Å^3, $Z = 4$, $D_x = 3{\cdot}024$ Mg m^{-3}, λ(Ag $K\alpha$) $= 0{\cdot}5608$ Å, $\mu = 8{\cdot}017$ mm^{-1}, $F(000) = 1520$, $T =$ 293 K, final $R = 0{\cdot}056$ for 1060 unique reflexions. Isolated PO_4 tetrahedra intercalate in a three-dimensional network of RbO_{10}, RbO_9 and RbO_8 polyhedra. Both P and Rb atoms are located in mirror planes. This compound is the first example of an alkali monophosphate with such a chemical formula.

Introduction. During experiments aimed at preparing the high-temperature form of RbH_2PO_4 a new type of alkali monophosphate has been characterized: $Rb_5H_7(PO_4)_4$. Schematically, the chemical reaction leading to this salt corresponds to the departure of one mole of H_3PO_4 for five moles of RbH_2PO_4:

$$5RbH_2PO_4 \rightarrow H_3PO_4 + Rb_5H_7(PO_4)_4.$$

Experimental. Rubidium dihydrogenmonophosphate is normally prepared by evaporation at room temperature of a solution containing stoichiometric amounts of H_3PO_4 and Rb_2CO_3. If a small excess of H_3PO_4 is added and the resulting solution kept at 353 K for 1 d, large orthorhombic prisms form, which were originally thought to be crystals of the high-temperature form of RbH_2PO_4. The present crystal structure study shows the correct chemical formula to be $Rb_5H_7(PO_4)_4$. D_m not measured. Prismatic crystal ($0{\cdot}20 \times 0{\cdot}20 \times 0{\cdot}20$ mm). Philips PW 1100 diffractometer. Systematic absences: $h0l$, $h = 2n$; $0kl$, $k + l = 2n$. 25 reflexions ($10 < \theta < 13°$) used for refining the unit cell. ω scan. 2235 reflexions measured ($3 < \theta < 30°$). $H_{max} = 50$, $K_{max} = 18$, $L_{max} = 9$. Scan width 1·20°, scan speed 0·02° s^{-1}, total background-measuring time 20 s. Lorentz–polarization correction; no absorption correction. Classical methods for structure determination: Patterson function and successive Fourier syntheses. Anisotropic full-matrix least-squares refinement on *F*. Unit weights. Total number of unique reflexions 2203. Final refinement cycles with 1060 reflexions ($F_o > 3\sigma_F$). Final $R = 0{\cdot}056$ ($wR = 0{\cdot}064$). For the complete set of unique reflexions (2203 *HKL*) $R = 0{\cdot}080$, $S = 5{\cdot}811$. Max. $\Delta/\sigma = 0{\cdot}38$ (extinction coefficient). Extinction coefficient refined, $g = 0{\cdot}586 \times 10^{-7}$ (Stout & Jensen, 1968). Max. peak height in difference Fourier map 0·46 e Å^{-3}. Scattering factors for neutral atoms and f', f'' from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius (1977) *SDP* used for all calculations.

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Discussion. Table 1 reports the final atomic coordinates,* while Fig. 1 is a projection of the atomic arrangement along **c**. Both P and Rb atoms are located on mirror planes at $z = \frac{1}{4}$ or $\frac{3}{4}$. Table 2 reports the main geometrical features of the four PO_4 tetrahedra, while Table 3 presents the Rb—O distances in the RbO_n polyhedra. Within a range of 3·50 Å Rb(1) and Rb(3) have ten O neighbours, Rb(2) nine and Rb(4) and Rb(5) eight. These polyhedra build up a three-dimensional network. Owing to the quality of the crystal H atoms could not be located.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42402 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates for* $Rb_5H_7(PO_4)_4$, *with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
Rb(1)	0·37420 (6)	0·1766 (2)	$\frac{3}{4}$	2·36 (6)
Rb(2)	0·42038 (7)	0·4120 (2)	$\frac{1}{4}$	2·23 (6)
Rb(3)	0·06097 (7)	0·2912 (2)	$\frac{1}{4}$	2·53 (6)
Rb(4)	0·28078 (7)	0·1424 (2)	$\frac{1}{4}$	2·33 (6)
Rb(5)	0·19212 (7)	0·2376 (2)	$\frac{3}{4}$	2·44 (6)
P(1)	0·2248 (2)	0·4547 (6)	$\frac{1}{4}$	1·6 (1)
P(2)	−0·0039 (2)	0·1908 (6)	$\frac{3}{4}$	1·7 (1)
P(3)	0·3474 (2)	0·5170 (6)	$\frac{3}{4}$	1·8 (1)
P(4)	0·4142 (2)	0·0131 (6)	$\frac{1}{4}$	1·8 (1)
O(11)	0·2796 (5)	0·096 (2)	$\frac{3}{4}$	2·7 (5)
O(12)	0·1732 (5)	0·396 (2)	$\frac{1}{4}$	2·6 (5)
O(13)	0·2480 (3)	0·401 (1)	0·044 (2)	2·5 (3)
O(21)	0·4773 (4)	0·248 (1)	0·956 (2)	2·6 (3)
O(22)	0·9848 (5)	0·040 (1)	$\frac{3}{4}$	2·1 (4)
O(23)	0·0501 (4)	0·186 (1)	$\frac{3}{4}$	2·1 (4)
O(31)	0·3333 (4)	0·434 (1)	0·543 (2)	3·3 (4)
O(32)	0·1777 (5)	0·143 (2)	$\frac{1}{4}$	3·3 (6)
O(33)	0·1010 (5)	0·025 (2)	$\frac{1}{4}$	3·2 (6)
O(41)	0·0536 (3)	0·522 (1)	0·958 (2)	2·6 (3)
O(42)	0·1049 (4)	0·375 (1)	$\frac{3}{4}$	2·0 (4)
O(43)	0·3792 (4)	0·118 (1)	$\frac{1}{4}$	2·1 (4)

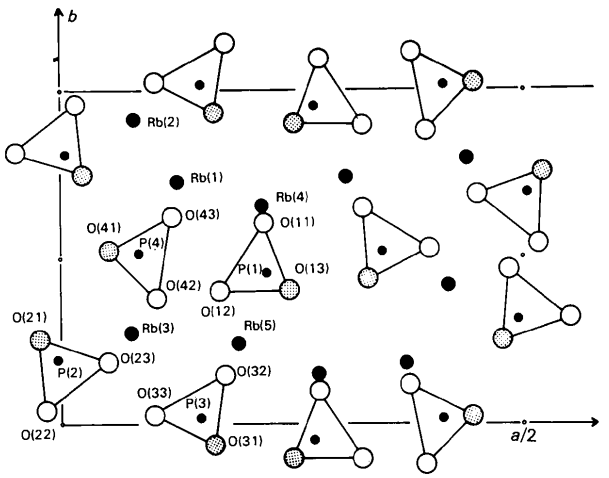

Fig. 1. Projection of the atomic arrangement of $Rb_5H_7(PO_4)_4$ along **c**. Dotted atoms correspond to superimposed O atoms related by the mirror symmetry.

Table 2. *Main geometrical features of the four independent* PO_4 *tetrahedra (distances in* Å, *angles in* °)

P(1)O_4 tetrahedron			
P(1)–O(13)	1·524 (5) (×2)	P(1)–O(11)	1·462 (8)
P(1)–O(12)	1·594 (6)		
O(11)–P(1)–O(12)	107·2 (4)	O(11)–O(12)	2·462 (10)
O(11)–P(1)–O(13)	113·4 (2)	O(11)–O(13)	2·496 (7)
O(12)–P(1)–O(13)	105·4 (2)	O(12)–O(13)	2·480 (7)
O(13)–P(1)–O(13)	111·2 (4)	O(13)–O(13)	2·515 (9)
P(2)O_4 tetrahedron			
P(2)–O(21)	1·502 (5) (×2)	P(2)–O(23)	1·545 (6)
P(2)–O(22)	1·587 (7)		
O(21)–P(2)–O(21)	113·1 (4)	O(21)–O(21)	2·507 (10)
O(21)–P(2)–O(22)	109·7 (2)	O(21)–O(22)	2·527 (7)
O(21)–P(2)–O(23)	111·8 (2)	O(21)–O(23)	2·523 (6)
O(22)–P(2)–O(23)	99·9 (3)	O(22)–O(23)	2·399 (8)
P(3)O_4 tetrahedron			
P(3)–O(31)	1·579 (6) (×2)	P(3)–O(33)	1·476 (6)
P(3)–O(32)	1·480 (8)		
O(31)–P(3)–O(31)	106·2 (4)	O(31)–O(31)	2·525 (12)
O(31)–P(3)–O(32)	110·5 (2)	O(31)–O(32)	2·514 (8)
O(31)–P(3)–O(33)	106·7 (3)	O(31)–O(33)	2·451 (8)
O(32)–P(3)–O(33)	115·7 (5)	O(32)–O(33)	2·503 (10)
P(4)O_4 tetrahedron			
P(4)–O(41)	1·568 (5) (×2)	P(4)–O(43)	1·474 (6)
P(4)–O(42)	1·516 (6)		
O(41)–P(4)–O(41)	107·5 (4)	O(41)–O(41)	2·529 (10)
O(41)–P(4)–O(42)	105·3 (2)	O(41)–O(42)	2·452 (7)
O(41)–P(4)–O(43)	110·9 (2)	O(41)–O(43)	2·506 (6)
O(42)–P(4)–O(43)	116·2 (4)	O(42)–O(43)	2·538 (9)

Table 3. Rb—O *distances* (Å) *in the* RbO_n *polyhedra*

Rb(1)O_{10} polyhedron			
Rb(1)–O(11)	2·826 (6)	Rb(1)–O(31)	3·152 (6) (×2)
Rb(1)–O(12)	3·186 (7)	Rb(1)–O(41)	3·157 (5) (×2)
Rb(1)–O(21)	3·283 (5) (×2)	Rb(1)–O(43)	3·106 (1) (×2)
Rb(2)O_9 polyhedron			
Rb(2)–O(21)	2·949 (5) (×2)	Rb(2)–O(31)	3·069 (5) (×2)
Rb(2)–O(22)	3·010 (6)	Rb(2)–O(33)	3·316 (3) (×2)
Rb(2)–O(23)	2·941 (6)	Rb(2)–O(43)	3·239 (7)
Rb(3)O_{10} polyhedron			
Rb(3)–O(12)	3·383 (6)	Rb(3)–O(33)	2·964 (8)
Rb(3)–O(21)	3·016 (5) (×2)	Rb(3)–O(41)	2·972 (5) (×2)
Rb(3)–O(23)	3·246 (2) (×2)	Rb(3)–O(42)	3·405 (3) (×2)
Rb(4)O_8 polyhedron			
Rb(4)–O(11)	3·082 (1) (×2)	Rb(4)–O(32)	2·946 (7)
Rb(4)–O(13)	3·087 (5) (×2)	Rb(4)–O(43)	2·822 (5)
Rb(4)–O(13)	3·165 (5) (×2)		
Rb(5)O_8 polyhedron			
Rb(5)–O(11)	2·890 (6)	Rb(5)–O(32)	3·223 (2) (×2)
Rb(5)–O(12)	3·495 (3) (×2)	Rb(5)–O(42)	2·868 (6)
Rb(5)–O(13)	2·927 (5) (×2)		

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