observation can probably be explained by the existence of the cluster of water molecules discussed below.

The water molecule cluster. The water molecules of this compound are not dispersed inside the arrangement but they assemble themselves to form a centrosymmetric cluster of eight molecules. The central part of this group is an almost regular hexagon built by O(*W*2), O(*W*3) and O(*W*4). Angles O(*W*3)—O(*W*2)—O(*W*4), O(*W*2)—O(*W*3)—O(*W*4) and O(*W*2)—O(*W*4)—O(*W*3) are respectively 123·7 (1), 120·4 (1) and 111·1 (1)° while the distances O(*W*2)—O(*W*3), O(*W*3)—O(*W*4) and O(*W*2)—O(*W*4) are respectively 2·775 (4), 2·751 (5) and 2·770 (5) Å. Inside this ring the water molecules are tied by strong hydrogen bonds (Table 2). Two centrosymmetric branches complete this cluster; they start from the O(*W*3) water molecule of the hexagon and are built by O(*W*1) with a distance O(*W*3)—O(*W*1) = 2·917 (3) Å. O(*W*1) is not connected by hydrogen bonds to the central hexagon. Similar polygonal clusters of water molecules have already been observed by the authors in condensed phosphate chemistry: an almost regular pentagon of water molecules has been observed in $Li_4P_4O_{12}.5H_2O$ (Averbuch-Pouchot & Durif, 1986), and an almost regular hexagon with $\bar{3}$ internal symmetry in $Cd_3P_6O_{18}.6H_2O$ (Averbuch-Pouchot, 1989*b*) and $Mn_3P_6O_{18}.6H_2O$ (Averbuch-Pouchot & Durif, 1989). The present arrangement thus provides the first example of a branched polygon of water molecules.

$(NH_3NH_3)_2(NH_2NH_3)_2P_6O_{18}$. For the second title compound the atomic arrangement is rather simple. Here the ring anion is centrosymmetric and develops around the inversion centre located at $0,0,\frac{1}{2}$. If compared with the other P_6O_{18} ring anions of $\bar{1}$ internal symmetry the present one is not very distorted, the P—P—P angles ranging from 94·06 to 110·31°. The main geometrical features of this ring are reported in Table 4. As mentioned in the *Experimental* section two types of cations coexist in the atomic arrangement: a hydrazinium(1+), NH_2—NH_3, and a hydrazinium(2+), NH_3—NH_3. The first one consists of N(3) and N(4) and the second one consists of N(1) and N(2) with almost identical N—N distances (1·436 and 1·439 Å). As can be seen in Fig. 2 these groups are lining the central channel parallel to the *c* axis and are connected to the external O atoms of the rings by hydrogen bonds whose details are reported in Table 4.

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Structure of a Cobalt Magnesium Diphosphate: $(Mg_xCo_{1-x})_2P_2O_7$

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Abstract. $(Mg_{0.54}Co_{0.46})_2P_2O_7$, $M_r = 254.42$, monoclinic, $P2_1/c$, $a = 6.977$ (1), $b = 8.330$ (2), $c = 9.032$ (9) Å, $\beta = 113.74$ (1)°, $V = 480.45$ Å^3, $Z = 4$, $D_x = 3.519$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, μ = 47·916 cm^{-1}, $F(000) = 495.2$, $T = 293$ K, $R = 0.029$, $wR = 0.034$ for 1306 independent reflections with $I \geq 3\sigma(I)$. The structure of $(Mg_{0.54}Co_{0.46})_2P_2O_7$ is isotypic with α-$Mg_2P_2O_7$ and α-$Co_2P_2O_7$. The Co and Mg

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Table 1. *Atomic positional parameters and equivalent isotropic thermal parameters* (Å^2)

	x	y	z	B_{eq}
(Mg,Co)(1)	0·7329 (1)	0·07039 (8)	0·1097 (1)	0·534 (7)
(Mg,Co)(2)	0·7989 (1)	0·44150 (9)	0·17327 (8)	0·608 (9)
P(1)	0·0311 (1)	0·2268 (1)	0·46732 (9)	0·34 (1)
P(2)	0·4423 (1)	0·2672 (1)	0·2599 (1)	0·38 (1)
O(1)	0·2495 (4)	0·3273 (3)	0·1008 (3)	0·83 (4)
O(2)	0·0511 (4)	0·3880 (3)	0·3962 (3)	0·60 (4)
O(3)	0·8765 (3)	0·2642 (3)	0·0477 (3)	0·51 (3)
O(4)	−0·0168 (4)	0·0906 (3)	0·3454 (3)	0·52 (3)
O(5)	0·4770 (4)	0·4027 (3)	0·3822 (3)	0·56 (4)
O(6)	0·3898 (4)	0·1100 (3)	0·3139 (3)	0·98 (4)
O(7)	0·6199 (3)	0·2589 (3)	0·2029 (3)	0·51 (3)

Table 2. *Interatomic distances* (Å) *and angles* (°) *in* $[P_2O_7]$, $[MO_6]$ *and* $[MO_5]$

$[P_2O_7]$ group

P(1)	O(1^{v})	O(2)	O(3^{iv})	O(4)
O(1^{v})	1·580 (3)	2·544 (4)	2·502 (3)	2·405 (3)
O(2)	110·5 (1)	1·517 (3)	2·508 (4)	2·526 (4)
O(3^{iv})	107·5 (1)	111·2 (2)	1·524 (3)	2·538 (3)
O(4)	101·7 (1)	112·5 (2)	113·0 (1)	1·520 (2)

P(2)	O(1)	O(5)	O(6)	O(7)
O(1)	1·601 (3)	2·474 (4)	2·532 (4)	2·433 (3)
O(5)	104·5 (1)	1·528 (3)	2·526 (4)	2·518 (4)
O(6)	109·8 (2)	113·5 (2)	1·492 (3)	2·531 (4)
O(7)	102·4 (1)	111·4 (1)	114·2 (2)	1·521 (3)

$[MO_5]$ square pyramid

(Mg,Co)(2)	O(2^{i})	O(3)	O(4^{iii})	O(6^{iii})	O(7)
O(2^{i})	2·119 (3)	3·059 (4)	2·690 (4)	3·429 (4)	3·001 (3)
O(3)	94·1 (1)	2·061 (3)	2·916 (4)	3·892 (4)	2·680 (3)
O(4^{iii})	81·0 (1)	91·2 (1)	2·020 (3)	2·964 (4)	4·053 (3)
O(6^{iii})	114·5 (1)	151·2 (1)	96·4 (1)	1·957 (3)	2·924 (4)
O(7)	92·1 (1)	81·36 (9)	169·5 (1)	93·7 (1)	2·051 (3)

$[MO_6]$ octahedra

(Mg,Co)(1)	O(2^{ii})	O(3)	O(4^{i})	O(5^{ii})	O(5^{v})	O(7)
O(2^{ii})	2·154 (3)	3·179 (4)	2·690 (4)	3·024 (3)	3·543 (4)	4·152 (4)
O(3)	97·0 (1)	2·090 (3)	2·872 (3)	4·096 (4)	2·926 (4)	2·680 (3)
O(4^{i})	77·4 (1)	85·4 (1)	2·147 (2)	3·413 (4)	4·249 (3)	2·721 (3)
O(5^{ii})	92·15 (9)	164·4 (1)	109·0 (1)	2·044 (3)	2·587 (5)	3·068 (4)
O(5^{v})	112·0 (1)	88·0 (1)	169·16 (9)	76·8 (1)	2·121 (3)	2·976 (3)
O(7)	157·6 (1)	80·01 (8)	80·16 (9)	96·1 (1)	90·2 (1)	2·079 (3)

Symmetry codes: (i) $1+x, y, z$; (ii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iv) $x-1, \frac{1}{2}-y, \frac{1}{2}+z$; (v) $x, \frac{1}{2}-y, z-\frac{1}{2}$.

atoms are distributed at random between $[MO_5]$ pyramids and $[MO_6]$ octahedra, which share their edges forming $[(Mg_xCo_{1-x})_2O_3]_\infty$ layers connected by layers of $[P_2O_7]$ groups. A new description of the structure is proposed, in which the $[MO_5]$ pyramids are replaced by very distorted octahedra, showing its great similarity with β-$V_2P_2O_9$.

Introduction. Former investigations in the AMP_2O_7 diphosphate series (A = alkaline-earth, M = divalent metal) have shown the existence of three structural types. $CaCoP_2O_7$ (Riou, Labbe & Goreaud, 1988*a*) and $BaCoP_2O_7$ (Riou, Labbe & Goreaud, 1988*b*) present a mixed framework of $[CoO_6]$ octahedra and tetrahedral $[P_2O_7]$ diphosphate groups, the octahedra being paired by an edge. $CaCoP_2O_7$ is a cage structure with the Ca^{2+} ions inside the cages whereas $BaCoP_2O_7$ is a sheet structure stabilized by the large Ba^{2+} ions located between the sheets. $CaCuP_2O_7$ (Riou & Goreaud, 1990) is a structure derived from α-$Ca_2P_2O_7$ (Calvo, 1968) where the Cu atoms have a fivefold square pyramidal coordination.

We report here on the structure of $(Mg_xCo_{1-x})_2P_2O_7$, isotypic with α-$Mg_2P_2O_7$ (Calvo, 1967) and α-$Co_2P_2O_7$ (Krishnamachari & Calvo, 1972).

Experimental. Crystals were synthesized from a mixture of $(NH_4)_2HPO_4$, $MgCO_3$ and $CoCO_3$ in a 2:1:1 ratio, first heated in a platinum crucible for one hour at 673 K to decompose entirely the phosphate and carbonates. Then the product was ground up and placed in an evacuated silica ampoule for 4 days at 1373 K. Violet crystal without definite shape: 0·033 × 0·057 × 0·103 mm. $2/m$ symmetry with systematic absences $0k0$ for k odd and $h0l$ for l odd. Space group $P2_1/c$. Enraf–Nonius CAD-4 diffractometer. Unit cell: least squares on 25 reflections, $\pm 2\theta$, $8 \leq 2\theta \leq 34°$. Intensity measurement by ω–$\frac{2}{3}\theta$ of $(0·85 + 0·35\tan\theta)°$ and $(1+\tan\theta)$ mm counter slit aperture

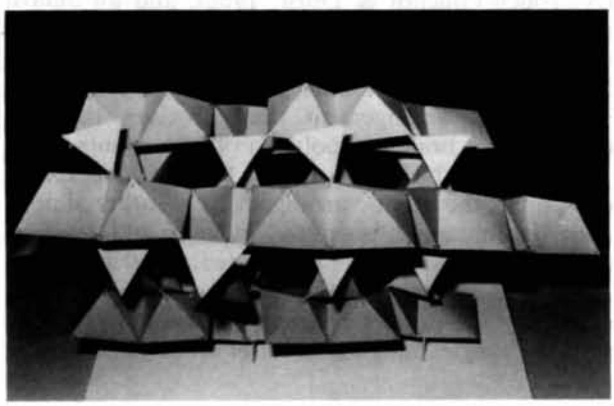

Fig. 1. $(Mg_xCo_{1-x})_2P_2O_7$: polyhedron model viewed along [001].

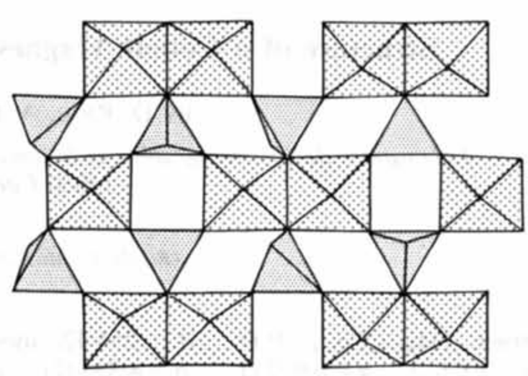

Fig. 2. β-$V_2P_2O_9$: structure viewed along the *a* axis (Leclaire, Chahboun, Groult & Raveau, 1988).

determined by a study of some reflections in the ω–θ plane. Scanning speed adjusted to obtain $\sigma(I)/I < 0{\cdot}018$ or to approach it in a time limited to 60 s. Three standards (13,0,0, 0,16,0 and 0,0,17) for count every 7000 s and orientation every 1200 reflections: no decay. 4295 reflections measured, 1306 reflections ($h \pm 13$, k 16, l 17, $\theta \leq 45°$) with $I \geq 3\sigma(I)$ used to solve and refine the structure. No correction made for extinction or absorption. All subsequent calculation on a MicroVAX II with the *SDP* (B. A. Frenz & Associates, Inc., 1985) system. Composition determined by refinement of the multiplicities of Co(1) and Co(2) and by microprobe analysis. All atoms refined anisotropically. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). $(\Delta/\sigma)_{max} = 0{\cdot}00$, $\Delta\rho < 0{\cdot}675$ e Å^{-3}, $R = 0{\cdot}029$, $wR = 0{\cdot}034$, $w = 1$, $S = 1{\cdot}335$. Atomic parameters are given in Table 1.*

Discussion. These results confirm the ability of $Mg_2P_2O_7$ and $Co_2P_2O_7$ to form a solid solution. The Mg and Co atoms are distributed at random over two sorts of sites. One can notice the large distortion of the $[MO_6]$ octahedra and $[MO_5]$ pyramids (Table 2) which can be considered as a 3 + 3 and 4 + 1 coordination for the metallic atoms respectively. The diphosphate groups have a staggered configuration, with usual P—O bonds, *i.e.* involving three short P—O distances with the terminal O atoms and a longer one with the bridging oxygen (Table 2).

This structure (Fig. 1) can be described as the stacking along [101] of two sorts of layers, mixed layers $[(Mg_{1-x}Co_x)_2O_3]_\infty$ built up from edge-sharing $[MO_6]$ octahedra and $[MO_5]$ pyramids and tetrahedral $[P_2O_4]_\infty$ formed of isolated diphosphate groups. In fact, this oxide exhibits a great similarity with the structure of β-$V_2P_2O_9$ (Gorbunova & Linde, 1978). The $[MO_5]$ pyramids form, with the oxygen O(6), a very distorted octahedron so that the atom M can also be described as being strongly off-centre in this octahedron with an M(2)—O(6) distance of 3·375 (3) Å (against 2·272 Å in β-$V_2P_2O_9$). A comparison of Figs. 1 and 2 shows the marked similarity of the two structures. β-$V_2P_2O_9$ can be described as units of two edge-sharing octahedra linked through $[P_2O_7]$ groups. Such an arrangement is also observed in $(Mg_xCo_{1-x})_2P_2O_7$, in the (014) plane, but in addition, these bi-octahedra share their edges in the orthogonal direction forming the $[(Mg_{1-x}Co_x)_2O_3]_\infty$ layers. (See Fig. 1.)

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53929 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of $Ho_4Mo_4O_{11}$

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Abstract. $Ho_4Mo_4O_{11}$, $M_r = 1219{\cdot}47$, orthorhombic, *Pbam*, $a = 10{\cdot}683$ (3), $b = 15{\cdot}848$ (4), $c = 5{\cdot}6592$ (1) Å, $V = 958{\cdot}1$ (3) Å^3, $Z = 4$, $D_x = 8{\cdot}453$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 380{\cdot}3$ cm^{-1}, $F(000) = 2096$, $T = 295$ K, $R = 0{\cdot}018$ for 1534 observed reflections. The title compound constitutes a new structure type containing infinite chains of *trans*-edge-shared Mo_6 octahedra clusters. The

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