for the P—O distances and the O—P—O angles (1·540 Å and 109·1°) are the normal ones.

The two Cu atoms are located on inversion centres, Cu(1) at (0,0,0) Cu(2) at (1/2, 1/2, 0). Both have an octahedral coordination made by four O atoms and two water molecules for Cu(1) and four water molecules and two O atoms for Cu(2). The distortion of the Cu(2)O_6 octahedron is significantly larger than that of Cu(1)O_6, Cu—O distances varying from 1·962 to 2·287 Å in Cu(1)O_6 and from 1·929 to 2·499 Å in Cu(2)O_6. Within a range of 3·50 Å the ammonium group has an eightfold coordination made by five O atoms and three water molecules with N—O distances varying from 2·794 to 3·384 Å.

The atomic arrangement can be easily described as a succession of ribbons built up by the P_6O_{18} phosphoric ring anions and by the Cu(1)O_6 octahedra spreading parallel to the **c** direction. These ribbons are themselves interconnected along the **b** direction by the Cu(2)O_6 octahedra so as to form a layer parallel to the *bc* plane. The connections so established by the CuO_6 octahedra between the phosphoric groups are not identical; the Cu(1)O_6 octahedra share four of their O atoms with the two adjacent P_6O_{18} groups while Cu(2)O_6 octahedra share only two. Fig. 1 is a projection of this atomic arrangement along the *a* axis. The three-dimensional cohesion is established between these layers by the ammonium polyhedra and the hydrogen-bond network. Table 2 gives the main interatomic distances and bond angles in this atomic arrangment. The drawing was made with *STRUPLO* (Fischer, 1985).

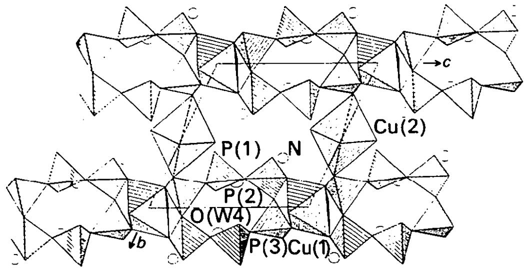

Fig. 1. Projection along the *a* axis of the atomic arrangement of $Cu_2(NH_4)_2P_6O_{18}.8H_2O$. The H atoms have been omitted.

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Crystal Chemistry of *cyclo*-Hexaphosphates. XIII. Structure of Silver Lithium *cyclo*-Hexaphosphate Dihydrate

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Abstract. $Ag_4Li_2P_6O_{18}.2H_2O$, $M_r = 955·206$, triclinic, $P\bar{1}$, $a = 8·408$ (2), $b = 7·602$ (2), $c = 7·566$ (2) Å, $\alpha = 107·47$ (3), $\beta = 106·09$ (3), $\gamma = 72·64$ (3)°, $V = 430·8$ (4), $Z = 1$, $D_x = 3·681$ Mg m^{-3}, $\lambda(Ag\ K\alpha) = 0·5608$ Å, $\mu = 2·562$ mm^{-1}, $F(000) = 448$, room temperature, final $R = 0·033$ for 3318 reflections. The present atomic arrangement has a layer organization. Planes of corner-sharing LiO_4 tetrahedra and P_6O_{18} ring anions alternate with planes of Ag atoms. The hydrogen-bond scheme is described.

Introduction. Up to now only two mixed monovalent cation *cyclo*-hexaphosphates have been investigated: $Li_3Na_3P_6O_{18}.12H_2O$ and $Li_3K_3P_6O_{18}.H_2O$ (Averbuch-Pouchot, 1989). These two compounds correspond to a 1/1 order between the associated cations. In the present work we describe the chemical preparation and crystal structure of a 1/2 order in this class of compound: $Ag_4Li_2P_6O_{18}.2H_2O$.

Experimental. Crystals of the title compound have been prepared by adding an aqueous solution of silver nitrate to an aqueous solution of lithium *cyclo*-hexaphosphate so as to obtain a ratio Ag/Li = 2 in the resulting solution. After some days of evaporation at room temperature large elongated irregular prisms of $Ag_4Li_2P_6O_{18}.2H_2O$ appeared in the solution. The compound is very sparingly soluble in water.

Crystal size: 0·16 × 0·24 × 0·30 mm. Density not measured. Philips PW1100 diffractometer, graphite monochromator. 16 reflections ($10·0 < \theta < 17·0°$) for

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Table 1. *Final atomic coordinates and* B_{eq}(Å^2) *for non-*H *atoms with e.s.d.'s in parentheses*

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

	x	*y*	*z*	B_{eq}/B_{iso}
Ag(1)	−0·40114 (3)	−0·56481 (4)	−0·66557 (4)	1·933 (5)
Ag(2)	0·01486 (4)	−0·30668 (4)	−0·58576 (5)	2·364 (6)
Li	0·6644 (7)	0·7130 (9)	0·0867 (9)	1·7 (1)
P(1)	−0·26852 (9)	−0·2386 (1)	−0·2632 (1)	1·01 (1)
P(2)	−0·09294 (9)	0·2881 (1)	0·0999 (1)	0·97 (1)
P(3)	−0·32677 (9)	0·1752 (1)	−0·2596 (1)	1·02 (1)
O(*E*11)	−0·3953 (3)	−0·2614 (4)	−0·1754 (4)	2·07 (4)
O(*E*12)	−0·2160 (3)	−0·3846 (4)	−0·4318 (4)	1·98 (5)
O(*L*13)	−0·3294 (4)	−0·0428 (3)	−0·3228 (4)	2·59 (5)
O(*L*12)	−0·1057 (3)	−0·2149 (3)	−0·0984 (3)	1·23 (3)
O(*E*21)	−0·3067 (4)	0·2200 (4)	−0·4286 (4)	2·34 (5)
O(*E*22)	0·5288 (3)	0·2910 (4)	−0·1696 (4)	2·29 (5)
O(*L*23)	−0·1517 (3)	0·1725 (3)	−0·1100 (4)	1·76 (4)
O(*E*31)	−0·1328 (3)	0·2125 (3)	0·2382 (4)	1·93 (4)
O(*E*32)	−0·1487 (3)	−0·5048 (3)	0·1152 (4)	1·82 (5)
O(*W*)	0·2201 (3)	0·0899 (3)	0·7261 (3)	1·73 (4)
H(1)	0·196 (8)	0·020 (9)	0·77 (1)	2(1)*
H(2)	0·710 (8)	0·982 (9)	0·31 (1)	2(1)*

* B_{iso}.

refining unit-cell dimensions. $\omega/2\theta$ scan, scan width 1·20°, scan speed 0·03° s^{-1}, total background measuring time 6 s. 4343 reflections collected ($3 < \theta < 30°$), $\pm h$, $\pm k$, l, $h_{max} = 14$, $k_{max} = 13$, $l_{max} = 13$. Two orientation and intensity control reflections ($\bar{2}0\bar{4}$ and 204) measured every three hours without any significant variation. 4058 independent reflections kept after averaging Friedel pairs ($R_{int} = 0·022$). Lorentz and polarization corrections, no absorption correction. Structure solved by classical methods: interpretation of the Patterson map and successive Fourier syntheses. H atoms located by difference Fourier syntheses. Anisotropic full-matrix least-squares refinement (on *F*), isotropic for H atoms. Unit weights. Final refinements with 3318 reflections corresponding to $F > 2\sigma_F$. Final $R = 0·033$ ($wR = 0·034$), $S = 0·979$, max. $\Delta/\sigma = 0·02$, max. peak height in the final difference Fourier synthesis = 1·026 e Å^{-3}. No secondary-extinction correction. Scattering factors for neutral atoms and f', f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B). Enraf–Nonius (1977) *SDP* used for all calculations. Computer used: MicroVAX II.

Table 2. *Main interatomic distances* (Å) *and bond angles* (°) *in the atomic arrangement of* $Ag_4Li_2P_6O_{18}.2H_2O$ *with e.s.d.'s in parentheses*

$P(1)O_4$ tetrahedron

P(1)	O(*E*11)	O(*E*12)	O(*L*13)	O(*L*12)
O(*E*11)	1·476 (3)	2·569 (4)	2·513 (5)	2·449 (4)
O(*E*12)	119·9 (2)	1·491 (3)	2·452 (3)	2·520 (3)
O(*L*13)	110·5 (2)	105·8 (2)	1·582 (3)	2·495 (3)
O(*L*12)	106·0 (1)	109·7 (1)	103·8 (1)	1·589 (2)

$P(2)O_4$ tetrahedron

P(2)	O(*L*12)	O(*L*23)	O(*E*21)	O(*E*22)
O(*L*12)	1·598 (2)	2·344 (3)	2·520 (4)	2·530 (3)
O(*L*23)	94·7 (1)	1·589 (2)	2·525 (4)	2·535 (3)
O(*E*21)	109·5 (1)	110·3 (2)	1·486 (3)	2·542 (4)
O(*E*22)	110·5 (2)	111·3 (1)	117·9 (2)	1·480 (2)

$P(3)O_4$ tetrahedron

P(3)	O(*L*13)	O(*E*31)	O(*E*32)	O(*L*23)
O(*L*13)	1·586 (3)	2·437 (5)	2·529 (4)	2·460 (4)
O(*E*31)	105·2 (2)	1·481 (4)	2·543 (5)	2·478 (4)
O(*E*32)	111·4 (2)	118·7 (2)	1·474 (3)	2·522 (3)
O(*L*23)	101·6 (1)	107·6 (2)	110·8 (1)	1·588 (2)

P(1)—P(2)	2·902 (1)	P(2)—P(3)	2·940 (1)
P(1)—P(3)	3·029 (1)		

P(1)—O(*L*12)—P(2)	131·1 (1)	P(2)—P(1)—P(3)	94·90 (3)
P(1)—O(*L*13)—P(3)	145·9 (2)	P(1)—P(2)—P(3)	140·63 (3)
P(2)—O(*L*23)—P(3)	135·5 (1)	P(1)—P(3)—P(2)	98·46 (3)

P(1)—Li	2·999 (8)	P(3)—Li	3·232 (7)
P(2)—Li	3·279 (6)		

Ag(1) polyhedron

Ag(1)—O(*E*11)	2·318 (3)	Ag(1)—O(*E*31)	2·592 (3)
Ag(1)—O(*E*12)	2·391 (3)	Ag(1)—O(*E*32)	2·554 (3)
Ag(1)—O(*E*22)	2·526 (2)		

Ag(2) polyhedron

Ag(2)—O(*E*12)	2·799 (3)	Ag(2)—O(*E*21)	2·502 (2)
Ag(2)—O(*E*12)	2·466 (2)	Ag(2)—O(*E*22)	2·597 (2)
Ag(2)—O(*E*31)	2·691 (4)	Ag(2)—O(*W*)	2·386 (2)

LiO_4 tetrahedron

Li—O(*E*11)	1·960 (7)	Li—O(*W*)	2·006 (6)
Li—O(*E*32)	1·908 (8)	Li—O(*E*22)	1·931 (6)

O—H···O	O—H	H···O	O—O	O—H—O
O(*W*)—H(1)···O(*E*21)	0·81 (8)	1·97 (8)	2·718 (4)	154 (6)
O(*W*)—H(2)···O(*E*31)	0·73 (6)	2·24 (6)	2·801 (3)	134 (6)

H(1)—O(*W*)—H(2) 95 (8)

Discussion. Table 1 reports the final atomic coordinates.* The P_6O_{18} phosphoric ring anion is centrosymmetric, located around the inversion centre at 0,0,0. The main geometrical features do not depart significantly from what is commonly observed for such anions with $\bar{1}$ internal symmetry. The interatomic distances and bond angles are reported in Table 2. The Li atom has an almost regular tetrahedral coordination built up by three O atoms and one water molecule. The Li—O distances vary from 1·908–2·006 Å (Table 2) while the O—Li—O angles range between 97·3 and 114·6° with an average O—P—O value of 109·3°. These LiO_4 tetrahedra share their three O atoms with three different P_6O_{18} ring anions to build a tetrahedral layer parallel to the *ab* plane. Each P_6O_{18} group shares six of its external O atoms with its six adjacent LiO_4 tetrahedra. Such a layer, in a projection along the *c* axis, is given in Fig. 1. All Ag atoms are situated between these layers. In Fig. 2 the projection of the atomic arrangement along the *b* axis shows clearly this layer organization. Within a range of 3 Å the Ag(1) atom

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53866 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

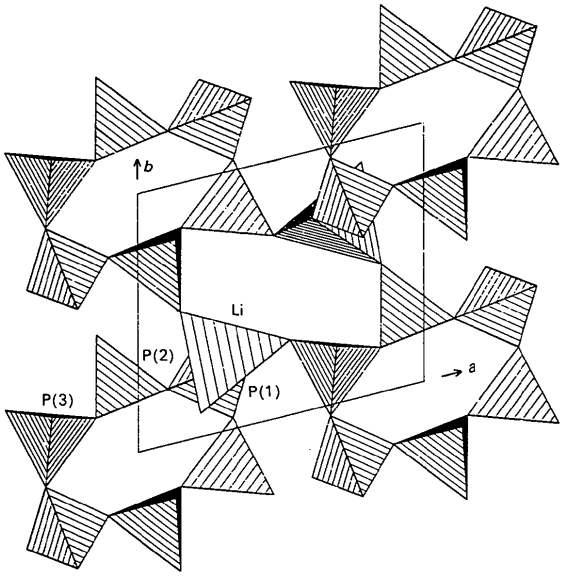

Fig. 1. Projection along the *c* axis of a layer of P_6O_{18} groups and LiO_4 tetrahedra. Ag atoms are not represented. Projection made for $-0·45 < z < 0·45$.

has five O-atom neighbours while Ag(2) has a sixfold coordination made by five O atoms and one water molecule. Inside the Ag-atom layer the shortest Ag—Ag distances are 3·140 for Ag(1)—Ag(1) and 3·304 Å for Ag(1)—Ag(2).

The main features of the hydrogen-bond scheme are described in Table 2. The drawings were prepared using the *STRUPLO* (Fischer, 1985) program.

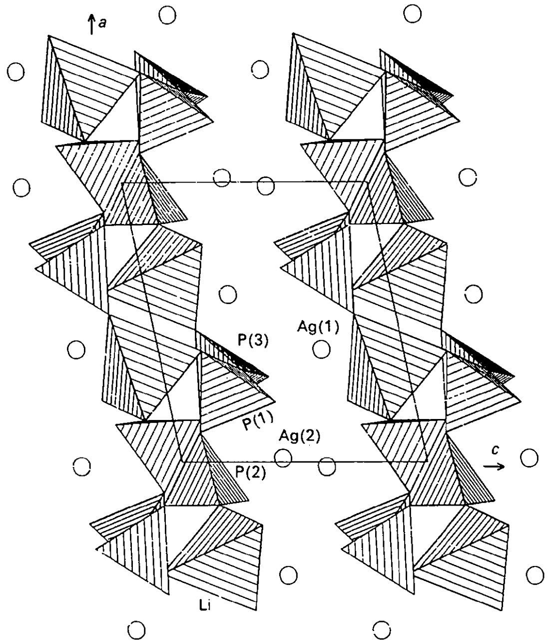

Fig. 2. Projection of the atomic arrangement of $Ag_4Li_2P_6O_{18}.2H_2O$ along the *b* axis.

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Layer Structure of $[CoCl(H_2PO_2)].H_2O$

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Abstract. Chloro(phosphinato)cobalt(II) monohydrate, $M_r = 177·39$, orthorhombic, *Pbca*, $a = 7·416\,(4)$, $b = 13·082\,(7)$, $c = 9·483\,(5)$ Å, $V = 920\,(1)$ Å^3, $Z = 8$, $D_x = 2·56$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 4·48$ mm^{-1}, $F(000) = 696$, room temperature, $R = 0·025$ for 1698 unique observed reflections; crystal growth by slow evaporation from an aqueous solution of $CoCl_2.6H_2O$ and H_3PO_2. Layers are built up from distorted $[CoO_3Cl_2(H_2O)]^{6-}$ octahedra, with Cl atoms in *cis* positions, sharing an edge of two O atoms, forming dimers connected together by corners (Cl atoms only). The $(H_2PO_2)^-$ tetrahedra connect two octahedra dimers and their O atoms are directed towards the next layer. The water