Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.*

Related literature. The results of a study of divalent manganese phosphates are reported in a recent paper (Cudennec, Riou & Gerault, 1986). Several well crystallized phases were prepared, mainly manganese hydrogenphosphates. Little is known about such solid-phase structures. Recently we have published the structural determination of a new manganese phosphate hydrogenphosphate: $Mn_7(PO_4)_2[PO_3(OH)]_4$ (Riou, Cudennec & Gerault, 1987), and refined the structure of synthetic hureaulite of pure manganese: $Mn_5(PO_4)_2[PO_3(OH)]_2.4H_2O$ (Gerault, Riou & Cudennec, 1987). The isomorphism of $Mn[PO_3(OH)].3H_2O$ with $Mg[PO_3(OH)].3H_2O$ (newberyite) was first established by Durif (1971).

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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 51833 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Cobalt(II) Orthophosphate Octahydrate

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(*Received* 8 *February* 1989; *accepted* 15 *March* 1989)

Abstract. $Co_3(PO_4)_2.8H_2O$, $M_r = 510{\cdot}86$, monoclinic, $C2/m$, $a = 10{\cdot}021\ (3)$, $b = 13{\cdot}331\ (3)$, $c = 4{\cdot}673\ (2)$ Å, $\beta = 104{\cdot}90\ (6)°$, $V = 603{\cdot}3\ (0{\cdot}8)$ Å^3, $D_x = 2{\cdot}812\ (4)$, $D_m = 2{\cdot}78\ (2)$ Mg m^{-3}, $Z = 2$, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 4{\cdot}425$ mm^{-1}, $F(000) = 510$, room temperature, $R = 0{\cdot}018$ for 778 observed reflections. The structure is isotypic with vivianite: $Fe_3(PO_4)_2.8H_2O$.

Experimental. Crystals of $Co_3(PO_4)_2.8H_2O$ were obtained from a mixture of cobalt carbonate, phosphoric acid and water. After elimination of carbon dioxide, the reacting system belongs to the ternary system CoO,P_2O_5,H_2O. A system composed of 8% CoO, 10% P_2O_5, 82% H_2O (in weight) was slowly dehydrated at room temperature for several months. Pink rectangular plate-like crystals appeared on the edge of the reactor bottle. A suitable crystal (0·18 × 0·16 × 0·05 mm) was mounted on an Enraf–Nonius CAD-4 diffractometer using Mo $K\alpha$ radiation. Density measured by flotation. The unit-cell constants were determined and refined on the basis of 25 carefully centred reflections in the angular range $2 < 2\theta < 30°$. Data were collected in the ω–2θ scan mode with ω-scan width $(2{\cdot}00 + 0{\cdot}50\mathrm{tg}\theta)°$ and slit amplitude $(1{\cdot}00 + 0{\cdot}35\mathrm{tg}\theta)$mm. A total of 1022 reflections were measured. The h,k,l range was $0 \le h \le 13$, $0 \le k \le 18$, $-6 \le l \le 6$ ($2\theta < 56°$). A periodic check of three standard reflections showed no significant intensity variation. The internal consistency index, R_{int}, was 0·016. Reflections were merged to a unique set of 890. According to $I > \sigma(I)$, 778 reflections were

Table 1. *Final atomic coordinates and equivalent isotropic temperature factors for* $Co_3(PO_4)_2.8H_2O$

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

	x	y	z	B_{eq}(Å^2)
Co1	0·000	0·000	0·000	0·664 (6)
Co2	0·000	0·38993 (2)	0·000	0·656 (4)
P	0·31424 (5)	0·000	0·3811 (1)	0·546 (8)
O1	0·1573 (1)	0·000	0·3711 (3)	0·84 (2)
O2	0·3935 (2)	0·000	0·7092 (3)	0·78 (2)
O3	0·3444 (1)	0·09648 (8)	0·2282 (2)	0·81 (2)
O4(*w*)	0·4009 (1)	0·38454 (9)	0·1956 (2)	1·07 (2)
O5(*w*)	0·1029 (1)	0·2760 (1)	0·2820 (3)	1·36 (2)
H1(O4)	0·380 (3)	0·411 (2)	0·319 (6)	2·0*
H2(O4)	0·348 (4)	0·372 (2)	0·123 (6)	2·0*
H3(O5)	0·116 (4)	0·299 (2)	0·382 (7)	2·0*
H4(O5)	0·058 (3)	0·221 (2)	0·286 (7)	2·0*

* Atoms refined isotropically.

regarded as observed. Data were corrected for Lorentz and polarization effects and absorption corrections were applied. The structural determination was carried out on a PDP 11/60 computer using the *SDP* package (Frenz, 1978). Scattering factors were from Cromer & Waber (1965). The structure was determined on the basis of isomorphism with $Mg_3(PO_4)_2.8H_2O$. Refinement was started with atomic coordinates reported by Tagaki, Mathew & Brown (1986). The four H atoms were refined isotropically. Full-matrix least-squares refinement was based on *F* and the function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w(F) = 1/\sigma^2(F)$. The weighting scheme had a non-Poisson contribution with $p =$ 0·050. Corrections were made for anomalous dispersion and secondary-extinction effects ($g = 3·6 \times 10^{-7}$). During the last refinement cycle, the r.m.s. shift/e.s.d. was 0·39. The final difference Fourier map showed no features higher than 0·78 e Å^{-3}. In these conditions, the final $R = 0·018$ and $wR = 0·026$. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.*

Related literature. Little is known about cobalt phosphate hydrates. In a recent study (Cudennec, Lecerf, Riou & Gerault, 1987), we prepared crystals of $Co_3(PO_4)_2.8H_2O$. This solid phase belongs to the large vivianite family: $M^{II}_3(XO_4)_2.8H_2O$ with M^{II} = Fe, Co, Ni, Zn, Mg and $XO_4 = PO_4$, AsO_4. The structure of vivianite: $Fe_3(PO_4)_2.8H_2O$, was proposed by Mori & Ito (1950), without refinement of the atomic coordinates. More recently Hill (1979) has determined the structure of köttigite: $Zn_3(AsO_4)_2.8H_2O$, which was refined with the H atoms. Lastly, Tagaki *et al.* (1986) have determined the structure of $Mg_3(PO_4)_2.8H_2O$.

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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 51834 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of $Cs_3Mo_{15}Se_{17}$

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(*Received* 16 *April* 1989; *accepted* 11 *May* 1989)

Abstract. Caesium molybdenum selenide, $Cs_3Mo_{15}Se_{17}$, $M_r = 3180·14$, hexagonal, $P6_3/m$, $a = 9·624$ (2), $c = 20·898$ (8) Å, $V = 1676·4$ (7) Å^3, $Z = 2$, $D_x = 6·276$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71073$ Å, $\mu = 266·5$ cm^{-1}, $F(000) = 2746$, $T = 293$ K, $R = 0·028$ for 1028 reflections with $I \geq 2\sigma(I)$ and 58 parameters. The title compound is isostructural with $Rb_3Mo_{15}Se_{17}$ and thus its structure contains as the main building block the $Mo_{15}Se_{17}$ cluster unit whose Mo core is built up by four face-sharing Mo_6 octahedra. Whereas the intracluster Mo—Mo distances as well as the Mo—Se ones are identical to within ±0·01 Å in both compounds, owing to the same cationic charge transfer towards the $Mo_{15}Se_{17}$ unit, a slight lengthening of the intercluster Mo—Mo distance from 3·268 to 3·318 Å is observed, as expected, when the size of the cation increases.

Experimental. Single crystals were obtained by heating a mixture of $CsMo_3Se_3$ and Mo_3Se_4 (3:2 ratio) in a sealed molybdenum crucible at about 2193 K for one hour and then cooling at 100 K h^{-1} to 1273 K.

Intensities were measured from a crystal fragment with dimensions 0·06 × 0·10 × 0·12 mm on an Enraf–Nonius CAD-4 diffractometer operating with graphite-monochromatized Mo $K\alpha$ radiation. Accurate cell parameters were obtained by a least-squares refinement of the setting angles of 25 reflections with $7 \leq \theta \leq 15°$. 5366 reflections were recorded over the range $1 \leq \theta \leq 35°$ with h: $-15 \rightarrow 0$, k: $0 \rightarrow 15$, l: $0 \rightarrow$

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