SHORT-FORMAT PAPERS

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Manganese(II) Hydrogenphosphate Trihydrate

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Abstract. $Mn[PO_3(OH)].3H_2O$, M_r = 204·96, orthorhombic, *Pbca*, *a* = 10·408 (3), *b* = 10·863 (3), *c* = 10·192 (4) Å, *V* = 1152·3 (1·1) Å^3, D_x = 2·353 (3) Mg m^{-3}, *Z* = 8, Mo *Kα*, λ = 0·71073 Å, μ = 2·440 mm^{-1}, *F*(000) = 824, room temperature, *R* = 0·022 for 1120 observed reflections. The structure is isotypic with newberyite: $Mg[PO_3(OH)].3H_2O$.

Experimental. Crystals of $Mn[PO_3(OH)].3H_2O$ were obtained from a mixture of manganese carbonate, phosphoric acid and water. After elimination of carbon dioxide, the reacting system belongs to the ternary system MnO,P_2O_5,H_2O. A mixture containing 8% MnO, 10% P_2O_5, 82% H_2O (in weight) was slowly dehydrated at room temperature, without stirring. Colourless flattened octahedral crystals appeared, of average size 0·2 mm. A suitable crystal (0·2 × 0·2 × 0·3 mm) was mounted on an Enraf–Nonius CAD-4 diffractometer using Mo *Kα* radiation. The unit-cell constants were determined and refined on the basis of 25 carefully centred reflections in the angular range $0 < 2\theta < 30°$. Data were collected in the ω–2θ scan mode with ω-scan width (2·00 + 0·50tgθ)° and slit amplitude (1·00 + 0·35tgθ) mm. A total of 1621 reflections were measured. The *h,k,l* range was $0 \leq h \leq 13$, $0 \leq k \leq 14$, $0 \leq l \leq 13$ ($2\theta < 56°$). A periodic check of three standard reflections showed no significant intensity variation. Reflections were merged to a set of 1385. According to $I > \sigma(I)$, 1120 reflections were 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 has been studied on the basis of isomorphism with $Mg[PO_3(OH)].3H_2O$, whose structure was determined by Sutor (1967), and refined with the H atoms by Abbona, Boistelle & Haser (1979). Refinement was started with atomic parameters reported by Abbona *et al.* (1979). The seven 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* = 1·29 × 10^{-7}). During the last refinement cycle, the r.m.s. shift/e.s.d. was 0·07. The final difference Fourier map showed no features higher than 0·39 e Å^{-3}. In these conditions, the final *R* was 0·022 and *wR* = 0·033.

Table 1. *Final atomic coordinates and equivalent isotropic temperature factors for* $Mn[PO_3(OH)].3H_2O$

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

	x	*y*	*z*	B_{eq}(Å^2)
Mn	0·29919 (3)	0·24726 (3)	0·08836 (3)	0·953 (5)
P	−0·00753 (5)	0·13224 (4)	0·15078 (5)	0·797 (8)
O1	−0·0872 (2)	0·2018 (1)	0·2480 (1)	1·60 (3)
O2	−0·0433 (1)	0·1595 (1)	0·0077 (1)	1·21 (3)
O3	0·1371 (1)	0·1500 (1)	0·1698 (1)	1·23 (2)
O4	−0·0356 (1)	−0·0098 (1)	0·1766 (2)	1·39 (3)
O5(*w*)	0·1637 (2)	0·3186 (2)	−0·0625 (2)	1·91 (3)
O6(*w*)	0·2495 (2)	0·4195 (1)	0·1929 (2)	1·88 (3)
O7(*w*)	0·3610 (2)	0·0906 (1)	−0·0354 (2)	2·50 (4)
H1(O5)	0·169 (4)	0·324 (3)	−0·140 (3)	3·0*
H2(O5)	0·107 (4)	0·294 (3)	−0.055 (4)	3·0*
H3(O6)	0·290 (3)	0·487 (4)	0·181 (4)	3·0*
H4(O6)	0·187 (3)	0·435 (3)	0·226 (4)	3·0*
H5(O7)	0·370 (4)	0·112 (3)	−0·107 (3)	3·0*
H6(O7)	0·352 (4)	0·018 (3)	−0·029 (4)	3·0*
H7(O4)	−0·015 (3)	−0·047 (3)	0·113 (4)	3·0*

* Atoms refined isotropically.

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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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Abstract. $Co_3(PO_4)_2.8H_2O$, $M_r = 510·86$, monoclinic, $C2/m$, $a = 10·021$ (3), $b = 13·331$ (3), $c = 4·673$ (2) Å, $\beta = 104·90$ (6)°, $V = 603·3$ (0·8) Å^3, $D_x = 2·812$ (4), $D_m = 2·78$ (2) Mg m^{-3}, $Z = 2$, Mo $K\alpha$, $\lambda = 0·71073$ Å, $\mu = 4·425$ mm^{-1}, $F(000) = 510$, room temperature, $R = 0·018$ 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 $(2·00 + 0·50\text{tg}\theta)°$ and slit amplitude $(1·00 + 0·35\text{tg}\theta)$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.

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