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Crystallographic Studies of the Farringtonite-type Phases γ-$Zn_2Co(PO_4)_2$ and γ-$(Zn_{0.50}Co_{0.50})_3(PO_4)_2$

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Abstract

The two solid solutions γ-$Zn_2Co(PO_4)_2$ and γ-$(Zn_{0.50}Co_{0.50})_3(PO_4)_2$ have been prepared and equilibrated at 1070 K. The structures ($P2_1/n$, $Z=2$) are isomorphous with 'γ-$Zn_3(PO_4)_2$' and with the mineral *farringtonite*, (Mg, Fe, Mn, Ca)$_3$(PO$_4$)$_2$, with $M_r = 379{\cdot}61$, $a = 7{\cdot}536\,(1)$, $b = 8{\cdot}413\,(1)$, $c = 5{\cdot}049\,(1)$ Å, $\beta = 94{\cdot}74\,(1)°$, $V = 319{\cdot}0\,(1)$ Å^3, $D_x = 3{\cdot}952$ Mg m^{-3}, and $M_r = 376{\cdot}40$, $a = 7{\cdot}545\,(2)$, $b = 8{\cdot}406\,(2)$, $c = 5{\cdot}054\,(2)$ Å, $\beta = 94{\cdot}56\,(2)°$, $V = 319{\cdot}4\,(2)$ Å^3, $D_x = 3{\cdot}914$ Mg m^{-3}, respectively. The crystal structures have been refined on the basis of neutron powder diffraction data ($\lambda \simeq 1{\cdot}55$ Å, $T = 295$ K) with the Rietveld full-profile refinement technique, to $R_p = 0{\cdot}078$ and $0{\cdot}066$ ($R_I = 0{\cdot}052$ and $0{\cdot}036$). The metal cations are strongly ordered, with Zn^{2+} dominating at the five- and Co^{2+} at the six-coordinated sites; $K_D(\mathrm{Zn, Co}) \simeq 9$. The MO_5 polyhedra are distorted trigonal bipyramids, while the MO_6 octahedra are almost regular. Some thermodynamical calculations are included. The JCPDS Diffraction File No. for γ-$Zn_2Co(PO_4)_2$ is 34-1491.

Introduction

As part of a project concerning the crystal chemistry of metal phosphates (*cf.* Nord & Kierkegaard, 1980), investigations with the aim of determining cation distributions between five- and six-coordinated sites have been undertaken. Many suitable reference structures are found among divalent-metal phosphate minerals, such as the *farringtonite*, (Mg, Fe, Mn, Ca)$_3$(PO$_4$)$_2$, structure, isomorphous with 'γ-$Zn_3(PO_4)_2$' (Calvo, 1963), and the *graftonite*, (Fe, Mn, Ca)$_3$(PO$_4$)$_2$, structure (Calvo, 1968; Kostiner & Rea, 1974).

Numerous farringtonite-type γ-$(Zn_{1-z}M_z)_3(PO_4)_2$ solid solutions may be prepared (Sarver, Katnack & Hummel, 1959; Brown & Hummel, 1963; Nord & Stefanidis, 1981), and some cation distribution studies have now been completed constituting the first systematic study of partitioning among five- and six-coordinated sites for M^{2+} ions. Various techniques have been used, such as X-ray diffraction (*e.g.* Nord, 1977), ^{57}Fe Mössbauer spectroscopy (Annersten, Ericsson & Nord, 1980), or Rietveld (1969) refinements

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based on neutron powder diffraction data (*e.g.* Nord & Stefanidis, 1982). Additional studies of two farringtonite-type γ-$(Zn, Co)_3(PO_4)_2$ solid solutions by means of Rietveld neutron-data refinements are reported here.

Experimental

Pure zinc and cobalt(II) orthophosphates were first prepared as described by Nord & Kierkegaard (1980). These two compounds are almost completely soluble in each other (Nord & Stefanidis, 1981). Mixtures of nominal stoichiometric composition γ-$Zn_2Co(PO_4)_2$ and γ-$(Zn_{0\cdot50}Co_{0\cdot50})_3(PO_4)_2$ were heated in open platinum crucibles at 1070 K [800(10)°C] for one month, quenched and ground once every week to ensure complete homogeneity and equilibrium, and finally quenched in liquid nitrogen.

The monoclinic ($P2_1/n$) unit-cell dimensions were determined by use of X-ray powder diffraction data, obtained at 295 K with an XDC-700 Guinier–Hägg-type focusing camera (Cr $K\alpha_1$ radiation, $\lambda = 2\cdot28975$ Å, quartz-crystal monochromator, KCl internal standard). In each cell-parameter refinement only one weak reflection out of 25 remained unindexed, so the samples were judged to have a purity of at least 98%. Neutron powder diffraction data were collected at the Studsvik (Nyköping, Sweden) R2 nuclear research reactor from 3 cm^3 ($\sim$10 g) of each powdered sample, kept in a thin-walled cylindrical vanadium capsule. A modified Hilger–Watts diffractometer, controlled by a PDP-8 computer, and a ^{3}He detector were used to collect data at 295 K for $5 \leq \theta \leq 40°$ ($\Delta\theta = 0\cdot04°$) with an effective scan time of about 20 min per step. The average flux was 10^{10} neutrons $m^{-2} s^{-1}$ for $\lambda \simeq 1\cdot55$ Å; the wavelength was selected with a double monochromator in parallel setting (copper crystals). Experiments showed that the absorption effects were negligible. Furthermore, a check of the samples with a JEOL JSM-35 scanning electron microscope indicated that no serious orientation effects were to be expected.

Structure refinements

Since the technique employed here has already been described in detail for related structures (*e.g.* Nord & Stefanidis, 1982; Nord, 1984), it is only briefly outlined below. The solid solution γ-$Zn_2Co(PO_4)_2$ was investigated first. The neutron intensity profile contained 180 independent partly overlapping Bragg reflections. The background was graphically determined and subtracted to give net intensities, which were processed with Rietveld's (1969) full-profile refinement technique, modified by S. Åsbrink and A. G. Nord (*cf.* also Albinati & Willis, 1982; Scott, 1983). A cation distribution parameter x was introduced according to the formula

$$\gamma\text{-}(Zn_{1-x}Co_x)_2^{M(1)}(Zn_{2x}Co_{1-2x})^{M(2)}(PO_4)_2 \quad (0 \leq x \leq 0\cdot50).$$

$M(1)$ is the five- and $M(2)$ the six-coordination site. After some trial refinements, the complete structure was refined with one scale factor, three peak-profile parameters, 18 atomic positional parameters, and three isotropic temperature factors (for metals, phosphorus, and oxygens). The neutron scattering lengths were taken from *International Tables for X-ray Crystallography* (1974). The value of x was determined through a series of refinements with systematically shifted x values, and the R_I values plotted *versus* x ($R_I = \sum|I_{\rm obs} - I_{\rm calc}|/\sum|I_{\rm obs}|$ for integrated reflections). A minimum of the $R_I(x)$ curve was afterwards located, through interpolation, at $x = 0\cdot18 \pm 0\cdot01$, for which value the final refinement was carried out ($R_I = 0\cdot052$, $R_p = 0\cdot078$, $R_{wp} = 0\cdot085$). For comparison, the R_I values obtained for $x = 0$ and $x = 0\cdot50$ (the end values) were 0·12 and 0·15, respectively. Since a random distribution of the cations is obtained for $x = 1/3$, the present value ($x = 0\cdot18$) indicates a fairly strong ordering of the cations with Zn^{2+} concentrated at the five-coordinated $M(1)$ sites.

This refinement technique has worked well for previously studied farringtonite-type phases as well as for three other structure types (sarcopsides, olivines and tetrametaphosphates). Moreover, the cation distribution results of four iron-containing solid solutions agree within statistical significance with results obtained from Mössbauer spectroscopy (*cf.* Nord, 1984). In these studies the x value giving a minimum of the $R_p(x)$ curve was equivalent within one standard deviation to that of the corresponding $R_I(x)$ curve; this was also the case for γ-$Zn_2Co(PO_4)_2$. γ-$(Zn_{0\cdot50}Co_{0\cdot50})_3(PO_4)_2$ was refined in a similar way, again with 180 independent reflections. The cation distribution pattern was in this case defined by the expression

$$\gamma\text{-}(Zn_{0\cdot75-x}Co_{0\cdot25+x})_2^{M(1)}(Zn_{2x}Co_{1-2x})^{M(2)}(PO_4)_2 \quad (0 \leq x \leq 0\cdot50).$$

The refinements converged at $x = 0\cdot085 \pm 0\cdot005$, with $R_I = 0\cdot036$, $R_p = 0\cdot066$ and $R_{wp} = 0\cdot073$. For x equal to 0 and 0·50, the R_I values were 0·058 and 0·14, respectively. The atomic parameters are given in Table 1. The observed and calculated intensity profiles are shown in Fig. 1. The poor agreement at the end of the profile is due to termination errors. No region has been excluded.*

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* The raw intensity data and the observed and calculated integrated intensities have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38862 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic positional parameters* ($\times 10^3$) *and isotropic temperature factors for the two* Zn,Co-*farringtonite phases*

Space group $P2_1/n$, No. 14 [$\pm(x, y, z)$; $\pm(\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z)$]. The e.s.d.'s are given in parentheses.

		γ-$Zn_2Co(PO_4)_2$	γ-$(Zn_{0.50}Co_{0.50})_3(PO_4)_2$
$M(1)$	x	617 (1)	617 (1)
	y	141 (1)	141 (1)
	z	79 (2)	85 (1)
$M(2)$	x	0	0
	y	0	0
	z	500	500
P	x	197 (1)	198 (1)
	y	194 (1)	193 (1)
	z	32 (2)	32 (1)
O(1)	x	49 (1)	52 (1)
	y	135 (1)	136 (1)
	z	825 (2)	827 (1)
O(2)	x	126 (1)	126 (1)
	y	197 (1)	193 (1)
	z	307 (2)	313 (1)
O(3)	x	257 (1)	255 (1)
	y	361 (1)	364 (1)
	z	939 (2)	941 (1)
O(4)	x	363 (1)	360 (1)
	y	78 (1)	80 (1)
	z	48 (2)	47 (1)
$B(M)$(Å^2)		1·0 (2)	1·1 (2)
B(P)(Å^2)		0·6 (3)	0·4 (2)
B(O)(Å^2)		0·3 (1)	0·6 (1)

Table 2. *Some interatomic distances* (Å) *and angles* (°)

	γ-$Zn_2Co(PO_4)_2$	γ-$(Zn_{0.50}Co_{0.50})_3(PO_4)_2$
M (1)–O (1)	2·34 (2)	2·30 (1)
M (1)–O (2)	1·94 (2)	1·96 (1)
M (1)–O (3)	2·03 (2)	2·01 (1)
M (1)–O (4)	1·98 (2)	2·00 (1)
M (1)–O (4^i)	1·96 (2)	1·98 (1)
M (1)–O mean	2·05 (2)	2·05 (1)
M (2)–O (1) (×2)	2·01 (1)	2·03 (1)
M (2)–O (2) (×2)	2·19 (1)	2·14 (1)
M (2)–O (3) (×2)	2·18 (1)	2·18 (1)
M (2)–O mean	2·13 (1)	2·12 (1)
P–O (1)	1·55 (2)	1·53 (1)
P–O (2)	1·53 (2)	1·56 (1)
P–O (3)	1·55 (2)	1·57 (1)
P–O (4)	1·58 (2)	1·55 (1)
P–O mean	1·55 (2)	1·55 (1)
O (1)–P–O (2)	109·0 (7)	109·5 (5)
O (1)–P–O (3)	107·2 (7)	106·8 (5)
O (1)–P–O (4)	111·0 (7)	111·1 (5)
O (2)–P–O (3)	112·9 (7)	113·2 (5)
O (2)–P–O (4)	107·4 (7)	106·4 (5)
O (3)–P–O (4)	109·2 (7)	110·0 (5)
O–P–O mean	109·5 (7)	109·5 (5)

Symmetry code: (i) $1-x, -y, -z$.

Discussion

Structural features

The structure of γ-$(Zn, Co)_3(PO_4)_2$ is built of somewhat distorted $M(1)O_5$ trigonal bipyramids and almost regular $M(2)O_6$ octahedra and PO_4 tetrahedra. An illustration of the crystal structure has been given by Nord & Stefanidis (1982). The two distinct metal–oxygen polyhedra are shown in Fig. 2. Some interatomic distances and angles in the two Zn, Co-phases studied here are given in Table 2. Since Zn^{2+} and Co^{2+} are almost equi-sized, the partial concentration of cobalt at the $M(2)$ sites does not significantly change the averaged metal–oxygen distances from the values observed in 'γ-$Zn_3(PO_4)_2$'; the $M(1)$–O and $M(2)$–O mean values in the latter phase are 2·05 and 2·15 Å, respectively (Calvo, 1973). Moreover, the cell volumes of the two Zn, Co-phases are very similar to each other [319·0 (1) and 319·4 (2) Å^3] and to 'pure' (extrapolated value) γ-$Zn_3(PO_4)_2$, with $V = 319{\cdot}7$ Å^3 (Nord & Stefanidis, 1981). The P–O distances are identical within statistical significance, and so are the O–P–O angles.

Cation distributions and thermodynamical considerations

One of the main purposes of this study was to evaluate the distribution of the cations among the five- and six-coordinated sites. The intracrystalline Zn^{2+}/M^{2+} cation exchange in a γ-$(Zn_{1-z}M_z)_3(PO_4)_2$ solid solution is conventionally (Calvo, 1963)

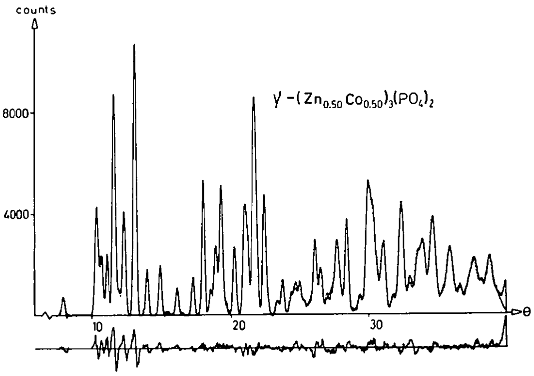

Fig. 1. The least-squares fit obtained between the observed intensities (continuous curve) and calculated intensities (points) for γ-$(Zn_{0.50}Co_{0.50})_3(PO_4)_2$ (neutron diffraction data). The discrepancy in the fit, defined as $I_{obs} - I_{calc}$, is plotted below on the same scale.

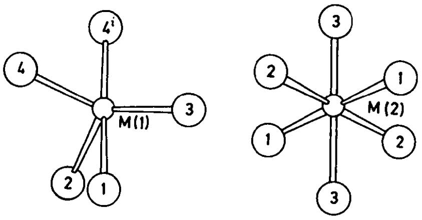

Fig. 2. The somewhat distorted trigonal bipyramid $M(1)O_5$ and the almost regular $M(2)O_6$ octahedron in γ-$(Zn, Co)_3(PO_4)_2$. The figure 1 refers to O(1), *etc.*

expressed by the equilibrium reaction

$$Zn^{2+}[M(2)]+M^{2+}[M(1)] \rightleftarrows Zn^{2+}[M(1)] + M^{2+}[M(2)]. \qquad (1)$$

The equilibrium constant at the temperature in question (T) is

$$K_{\rm eq}=\frac{X_{Zn'}f_{Zn'}X_{M''}f_{M''}}{X_{Zn''}f_{Zn''}X_{M'}f_{M'}},$$

where ′ stands for $M(1)$ and ″ for $M(2)$, $X_{Zn'}$ *etc.* are the site occupancy factors, and $f_{Zn'}$ *etc.* are the partial activity factors. Assuming ideal solid-solution conditions and equal activity factors, $(f_{Zn'}f_{M''})/(f_{Zn''}f_{M'})$ is equal to unity. Accordingly, the conventional cation distribution coefficient K_D is, roughly, equal to $K_{\rm eq}$, *i.e.*

$$K_D({\rm Zn}, M)=(X_{Zn'}X_{M''})/(X_{Zn''}X_{M'}).$$

For γ-$Zn_2Co(PO_4)_2$ the K_D value may be defined, utilizing the cation distribution parameter x, as

$$K_D({\rm Zn, Co})=(1-x)(1-2x)/2x^2.$$

Thus, $x=0\cdot18\pm0\cdot01$ gives $K_D({\rm Zn, Co})=8\cdot1$ ($T=1070$ K). Similarly, for γ-$(Zn_{0\cdot50}Co_{0\cdot50})_3(PO_4)_2$, with the evaluated cation distribution γ-$(Zn_{0\cdot665}Co_{0\cdot335})_2^{M(1)}(Zn_{0\cdot170}Co_{0\cdot830})^{M(2)}(PO_4)_2$, $K_D({\rm Zn, Co})$ is $9\cdot7[x=0\cdot085]$. In a previous study of γ-$({\rm Zn}, M)_3(PO_4)_2$ phases, it was noticed that the Zn,Co-phases very slightly disobeyed Vegard's law (Nord & Stefanidis, 1981). However, the present refinement results in terms of R factors and geometrical dimensions as well as the two rather similar $K_D({\rm Zn, Co})$ values obtained do not indicate any structural anomalies. The Gibbs free-energy change of (1) is calculated from

$$\Delta G^\circ=-RT\ln K_D.$$

Since $T=1070$ K, $\Delta G^\circ\simeq-19\ {\rm kJ\ mol^{-1}}$ for γ-$({\rm Zn, Co})_3(PO_4)_2$ (average value).

The cation distribution has been determined in some other γ-$({\rm Zn}, M)_3(PO_4)_2$ and $({\rm Mg}, M)_3(PO_4)_2$ solid solutions with the farringtonite structure. Thus, the value of $K_D({\rm Zn, Co})$ may also be *estimated* from thermodynamical considerations based on analogous K_D values. X-ray diffraction studies of γ-$Zn_2Mg(PO_4)_2$ phases have arrived at $K_D({\rm Zn, Mg})\simeq20$ at about 1300 K (Nord, 1977), *i.e.*

$$(X_{Zn'}X_{Mg''})/(X_{Zn''}X_{Mg'})\simeq20. \qquad (2)$$

Similar investigations of two $({\rm Mg, Co})_3(PO_4)_2$ solid solutions gave, at $T=1070$ K (Nord & Stefanidis, 1980):

$$(X_{Co'}X_{Mg''})/(X_{Co''}X_{Mg'})\simeq2\cdot2. \qquad (3)$$

Division of (2) by (3) gives $(X_{Zn'}X_{Co''})/(X_{Zn''}X_{Co'})=20/2\cdot2$, *i.e.* $K_D({\rm Zn, Co})\simeq9$. This value agrees very well with the $K_D({\rm Zn, Co})$ values obtained from the present neutron diffraction studies, indicating that the ideal solid-solution conditions assumed may be justified. It should be noted, though, that γ-$Zn_2Mg(PO_4)_2$ had been equilibrated at a higher temperature, so the good agreement is, partly, fortuitous.

Concluding remarks

The present results agree with an earlier preliminary sequence order for the M^{2+} preferences of $M(1)$ over $M(2)$ in the farringtonite-type structure, obtained from structural refinements and Mössbauer spectroscopy (*e.g.* Nord & Stefanidis, 1981, 1982; Annersten, Ericsson & Nord, 1980). Unfortunately, the divalent ions Cu^{2+}, Cd^{2+} or Ca^{2+} cannot be incorporated in γ-$Zn_3(PO_4)_2$ or $Mg_3(PO_4)_2$ in amounts large enough to enable a moderately accurate determination of the cation distribution from diffraction methods (Kreidler & Hummel, 1967; Nord & Stefanidis, 1981). However, work is in progress to *estimate* $K_D({\rm Zn, Cd})$ *etc.* from thermodynamical calculations based on Mössbauer results on ternary farringtonite-type solid solutions. Some preliminary results indicate that Cd^{2+}, as expected from size effects, has a very slight tendency for the five-coordinated $M(1)$ sites.

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