



Crystal structure of dimanganese(II) zinc bis[orthophosphate(V)] monohydrate

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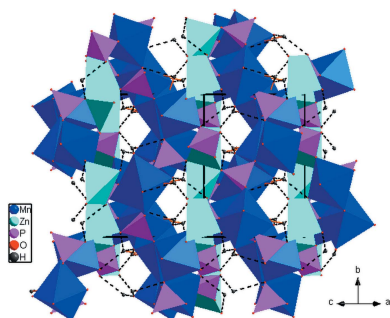
The title compound, $\text{Mn}_2\text{Zn}(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$, was obtained under hydrothermal conditions. The structure is isotopic with other transition metal phosphates of the type $M_{3-x}M'_x(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$, but shows no statistical disorder of the three metallic sites. The principal building units are distorted $[\text{MnO}_6]$ and $[\text{MnO}_5(\text{H}_2\text{O})]$ octahedra, a distorted $[\text{ZnO}_5]$ square pyramid and two regular PO_4 tetrahedra. The connection of the polyhedra leads to a framework structure. Two types of layers parallel to $(\bar{1}01)$ can be distinguished in this framework. One layer contains $[\text{Zn}_2\text{O}_8]$ dimers linked to PO_4 tetrahedra *via* common edges. The other layer is more corrugated and contains $[\text{Mn}_2\text{O}_8(\text{H}_2\text{O})_2]$ dimers and $[\text{MnO}_6]$ octahedra linked together by common edges. The PO_4 tetrahedra link the two types of layers into a framework structure with channels parallel to $[101]$. The H atoms of the water molecules point into the channels and form $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds (one of which is bifurcated) with framework O atoms across the channels.

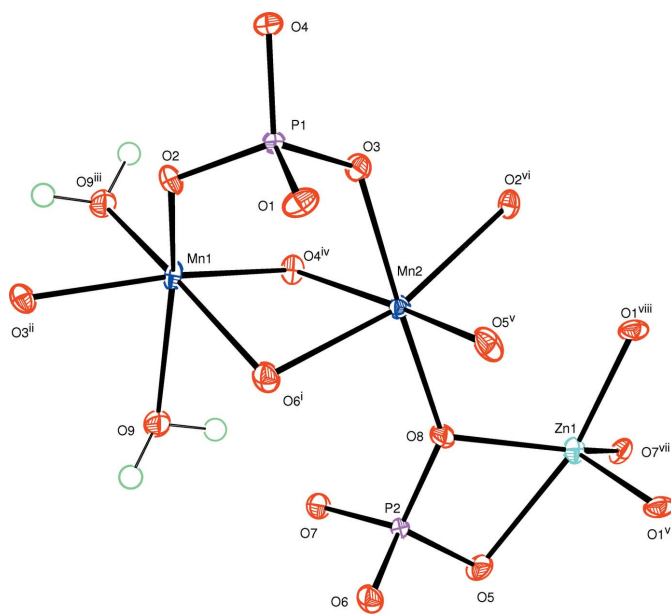
1. Chemical context

The great structural diversity of metal-based phosphates, associated with their physical properties makes this family of compounds interesting as potential functional materials, *e.g.* as catalysts (Viter & Nagorny, 2009; Weng *et al.*, 2009) or ion-exchangers (Jignasa *et al.*, 2006). Among the wide variety of metal phosphates, one of our interests is focused on mixed metallic orthophosphates of general formula $M_{3-x}M'_x(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$. The present communication reports the hydrothermal synthesis and structural characterization of a new member of this family, $\text{Mn}_2\text{Zn}(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$.

2. Structural commentary

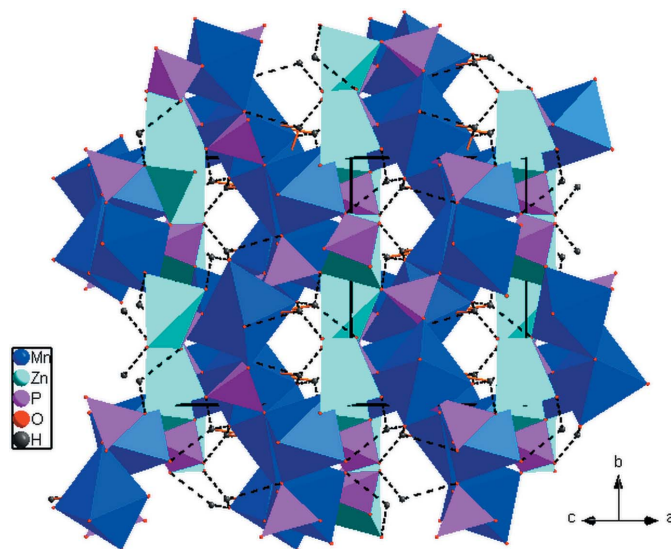
The structure of the title compound is built up from four different types of building units: $[\text{MnO}_6]$ and $[\text{MnO}_5(\text{H}_2\text{O})]$ octahedra, $[\text{ZnO}_5]$ square pyramids and PO_4 tetrahedra, as shown in Fig. 1. Whereas the $[\text{MnO}_6]$ octahedron is more or less regular with Mn—O distances in the range 2.1254 (13) to 2.2590 (13) Å, the $[\text{MnO}_5(\text{H}_2\text{O})]$ octahedron is significantly distorted with five equal Mn—O distances in the range 2.1191 (13) to 2.1556 (16) and one considerably longer Mn—O distance to the water ligand of 2.5163 (15) Å; the ZnO_5 square pyramid is also distorted with four shorter Zn—O distances between 1.9546 (13) and 2.0347 (12) Å and one longer Zn—O distance, likewise to the water O atom [2.3093 (14) Å]; the two PO_4 tetrahedra are rather regular [P—O distances between 1.5322 (13) and 1.5570 (13) Å; O—P—O angles between 102.92 (7) and 111.62 (8)°]. These polyhedra are arranged in such a way as to build up two types of layers parallel to $(\bar{1}01)$.




Figure 1

The principal building units in the structure of $\text{Mn}_2\text{Zn}(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are indicated by dashed lines. [Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $-x + 2, -y + 1, -z + 1$; (iv) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (v) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (vi) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (vii) $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$; (viii) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.]

One layer contains two $[\text{ZnO}_5]$ polyhedra linked together by edge-sharing into a $[\text{Zn}_2\text{O}_8]$ dimer that in turn is linked to PO_4 tetrahedra. The other layer contains dimers of the type $[\text{Mn}_2\text{O}_8(\text{H}_2\text{O})_2]$ (also formed by edge-sharing of two $[\text{MnO}_5(\text{H}_2\text{O})]$ octahedra), connecting $[\text{MnO}_6]$ octahedra and PO_4 tetrahedra through common vertices. The two types of layers are linked by common edges and vertices into a


Figure 2

Polyhedral representation of $\text{Mn}_2\text{Zn}(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ showing channels extending parallel to $[101]$. Hydrogen bonds are shown as dashed lines.

Table 1

 Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O9}-\text{H1} \cdots \text{O7}$	0.89	1.97	2.7866 (19)	151
$\text{O9}-\text{H2} \cdots \text{O5}^{\text{i}}$	0.91	2.16	2.8687 (19)	134
$\text{O9}-\text{H2} \cdots \text{O1}^{\text{ii}}$	0.91	2.48	3.0494 (19)	120

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$; (ii) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$.

framework structure with channels parallel to $[101]$. The water molecules of the $[\text{MnO}_5(\text{H}_2\text{O})]$ octahedra protrude into these channels and develop hydrogen bonds (one bifurcated) of medium-to-weak strength to framework O atoms across the channels (Fig. 2; Table 1).

The title compound adopts the $\text{Fe}_3(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ structure type (Moore & Araki, 1975) and is isotopic with various structures of general formula $M_{3-x}M'_x(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$: $\text{CuMn}_2(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ (Liao *et al.*, 1995); $\text{Co}_{2.59}\text{Zn}_{0.41}(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ (Sørensen *et al.*, 2005); $\text{Co}_{2.39}\text{Cu}_{0.61}(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ (Assani *et al.*, 2010); $\text{Mg}_{1.65}\text{Cu}_{1.35}(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ (Khmiyas *et al.* 2015).

3. Synthesis and crystallization

Crystals of $\text{Mn}_2\text{Zn}(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$ were obtained by hydrothermal treatment of zinc oxide (0.0406 g), metallic manganese (0.0824 g), phosphoric acid (0.1 ml) and 12.5 ml of distilled water, in a proportion corresponding to the molar ratio Zn: Mn: P = 1: 3: 3. The hydrothermal reaction was

Table 2

Experimental details.

Crystal data	$\text{Mn}_2\text{Zn}(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$
Chemical formula	383.21
M_r	Monoclinic, $P2_1/n$
Crystal system, space group	296
Temperature (K)	8.1784 (2), 10.1741 (2), 9.0896 (2)
a, b, c (\AA)	114.142 (1)
β ($^\circ$)	690.17 (3)
V (\AA^3)	4
Z	Mo $K\alpha$
Radiation type	7.54
μ (mm^{-1})	$0.32 \times 0.27 \times 0.19$
Crystal size (mm)	
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (SADABS; Bruker, 2009)
$T_{\text{min}}, T_{\text{max}}$	0.574, 0.748
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	11327, 2407, 2305
R_{int}	0.023
$(\sin \theta/\lambda)_{\text{max}}$ (\AA^{-1})	0.746
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.019, 0.052, 1.11
No. of reflections	2407
No. of parameters	127
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e \AA^{-3})	0.94, -0.84

Computer programs: APEX2 and SAINT (Bruker, 2009), SHELXS97 and SHELXL97 (Sheldrick, 2008), ORTEP-3 for Windows (Farrugia, 2012), DIAMOND (Brandenburg, 2006) and publCIF (Westrip, 2010).

conducted in a 23 ml Teflon-lined autoclave under autogenous pressure at 493 K for five days. After being filtered, washed with deionized water and dried in air, the reaction product consisted of two types of crystals, the first as off-white parallelepipeds corresponding to $\text{Mn}_7(\text{PO}_4)_2(\text{HPO}_4)_4$ (Riou *et al.*, 1987) and the second as colourless parallelepipeds corresponding to the title compound.

4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The O-bound H atoms were initially located in a difference map. In the last refinement cycle the distances were fixed at 0.89 and 0.91 Å, respectively, and the H atoms refined in the riding-model approximation with $U_{\text{iso}}(\text{H})$ set to $1.5U_{\text{eq}}(\text{O})$. The highest peak and the deepest hole in the final Fourier map are at 0.32 Å and 0.30 Å, respectively, from Mn1 and Zn1.

Acknowledgements

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supporting information

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Crystal structure of dimanganese(II) zinc bis[orthophosphate(V)] monohydrate

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Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Dimanganese(II) zinc bis[orthophosphate(V)] monohydrate

Crystal data

$\text{Mn}_2\text{Zn}(\text{PO}_4)_2 \cdot \text{H}_2\text{O}$
 $M_r = 383.21$
 Monoclinic, $P2_1/n$
 Hall symbol: -P 2yn
 $a = 8.1784$ (2) Å
 $b = 10.1741$ (2) Å
 $c = 9.0896$ (2) Å
 $\beta = 114.142$ (1)°
 $V = 690.17$ (3) Å³
 $Z = 4$

$F(000) = 736$
 $D_x = 3.688$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 2407 reflections
 $\theta = 2.8\text{--}32.0^\circ$
 $\mu = 7.54$ mm⁻¹
 $T = 296$ K
 Parallelepiped, off-white
 $0.32 \times 0.27 \times 0.19$ mm

Data collection

Bruker X8 APEX
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2009)
 $T_{\min} = 0.574$, $T_{\max} = 0.748$

11327 measured reflections
 2407 independent reflections
 2305 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$
 $\theta_{\max} = 32.0^\circ$, $\theta_{\min} = 2.8^\circ$
 $h = -12 \rightarrow 12$
 $k = -14 \rightarrow 15$
 $l = -13 \rightarrow 13$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.019$
 $wR(F^2) = 0.052$
 $S = 1.11$
 2407 reflections
 127 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0248P)^2 + 1.0141P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.94$ e Å⁻³
 $\Delta\rho_{\min} = -0.84$ e Å⁻³

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against all reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on all data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mn1	0.88638 (3)	0.35884 (3)	0.46580 (3)	0.00768 (6)
Mn2	0.48057 (3)	0.38305 (3)	0.21880 (3)	0.00726 (6)
Zn1	0.12609 (3)	0.62028 (2)	0.06179 (2)	0.00934 (6)
P1	0.70438 (5)	0.08456 (4)	0.32706 (5)	0.00553 (8)
P2	0.38560 (5)	0.67442 (4)	0.36388 (5)	0.00613 (8)
O1	0.58212 (17)	0.03301 (13)	0.40831 (15)	0.0102 (2)
O2	0.87050 (16)	0.15076 (13)	0.45546 (15)	0.0094 (2)
O3	0.59293 (17)	0.18429 (12)	0.19887 (15)	0.0092 (2)
O4	0.76145 (17)	−0.03217 (12)	0.25178 (15)	0.0092 (2)
O5	0.23736 (18)	0.77279 (13)	0.26723 (16)	0.0123 (2)
O6	0.36400 (17)	0.63194 (13)	0.51688 (15)	0.0104 (2)
O7	0.57269 (16)	0.73311 (13)	0.41028 (15)	0.0110 (2)
O8	0.35411 (17)	0.55914 (13)	0.24330 (15)	0.0107 (2)
O9	0.88135 (18)	0.58568 (14)	0.57419 (16)	0.0130 (2)
H1	0.7876	0.6203	0.4923	0.019*
H2	0.8793	0.5969	0.6731	0.019*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.00519 (11)	0.00776 (11)	0.00860 (11)	−0.00070 (8)	0.00129 (8)	0.00274 (8)
Mn2	0.00657 (11)	0.00704 (11)	0.00777 (11)	−0.00016 (8)	0.00254 (9)	−0.00025 (8)
Zn1	0.00839 (10)	0.00949 (10)	0.00884 (9)	0.00028 (6)	0.00219 (7)	0.00093 (6)
P1	0.00538 (16)	0.00594 (17)	0.00519 (16)	0.00046 (13)	0.00209 (13)	−0.00030 (13)
P2	0.00556 (16)	0.00676 (17)	0.00569 (16)	0.00008 (13)	0.00193 (13)	−0.00044 (13)
O1	0.0107 (5)	0.0111 (5)	0.0126 (5)	0.0015 (4)	0.0087 (5)	0.0025 (4)
O2	0.0079 (5)	0.0105 (5)	0.0073 (5)	−0.0020 (4)	0.0008 (4)	−0.0025 (4)
O3	0.0099 (5)	0.0080 (5)	0.0081 (5)	0.0022 (4)	0.0020 (4)	0.0013 (4)
O4	0.0083 (5)	0.0091 (5)	0.0099 (5)	0.0017 (4)	0.0033 (4)	−0.0027 (4)
O5	0.0121 (5)	0.0135 (6)	0.0108 (5)	0.0070 (5)	0.0041 (4)	0.0035 (4)
O6	0.0106 (5)	0.0137 (6)	0.0071 (5)	−0.0006 (4)	0.0038 (4)	0.0016 (4)
O7	0.0081 (5)	0.0129 (6)	0.0120 (5)	−0.0031 (4)	0.0041 (4)	−0.0030 (4)
O8	0.0107 (5)	0.0093 (5)	0.0099 (5)	0.0011 (4)	0.0021 (4)	−0.0035 (4)
O9	0.0109 (5)	0.0181 (6)	0.0106 (5)	0.0020 (5)	0.0051 (4)	0.0007 (5)

Geometric parameters (Å, °)

Mn1—O6 ⁱ	2.1191 (13)	Zn1—O1 ^{vi}	2.0242 (13)
Mn1—O2	2.1208 (14)	Zn1—O1 ^{viii}	2.0347 (12)
Mn1—O3 ⁱⁱ	2.1464 (12)	Zn1—O5	2.3093 (14)
Mn1—O9 ⁱⁱⁱ	2.1504 (14)	P1—O3	1.5327 (13)
Mn1—O4 ^{iv}	2.1556 (13)	P1—O4	1.5355 (13)
Mn1—O9	2.5163 (15)	P1—O2	1.5377 (13)
Mn2—O8	2.1254 (13)	P1—O1	1.5570 (13)
Mn2—O5 ^v	2.1533 (13)	P2—O7	1.5322 (13)
Mn2—O4 ^{iv}	2.1921 (13)	P2—O6	1.5340 (13)
Mn2—O2 ^{vi}	2.2126 (13)	P2—O5	1.5401 (13)
Mn2—O6 ⁱ	2.2166 (13)	P2—O8	1.5532 (13)
Mn2—O3	2.2590 (13)	O9—H1	0.8939
Zn1—O7 ^{vii}	1.9546 (13)	O9—H2	0.9131
Zn1—O8	2.0174 (13)		
O6 ⁱ —Mn1—O2	90.23 (5)	O4 ^{iv} —Mn2—O3	87.66 (5)
O6 ⁱ —Mn1—O3 ⁱⁱ	109.27 (5)	O2 ^{vi} —Mn2—O3	76.87 (5)
O2—Mn1—O3 ⁱⁱ	81.31 (5)	O6 ⁱ —Mn2—O3	87.34 (5)
O6 ⁱ —Mn1—O9 ⁱⁱⁱ	161.48 (5)	O7 ^{vii} —Zn1—O8	132.60 (5)
O2—Mn1—O9 ⁱⁱⁱ	107.27 (5)	O7 ^{vii} —Zn1—O1 ^{vi}	100.19 (6)
O3 ⁱⁱ —Mn1—O9 ⁱⁱⁱ	80.06 (5)	O8—Zn1—O1 ^{vi}	99.81 (5)
O6 ⁱ —Mn1—O4 ^{iv}	81.32 (5)	O7 ^{vii} —Zn1—O1 ^{viii}	117.98 (5)
O2—Mn1—O4 ^{iv}	118.14 (5)	O8—Zn1—O1 ^{viii}	107.48 (5)
O3 ⁱⁱ —Mn1—O4 ^{iv}	158.49 (5)	O1 ^{vi} —Zn1—O1 ^{viii}	80.41 (5)
O9 ⁱⁱⁱ —Mn1—O4 ^{iv}	84.99 (5)	O7 ^{vii} —Zn1—O5	87.57 (5)
O6 ⁱ —Mn1—O9	76.07 (5)	O8—Zn1—O5	67.61 (5)
O2—Mn1—O9	157.28 (5)	O1 ^{vi} —Zn1—O5	167.20 (5)
O3 ⁱⁱ —Mn1—O9	86.18 (5)	O1 ^{viii} —Zn1—O5	105.05 (5)
O9 ⁱⁱⁱ —Mn1—O9	89.00 (5)	O3—P1—O4	111.58 (7)
O4 ^{iv} —Mn1—O9	78.15 (5)	O3—P1—O2	110.68 (7)
O8—Mn2—O5 ^v	89.03 (5)	O4—P1—O2	109.99 (7)
O8—Mn2—O4 ^{iv}	98.10 (5)	O3—P1—O1	106.62 (7)
O5 ^v —Mn2—O4 ^{iv}	167.53 (5)	O4—P1—O1	108.79 (7)
O8—Mn2—O2 ^{vi}	104.14 (5)	O2—P1—O1	109.08 (7)
O5 ^v —Mn2—O2 ^{vi}	90.14 (5)	O7—P2—O6	109.42 (7)
O4 ^{iv} —Mn2—O2 ^{vi}	97.96 (5)	O7—P2—O5	111.62 (8)
O8—Mn2—O6 ⁱ	91.85 (5)	O6—P2—O5	110.15 (7)
O5 ^v —Mn2—O6 ⁱ	91.25 (5)	O7—P2—O8	110.32 (7)
O4 ^{iv} —Mn2—O6 ⁱ	78.36 (5)	O6—P2—O8	112.31 (8)
O2 ^{vi} —Mn2—O6 ⁱ	163.97 (5)	O5—P2—O8	102.92 (7)
O8—Mn2—O3	173.90 (5)	H1—O9—H2	114.6
O5 ^v —Mn2—O3	84.95 (5)		

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+1/2, -y+1/2, z+1/2$; (iii) $-x+2, -y+1, -z+1$; (iv) $-x+3/2, y+1/2, -z+1/2$; (v) $-x+1/2, y-1/2, -z+1/2$; (vi) $x-1/2, -y+1/2, z-1/2$; (vii) $x-1/2, -y+3/2, z-1/2$; (viii) $-x+1/2, y+1/2, -z+1/2$.

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O9—H1 \cdots O7	0.89	1.97	2.7866 (19)	151
O9—H2 \cdots O5 ^{ix}	0.91	2.16	2.8687 (19)	134
O9—H2 \cdots O1 ⁱⁱ	0.91	2.48	3.0494 (19)	120

Symmetry codes: (ii) $x+1/2, -y+1/2, z+1/2$; (ix) $x+1/2, -y+3/2, z+1/2$.