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Dilead(II) trimanganese(II) bis(hydrogenphosphate) bis(phosphate)

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Key indicators: single-crystal X-ray study; T = 296 K; mean σ(Pb–O) = 0.004 Å; R factor = 0.020; wR factor = 0.051; data-to-parameter ratio = 10.6.

The title compound, $Pb_2Mn_3(HPO_4)_2(PO_4)_2$, was synthesized by a hydrothermal method. All atoms are in general positions except for one Mn atom which is located on an inversion center. The framework of the structure is built up from PO_4 tetrahedra and two types of MnO_6 octahedra, one almost ideal and the other very distorted with one very long Mn—O bond [2.610 (4) Å compared an average of 2.161 Å for the other bonds]. The centrosymetric octahedron is linked to two distorted MnO_6 octahedra by an edge common, forming infinite zigzag Mn_3O_{14} chains running along the b axis. Adjacent chains are linked by PO_4 and $PO_3(OH)$ tetrahedra through vertices or by edge sharing, forming sheets perpendicular to [100]. The Pb^{2+} cations are sandwiched between the layers and ensure the cohesion of the crystal structure. O—H···O hydrogen bonding between the layers is also observed.

Related literature

For properties of phosphates and their potential applications, see: Gao & Gao (2005); Viter & Nagornyi (2009); Clearfield (1988); Trad *et al.* (2010). For compounds with related structures, see: Assani *et al.* (2010, 2011*a*,*b*,*c*, 2012); Effenberger (1999). For bond-valence analysis, see: Brown & Altermatt (1985).

Experimental

Crystal data

$Pb_2Mn_3(HPO_4)_2(PO_4)_2$
M_r = 961.10
Monoclinic, $P2_1/c$
a = 7.9449 (2) Å
b = 8.8911 (2) Å
c = 9.5718 (3) Å
β = 100.917 (2)°
V = 663.90 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 28.63 mm^{-1}
T = 296 K
0.18 × 0.12 × 0.08 mm

Data collection

Bruker X8 APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003) T_{min} = 0.029, T_{max} = 0.117
8738 measured reflections
1225 independent reflections
1202 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.051
S = 1.10
1225 reflections
116 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.37 e Å^{-3}
$\Delta\rho_{min}$ = −2.03 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O8—H8···O4	0.86	1.60	2.437 (5)	164
O8—H8···O1	0.86	2.76	3.393 (5)	132

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; 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, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HP2043).

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Dilead(II) trimanganese(II) bis(hydrogenphosphate) bis(phosphate)

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S1. Comment

Owing to their remarkable variety of structures and to their outstanding potentialities in widespread applications such as catalysis (Viter & Nagornyi, 2009; Gao & Gao, 2005) and ion-exchangers (Clearfield, 1988) and in batteries performance (Trad *et al.*, (2010)), transition metal based phosphates have received great attention and still remains in the forefront of the developed scientific axes in our laboratory. Within this family of compounds, the resulting anionic frameworks, generally constructed from the alternation of PO_4 tetrahedra connected to metal cations in different coordinate geometry MO_n (with n=4, 5 and 6), generate pores and channels offering suitable environment to accommodate different other cations. Accordingly, we have succeeded to isolate new silver metal based orthophosphates for instance, $AgMg_3(PO_4)(HPO_4)_2$ wich represent a new member of the well known alluaudite-like structure family (Assani *et al.* 2011*a*); silver (nickel or cobalt) phosphate, namely, $Ag_2M_3(HPO_4)(PO_4)_2$ with *M*=Ni, Co (Assani *et al.* 2011*b*; Assani *et al.* 2011*c*). Furthermore, a special attention have been paid to the ternary system MO—*M′*O—P_2O_5 with *M*=Ba, Ca, Cd, Pb and Sr and *M′*= transition metals, Mg and Zn. Our recent investigation has allowed to the isolate the compounds $Ni_2Sr(PO_4)_2.2H_2O$ (Assani *et al.* 2010) and $Co_2Pb(HPO_4)(PO_4)OH\ H_2O$ (Assani *et al.* 2012).

Inline with the focus of our research, the present paper aims to develop the hydrothermal synthesis and the structural characterization of a new layered lead manganese orthophosphate, namely, $PbMn_{1.5}(PO_4)(HPO_4)$, which is characterized by Mn/P ratio =3/4, rarely encountered in the literature with the exception of some copper based orthophosphates, $Pb_3Cu_3(PO_4)_4$ and $Sr_3Cu_3(PO_4)_4$ (Effenberger 1999).

A partial three-dimensional plot of the crystal structure of $PbMn_{1.5}(PO_4)(HPO_4)$ is represented in Fig. 1. A l l atoms of this structure are in general positions, except one manganese Mn1 located in symmetry center ($\overline{1}$) 2a (0 0 0; 0 1/2 1/2) of $P2_1/c$ space group. The network is built up from two different types of polyhedra more or less distorted, *viz*. PO_4, HPO_4 tetrahedra and $Mn1O_6$ ($\overline{1}$ symmetry), $Mn2O_6$ octahedra. Moreover, the edge-sharing $Mn1O_6$ and $Mn2O_5(OH)$ octahedra form an infinite zigzag chains $^1\infty$ $[Mn_3O_{14}]$ running parallel to [010], as shown in Fig. 2. Adjacent chains are connected by PO_4 and HPO_4 tetrahedra *via* vertices in the way to build layers parallel to (100). These layers are in turn linked by Pb^{2+} cations as shown in Fig.2. The strong hydrogen bonding between the layers is also involved in the stability of this structure (Fig. 2 and Table 2).

Bond valence sum calculations (Brown & Altermatt, 1985) for $Pb1^{2+}$, $Mn1^{2+}$, $Mb2^{2+}$, $P1^{5+}$ and $P2^{5+}$ ions are as expected, *viz*. 1.82, 2.13, 1.97, 4.95 and 5.01 valence units, respectively. The values of the bond valence sums calculated for all oxygen atoms are between 1.93 and 2.05 except O4 and O8 which shown low values: 1.62 and 1.50 respectively. These atoms are considerably undersaturated and thus act as an acceptor with a very short H-bond (Table 2, Fig.1).

S2. Experimental

The crystals of the title compound is isolated from the hydrothermal treatment of the reaction mixture of lead oxide, metallic manganese and 85wt% phosphoric acid in a proportion corresponding to the molar ratio Pb:Mn:*P* = 1,5: 3:3.

The hydrothermal reaction was conducted in a 23 ml Teflon-lined autoclave, filled to 50% with distilled water and under autogeneous pressure at 483 K for twenty hours. After being filtered off, washed with deionized water and air dried, the reaction product consists of a light brown solid and colorless sheet shaped crystals corresponding to the title compound.

S3. Refinement

The O-bound H atoms were initially located in a difference map and refined with O—H distance restraints of 0.86 (1). In the last cycle they were refined in the riding model approximation with $U_{\rm iso}$(H) set to $1.2U_{\rm eq}$(O). The highest peak and the deepest hole in the final Fourier map are at 0.62 Å and 0.67 Å, respectively, from Pb1. The not significants bonds and angles were removed from the CIF file.

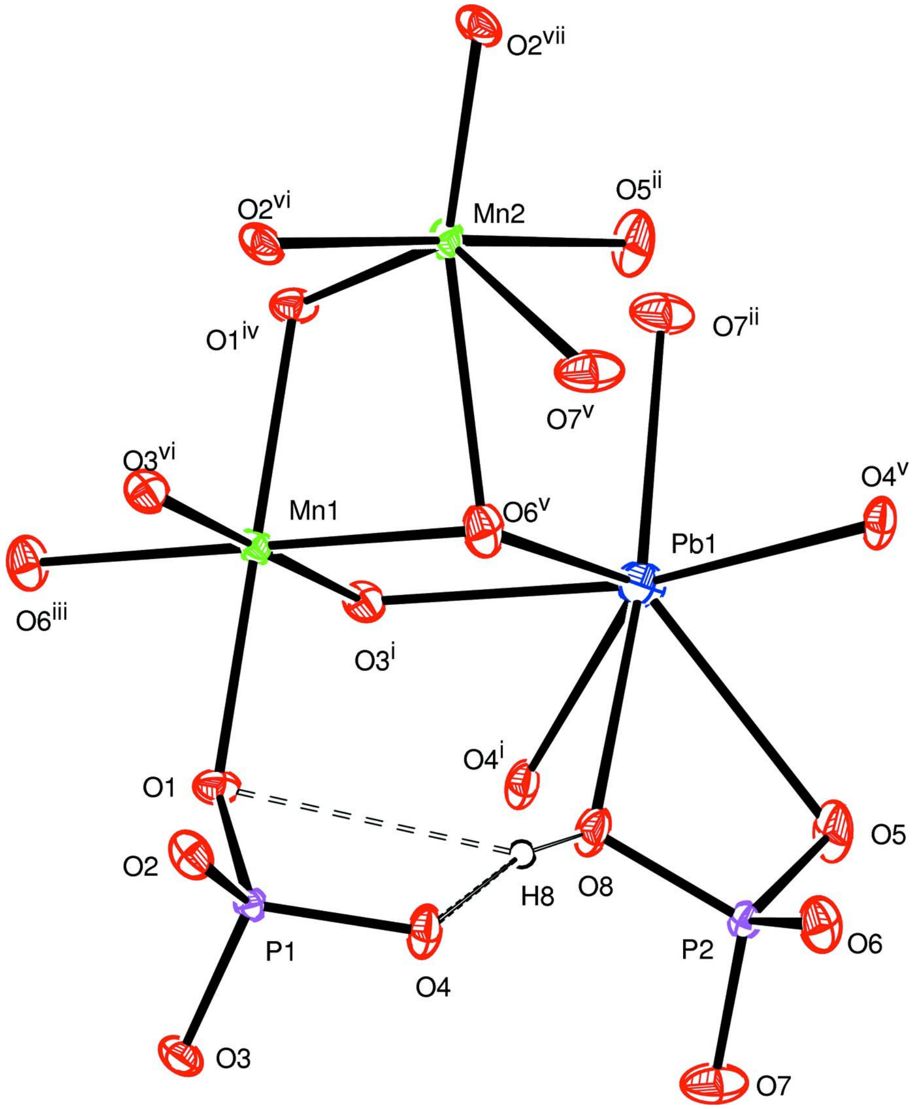

Figure 1

A partial three-dimensional plot of the crystal structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes:(i) x, $-y + 3/2$, $z + 1/2$; (ii) $-x + 1$, $-y + 1$, $-z + 1$; (iii) $-x + 1$, $y - 1/2$, $-z + 3/2$; (iv) $-x$, $y - 1/2$, $-z + 1/2$; (v) $x - 1$, y, z; (vi) $-x$, $-y + 1$, $-z + 1$; (vii) x, $-y + 1/2$, $z + 1/2$; (viii) x, $-y + 1/2$, $z - 1/2$; (ix) $-x$, $y + 1/2$, $-z + 1/2$; (x) x, $-y + 3/2$, $z - 1/2$; (xi) $-x + 1$, $y + 1/2$, $-z + 3/2$; (xii) $x + 1$, y, z.

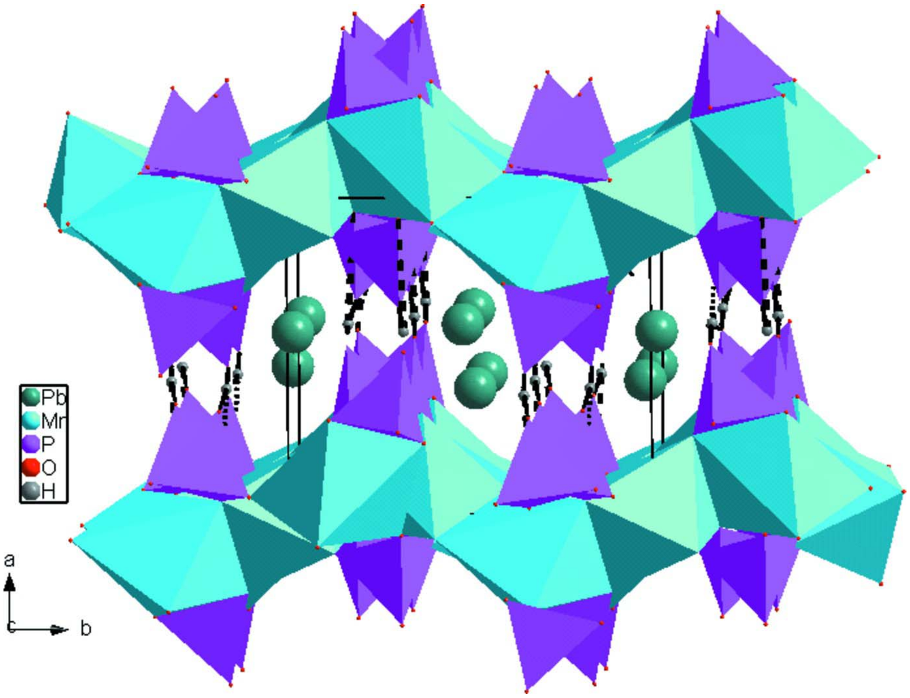

Figure 2

A three-dimensional polyhedral view of the crystal structure of the $PbMn_{1.5}(PO_4)(HPO_4)$, showing the stacking of layers along the *a* axis and the hydrogen bonding scheme (dashed lines).

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Crystal data

$Pb_2Mn_3(HPO_4)_2(PO_4)_2$
$M_r = 961.10$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.9449$ (2) Å
$b = 8.8911$ (2) Å
$c = 9.5718$ (3) Å
$\beta = 100.917$ (2)°
$V = 663.90$ (3) Å^3
$Z = 2$
$F(000) = 858$
$D_x = 4.808$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1225 reflections
$\theta = 2.6$–25.4°
$\mu = 28.63$ mm^{-1}
$T = 296$ K
Prism, pink
0.18 × 0.12 × 0.08 mm

Data collection

Bruker X8 APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.029$, $T_{\max} = 0.117$
8738 measured reflections
1225 independent reflections
1202 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$
$\theta_{\max} = 25.4°$, $\theta_{\min} = 2.6°$
$h = -9\rightarrow 9$
$k = -10\rightarrow 10$
$l = -11\rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.051$
$S = 1.10$
1225 reflections
116 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
$w = 1/[\sigma^2(F_o^2) + (0.0232P)^2 + 2.912P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.37$ e Å^{-3}
$\Delta\rho_{\min} = -2.03$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0091 (4)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (Å^2)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
Pb1	0.41097 (3)	0.52400 (2)	0.75343 (2)	0.01734 (13)
Mn1	0.0000	0.5000	0.5000	0.0091 (2)
Mn2	0.10770 (9)	0.13892 (8)	0.59303 (7)	0.00981 (19)
P1	0.14019 (16)	0.70441 (14)	0.23105 (12)	0.0076 (3)
P2	0.65619 (16)	0.70724 (14)	0.56583 (12)	0.0087 (3)
O1	0.0496 (5)	0.6808 (4)	0.3570 (4)	0.0123 (7)
O2	0.0740 (5)	0.5969 (4)	0.1080 (3)	0.0122 (7)
O3	0.1156 (5)	0.8690 (4)	0.1828 (4)	0.0127 (7)
O4	0.3370 (5)	0.6828 (4)	0.2816 (4)	0.0133 (7)
O5	0.7064 (5)	0.6754 (5)	0.7236 (4)	0.0208 (9)
O6	0.7536 (5)	0.6118 (4)	0.4742 (4)	0.0152 (8)
O7	0.6777 (5)	0.8718 (4)	0.5276 (4)	0.0212 (9)
O8	0.4617 (5)	0.6642 (4)	0.5341 (4)	0.0146 (7)
H8	0.4002	0.6667	0.4499	0.022*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.01783 (17)	0.01676 (17)	0.01923 (17)	0.00271 (7)	0.00812 (10)	0.00201 (7)
Mn1	0.0108 (6)	0.0084 (5)	0.0080 (5)	0.0006 (4)	0.0018 (4)	0.0002 (4)
Mn2	0.0096 (4)	0.0107 (4)	0.0082 (4)	0.0003 (3)	−0.0008 (3)	−0.0006 (3)
P1	0.0080 (6)	0.0090 (6)	0.0055 (6)	−0.0004 (5)	0.0003 (5)	0.0003 (4)
P2	0.0080 (6)	0.0111 (6)	0.0067 (6)	−0.0007 (5)	0.0009 (5)	−0.0005 (4)

O1	0.0154 (19)	0.0110 (17)	0.0120 (17)	−0.0005 (14)	0.0068 (14)	0.0016 (13)
O2	0.0154 (19)	0.0111 (17)	0.0076 (16)	0.0002 (14)	−0.0045 (14)	−0.0020 (13)
O3	0.0154 (19)	0.0099 (17)	0.0120 (17)	0.0005 (14)	0.0008 (14)	0.0026 (14)
O4	0.0083 (18)	0.0234 (19)	0.0078 (16)	0.0002 (14)	0.0004 (13)	−0.0002 (14)
O5	0.015 (2)	0.038 (2)	0.0076 (18)	0.0013 (17)	−0.0021 (14)	0.0035 (16)
O6	0.0113 (18)	0.0200 (19)	0.0153 (17)	0.0061 (15)	0.0048 (14)	0.0015 (15)
O7	0.030 (2)	0.0118 (18)	0.026 (2)	−0.0036 (16)	0.0151 (18)	−0.0013 (16)
O8	0.0087 (17)	0.0261 (19)	0.0079 (16)	−0.0034 (15)	−0.0012 (14)	0.0016 (15)

Geometric parameters (Å, º)

Pb1—O3^{i}	2.504 (4)	P1—O3	1.536 (3)
Pb1—O8	2.539 (4)	P1—O4	1.559 (4)
Pb1—O6^{ii}	2.614 (4)	P2—O5	1.514 (4)
Pb1—O4^{i}	2.697 (4)	P2—O7	1.526 (4)
Pb1—O7^{iii}	2.698 (4)	P2—O6	1.532 (4)
Pb1—O5	2.767 (4)	P2—O8	1.565 (4)
Pb1—O4^{ii}	2.785 (4)	O1—Mn2^{vi}	2.141 (4)
Mn1—O3^{iv}	2.157 (3)	O2—Mn2^{viii}	2.122 (4)
Mn1—O3^{i}	2.157 (3)	O2—Mn2^{ix}	2.208 (3)
Mn1—O6^{ii}	2.168 (3)	O3—Mn1^{ix}	2.157 (3)
Mn1—O6^{v}	2.168 (3)	O3—Pb1^{x}	2.504 (4)
Mn1—O1	2.195 (3)	O4—Pb1^{x}	2.697 (4)
Mn1—O1^{vi}	2.195 (3)	O4—Pb1^{ii}	2.785 (4)
Mn2—O5^{iii}	2.094 (4)	O5—Mn2^{xi}	2.094 (4)
Mn2—O2^{vii}	2.122 (4)	O6—Mn1^{xii}	2.168 (3)
Mn2—O1^{vi}	2.141 (4)	O6—Mn2^{ii}	2.610 (4)
Mn2—O2^{iv}	2.208 (3)	O6—Pb1^{ii}	2.614 (4)
Mn2—O7^{ii}	2.235 (4)	O7—Mn2^{ii}	2.235 (4)
Mn2—O6^{ii}	2.610 (4)	O7—Pb1^{xi}	2.698 (4)
P1—O2	1.530 (3)	O8—H8	0.8600
P1—O1	1.531 (3)		

O3^{i}—Pb1—O8	82.97 (11)	O3^{i}—Mn1—O1^{vi}	89.35 (13)
O3^{i}—Pb1—O6^{ii}	69.84 (11)	O6^{ii}—Mn1—O1^{vi}	81.84 (13)
O8—Pb1—O6^{ii}	70.75 (11)	O6^{v}—Mn1—O1^{vi}	98.16 (13)
O3^{i}—Pb1—O4^{i}	56.56 (11)	O1—Mn1—O1^{vi}	180.000 (1)
O8—Pb1—O4^{i}	71.32 (11)	O5^{iii}—Mn2—O2^{vii}	100.04 (15)
O6^{ii}—Pb1—O4^{i}	116.47 (11)	O5^{iii}—Mn2—O1^{vi}	92.62 (15)
O3^{i}—Pb1—O7^{iii}	91.78 (12)	O2^{vii}—Mn2—O1^{vi}	129.62 (14)
O8—Pb1—O7^{iii}	173.95 (12)	O5^{iii}—Mn2—O2^{iv}	176.09 (15)
O6^{ii}—Pb1—O7^{iii}	104.65 (12)	O2^{vii}—Mn2—O2^{iv}	79.73 (13)
O4^{i}—Pb1—O7^{iii}	108.25 (11)	O1^{vi}—Mn2—O2^{iv}	90.49 (14)
O3^{i}—Pb1—O5	123.87 (12)	O5^{iii}—Mn2—O7^{ii}	87.34 (15)
O8—Pb1—O5	53.50 (11)	O2^{vii}—Mn2—O7^{ii}	96.38 (14)
O6^{ii}—Pb1—O5	116.03 (11)	O1^{vi}—Mn2—O7^{ii}	133.01 (14)
O4^{i}—Pb1—O5	75.23 (12)	O2^{iv}—Mn2—O7^{ii}	88.81 (14)
O7^{iii}—Pb1—O5	132.49 (12)	O5^{iii}—Mn2—O6^{ii}	79.12 (14)

O3^{i}—Pb1—O4^{ii}	151.26 (10)	O2^{vii}—Mn2—O6^{ii}	157.01 (13)
O8—Pb1—O4^{ii}	89.67 (11)	O1^{vi}—Mn2—O6^{ii}	73.21 (12)
O6^{ii}—Pb1—O4^{ii}	81.50 (10)	O2^{iv}—Mn2—O6^{ii}	99.54 (12)
O4^{i}—Pb1—O4^{ii}	145.63 (8)	O7^{ii}—Mn2—O6^{ii}	60.65 (12)
O7^{iii}—Pb1—O4^{ii}	93.55 (11)	O2—P1—O1	112.1 (2)
O5—Pb1—O4^{ii}	70.46 (12)	O2—P1—O3	111.0 (2)
O3^{iv}—Mn1—O3^{i}	180.000 (1)	O1—P1—O3	108.4 (2)
O3^{iv}—Mn1—O6^{ii}	94.68 (14)	O2—P1—O4	109.9 (2)
O3^{i}—Mn1—O6^{ii}	85.32 (14)	O1—P1—O4	109.4 (2)
O3^{iv}—Mn1—O6^{v}	85.32 (14)	O3—P1—O4	105.9 (2)
O3^{i}—Mn1—O6^{v}	94.68 (14)	O5—P2—O7	113.5 (2)
O6^{ii}—Mn1—O6^{v}	180.00 (19)	O5—P2—O6	113.7 (2)
O3^{iv}—Mn1—O1	89.35 (13)	O7—P2—O6	107.6 (2)
O3^{i}—Mn1—O1	90.65 (13)	O5—P2—O8	102.2 (2)
O6^{ii}—Mn1—O1	98.16 (13)	O7—P2—O8	109.8 (2)
O6^{v}—Mn1—O1	81.84 (13)	O6—P2—O8	109.9 (2)
O3^{iv}—Mn1—O1^{vi}	90.65 (13)		

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+1, *y*−1/2, −*z*+3/2; (iv) −*x*, *y*−1/2, −*z*+1/2; (v) *x*−1, *y*, *z*; (vi) −*x*, −*y*+1, −*z*+1; (vii) *x*, −*y*+1/2, *z*+1/2; (viii) *x*, −*y*+1/2, *z*−1/2; (ix) −*x*, *y*+1/2, −*z*+1/2; (x) *x*, −*y*+3/2, *z*−1/2; (xi) −*x*+1, *y*+1/2, −*z*+3/2; (xii) *x*+1, *y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O8—H8···O4	0.86	1.60	2.437 (5)	164
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