

Synthesis, crystal structure and Raman spectrum of $K_2[(Pt_2)(HPO_4)_4(H_2O)_2]$ containing $(Pt_2)^{6+}$ ions

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Key indicators: single-crystal X-ray study; $T = 123\text{ K}$, $P = 0.0\text{ kPa}$; mean $\sigma(\text{P}-\text{O}) = 0.005\text{ \AA}$; R factor = 0.033; wR factor = 0.086; data-to-parameter ratio = 21.6.

In the crystal structure of the acid platinum phosphate dipotassium di- μ -hydrogenphosphato-bis[aquaplatinum(III)]- $(Pt-Pt)$, $K_2[Pt_2(HPO_4)_4(H_2O)_2]$, the $(Pt_2)^{6+}$ dumbbells within the paddle-wheel complex show $\text{Pt}-\text{Pt}$ distances of $2.4944(5)$ and $2.4892(5)\text{ \AA}$. The potassium ions are seven-fold coordinated by hydrogenphosphate groups. In the crystal, $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds help to establish the packing. The Raman spectrum was recorded.

Related literature

The structure of the title isotypic sodium compound, $\text{Na}_2[Pt_2(HPO_4)_4(H_2O)_2]$, was determined by Cotton *et al.* (1982a). For platinum phosphates, see: Wellmann & Liebau (1981). For related compounds containing dinuclear platinum(III), see: Bancroft *et al.* (1984); Baranovskii & Schelokow (1978); Che *et al.* (1982); Cotton & Walton (1982b); Dikareva *et al.* (1982, 1987); Muraveiskaya *et al.* (1974, 1976); Pley & Wickleder (2004a,b, 2005); Stein *et al.* (1983); Woollins & Kelly (1985). For the ternary system Pd/P/O, see: Palkina *et al.* (1978); Panagiotidis *et al.* (2005). For hydrogen bonds, see: Steiner (2002). For the Raman spectra of $\text{In}_3(\text{PO}_4)_2$ and $\text{In}_2\text{O}(\text{PO}_4)$, see: Thauern & Glaum (2004). For the synthesis, see: Brauer (1978).

Experimental

Crystal data

$K_2[Pt_2(HPO_4)_4(H_2O)_2]$	$\gamma = 65.528(1)^\circ$
$M_r = 888.32$	$V = 776.32(4)\text{ \AA}^3$
Triclinic, $\bar{P}\bar{1}$	$Z = 2$
$a = 7.8852(2)\text{ \AA}$	Mo $K\alpha$ radiation
$b = 7.9657(2)\text{ \AA}$	$\mu = 19.05\text{ mm}^{-1}$
$c = 13.7739(4)\text{ \AA}$	$T = 123\text{ K}$
$\alpha = 82.358(1)^\circ$	$0.24 \times 0.14 \times 0.08\text{ mm}$
$\beta = 81.509(1)^\circ$	

Data collection

Nonius Kappa CCD diffractometer
Absorption correction: multi-scan
(Blessing, 1995)
 $T_{\min} = 0.060$, $T_{\max} = 0.224$

18915 measured reflections
5589 independent reflections
4077 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.086$
 $S = 0.98$
5589 reflections
259 parameters
10 restraints

H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\max} = 3.46\text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -2.87\text{ e \AA}^{-3}$

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O1—H1A \cdots O31 ⁱ	0.83 (6)	1.73 (6)	2.559 (7)	173 (8)
O1—H1B \cdots O21 ⁱⁱ	0.84 (6)	2.09 (4)	2.860 (7)	153 (8)
O14—H14 \cdots O31 ⁱⁱⁱ	0.83 (2)	1.82 (5)	2.548 (7)	146 (9)
O24—H24 \cdots O11 ^{iv}	0.84 (7)	1.73 (7)	2.562 (7)	177 (9)
O34—H34 \cdots O41 ^v	0.83 (6)	1.74 (3)	2.546 (6)	162 (9)
O10—H10A \cdots O41 ^{vi}	0.86 (2)	1.94 (4)	2.713 (7)	148 (7)
O10—H10B \cdots O11 ^{vii}	0.86 (2)	1.86 (2)	2.694 (6)	164 (6)
O42—H42 \cdots O21 ^{viii}	0.84 (7)	2.27 (9)	2.475 (7)	94 (6)

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

Data collection: *COLLECT* (Hooft, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ATOMS* (Dowty, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2058).

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Synthesis, crystal structure and Raman spectrum of $K_2[(Pt_2)(HPO_4)_4(H_2O)_2]$ containing $(Pt_2)^{6+}$ ions

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Comment

Information on phosphates of noble metals like platinum and palladium is scarcely found in literature. During our recent investigation of the ternary system Pd/P/O we obtained the diphosphate $Pd^{II}P_2O_7$ (Panagiotidis *et al.*, 2005) in addition to the already known $Pd(PO_3)_2$ (Palkina *et al.*, 1978). In this context we were also interested in the crystal chemistry of platinum phosphates. Up to now, $Pt^{IV}P_2O_7$ (Wellmann & Liebau, 1981) is the only structurally characterized anhydrous phosphate of platinum. Since reactions starting from "PtO \times 3H₂O" and P₄O₁₀ did not yield products suitable for closer investigation we tried an alternative synthetic approach by reacting K₂PtCl₄ with conc. H₃PO₄ (see Experimental). This led to the formation of orange crystals of K₂[(Pt₂)(HPO₄)₄(H₂O)₂]. The acid phosphate is isotopic to the sodium compound (Cotton *et al.*, 1982a). In contrast to the structure of Na₂[(Pt₂)(HPO₄)₄(H₂O)₂] no disordered oxygen atoms are observed in the potassium compound. Distances d(Pt—Pt) for both structures are identical. The conventional residual as well as the standard deviations of the interatomic distances are slightly smaller for the refinement of K₂[(Pt₂)(HPO₄)₄(H₂O)₂]. The (Pt₂)⁶⁺ binuclear complex was first observed in the crystal structure of K₂[(Pt₂(SO₄)₄(H₂O)₂] (Muraveiskaya *et al.*, 1974; Muraveiskaya *et al.*, 1976). By reaction of elemental platinum with concentrated sulfuric acid various platinum(III) sulfates were recently synthesized and structurally characterized (Pley & Wickleder, 2004a,b; Pley & Wickleder, 2005).

In K₂[(Pt₂)(HPO₄)₄(H₂O)₂] two crystallographically equivalent platinum atoms are connected to form (Pt₂)⁶⁺ dinuclear complexes with surrounding (HPO₄)²⁻ groups (Fig. 1). The ligating oxygen atoms of the (HPO₄)²⁻ ions are arranged in a square-planar coordination around each platinum atom. Distances d(Pt—O) range from 1.978 Å to 2.030 Å. Angles $\langle O, Pt, O \rangle$ are deviating only slightly from 90° and 180°, respectively. Due to their different crystal chemical environment, the oxygen atoms of the hydrogenphosphate anions show significantly different bond lengths d(P—O). Oxygen atoms attached to platinum (coordination number of oxygen c.n.(O) = 2 (P, Pt)) show distances d(P—O) = 1.55 Å. Distances d(P—O) for those oxygen atoms which are coordinated to K⁺ ions (c.n.(O) = 2 (P, K)) range from 1.489 Å to 1.507 Å. Furthermore, for oxygen atoms which are attached to a hydrogen atom within the (HPO₄)²⁻ unit (c.n.(O) = 2, (P, H)), distances d(P—OH) around 1.565 Å are observed. The axial ligand positions of the Pt₂ dumbbells are occupied by water molecules (Fig. 1 & 2). Distances d(Pt—O) = 2.135 Å observed for the water ligands are significantly longer than those within the [Pt₂O₈] entity. The hydrogen atoms of [HPO₄] tetrahedra (H14, H24, H34, H42; numbering according to the oxygen atoms that carry the hydrogen) and the water molecules (H1A, H1B, H10A, H10B) are involved in hydrogen bonding with oxygen atoms of adjacent [(Pt₂)(HPO₄)(H₂O)₂]²⁻ units. Interatomic distances d(OH \cdots OP) range from 1.73 Å to 2.27 Å. They are in good accordance with those observed for strong hydrogen bonds (Steiner, 2002).

supplementary materials

As found for various other compounds containing the $(\text{Pt}_2)^{6+}$ dinuclear complex, the angle $\langle(\text{Pt}, \text{Pt}, O)\rangle$ between the dumbbell and the axial oxygen atoms deviates only slightly from 180° (Cotton *et al.*, 1982*b*; Pley & Wickleder *et al.*, 2005). $[\text{HPO}_4]$ tetrahedra in $[(\text{Pt}_2)(\text{HPO}_4)(\text{H}_2\text{O})_2]^{2-}$ show no significant angular distortion. Charge compensation of the anionic $[\text{Pt}^{\text{III}}_2(\text{HPO}_4)_4(\text{H}_2\text{O})_2]^{2-}$ unit is achieved by two crystallographically independent K^+ ions, which are surrounded by oxygen atoms of phosphate groups.

In addition to its structure refinement, we were able to record the Raman spectrum of the paddle-wheel complex $[\text{Pt}^{\text{III}}_2(\text{HPO}_4)_4(\text{H}_2\text{O})_2]^{2-}$ (Fig. 3). An unequivocal assignment of the observed signals is yet impossible. Comparison of the Raman spectrum to those of the In_2^{4+} containing indium phosphates $\text{In}_3(\text{PO}_4)_2$ and $\text{In}_2\text{O}(\text{PO}_4)$ (Thauern & Glaum, 2004) suggests the Pt—Pt valence vibration to be at $\nu = 222 \text{ cm}^{-1}$. In comparison to $\nu(\text{Pt—Pt})$ in complexes $[(\text{Pt}^{\text{III}}_2)L_4\text{L}'_2]^{n-}$ (Stein *et al.*, 1983) assignment of $\nu = 83 \text{ cm}^{-1}$ to the Pt—Pt vibration appears to be unreasonable. This is the more so, since $d(\text{Pt—Pt}) = 2.51 \text{ \AA}$ observed for $\text{K}_2[(\text{Pt}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$ is close to the lower limit of $2.47 \text{ \AA} < d(\text{Pt—Pt}) < 2.695 \text{ \AA}$ found for a series of dinuclear platinum(III) complexes (Che *et al.*, 1982, Stein *et al.*, 1983, Muraveiskaya *et al.*, 1974).

Experimental

Aiming at the crystallization of "Pt₂P₂O₇" a reaction starting from 150.0 mg $\text{K}_2\text{Pt}^{\text{II}}\text{Cl}_4$, which were dissolved in water and mixed with 5.0 ml conc. H_3PO_4 , was performed. After the obtained red solution was kept in a desiccator over P_2O_5 for two weeks, plate-like, orange crystals of $\text{K}_2[(\text{Pt}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$ with edge-lengths up to 0.3 mm were deposited besides microcrystalline platinum (eq. 1). The synthesis of $\text{K}_2[\text{Pt}^{\text{II}}\text{Cl}_4]$ was performed according to the procedure given by Brauer (1978).



Figures

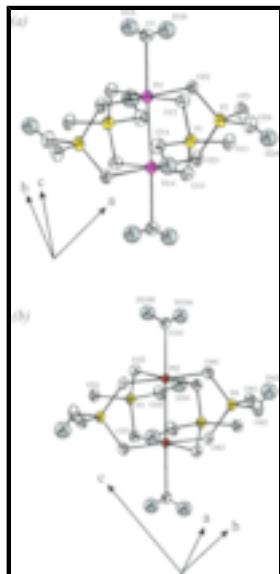


Fig. 1. Paddle-wheel complex $[(\text{Pt}^{\text{III}}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$ with anisotropic displacement parameters given at the 50% level (Progr. ATOMS v.6.2).

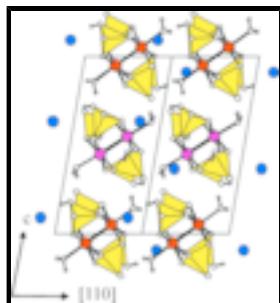


Fig. 2. Projection of the crystal structure of $\text{K}_2[(\text{Pt}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$ along $[1\bar{1}0]$. $[\text{PO}_4]$ tetrahedra (yellow), Pt_2^{6+} (red), K^+ (violet), H^+ (blue), O^{2-} (white) (Progr. ATOMS v.6.2).

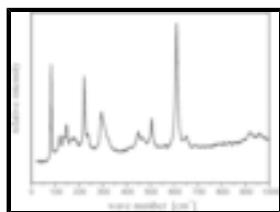


Fig. 3. Raman spectrum of $\text{K}_2[(\text{Pt}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$.

dipotassium di- μ -hydrogenphosphato-bis[aquaplatinum(III)] (Pt—Pt)

Crystal data

$\text{K}_2[\text{Pt}_2(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$

$F_{000} = 812$

$M_r = 888.32$

The lattice parameters of $\text{K}_2[\text{Pt}_2(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$ were determined from single crystal diffraction data.

Triclinic, PT

$D_x = 3.800 \text{ Mg m}^{-3}$

Hall symbol: -P 1

Mo $K\alpha$ radiation

$a = 7.8852 (2) \text{ \AA}$

$\lambda = 0.71073 \text{ \AA}$

$b = 7.9657 (2) \text{ \AA}$

Cell parameters from 5589 reflections

$c = 13.7739 (4) \text{ \AA}$

$\theta = 1.5\text{--}32.5^\circ$

$\alpha = 82.358 (1)^\circ$

$\mu = 19.05 \text{ mm}^{-1}$

$\beta = 81.509 (1)^\circ$

$T = 123 \text{ K}$

$\gamma = 65.528 (1)^\circ$

Cell measurement pressure: 101.3 kPa

$V = 776.32 (4) \text{ \AA}^3$

Plate, orange

$Z = 2$

$0.24 \times 0.14 \times 0.08 \text{ mm}$

Data collection

Nonius Kappa CCD
diffractometer

18915 measured reflections

Radiation source: Mo $K\alpha$

5589 independent reflections

Monochromator: graphite

4077 reflections with $I > 2\sigma(I)$

Detector resolution: 9 pixels mm^{-1}

$R_{\text{int}} = 0.064$

$T = 123 \text{ K}$

$\theta_{\text{max}} = 32.5^\circ$

$P = 101.3 \text{ kPa}$

$\theta_{\text{min}} = 1.5^\circ$

CCD rotation images scans

$h = -11 \rightarrow 11$

Absorption correction: multi-scan
(Blessing, 1995)

$k = -12 \rightarrow 12$

$T_{\text{min}} = 0.060, T_{\text{max}} = 0.224$

$l = -19 \rightarrow 20$

supplementary materials

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.086$	$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{\text{max}} = 0.001$
5589 reflections	$\Delta\rho_{\text{max}} = 3.46 \text{ e \AA}^{-3}$
259 parameters	$\Delta\rho_{\text{min}} = -2.87 \text{ e \AA}^{-3}$
10 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s 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 and 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 $F^2 > \sigma(F^2)$ 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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
K1	0.1242 (4)	-0.3657 (4)	0.6326 (2)	0.0790 (9)
K2	0.3845 (3)	-0.1884 (3)	0.90856 (15)	0.0472 (5)
O1	0.6393 (7)	0.2464 (8)	0.6286 (4)	0.0289 (11)
H1A	0.566 (8)	0.299 (10)	0.676 (4)	0.043*
H1B	0.734 (6)	0.175 (9)	0.656 (5)	0.043*
O12	0.6784 (7)	-0.1304 (6)	0.6372 (3)	0.0238 (10)
O14	0.4660 (7)	-0.2761 (7)	0.7170 (4)	0.0305 (11)
H14	0.486 (13)	-0.387 (4)	0.717 (6)	0.046*
O11	0.8028 (7)	-0.4742 (7)	0.6678 (3)	0.0256 (10)
O13	0.5849 (7)	-0.3216 (6)	0.5370 (3)	0.0245 (10)
O22	0.7832 (6)	0.0412 (7)	0.4628 (4)	0.0247 (10)
O24	0.9754 (7)	-0.3003 (7)	0.4569 (3)	0.0276 (11)
H24	1.045 (10)	-0.372 (10)	0.415 (5)	0.041*
O21	0.9903 (7)	-0.0893 (7)	0.3117 (4)	0.0298 (11)
O23	0.6936 (6)	-0.1446 (7)	0.3608 (3)	0.0267 (10)
O10	0.2526 (7)	0.5993 (7)	0.1437 (3)	0.0249 (10)
H10A	0.207 (11)	0.719 (3)	0.141 (5)	0.037*

H10B	0.226 (11)	0.582 (9)	0.2058 (19)	0.037*
O32	-0.1067 (6)	0.5907 (6)	0.1622 (3)	0.0212 (9)
O33	-0.3103 (6)	0.5226 (6)	0.0567 (3)	0.0199 (9)
O34	-0.4078 (7)	0.8408 (7)	0.1168 (4)	0.0286 (11)
H34	-0.520 (4)	0.896 (11)	0.107 (6)	0.043*
O31	-0.4038 (6)	0.5674 (7)	0.2362 (3)	0.0231 (10)
O44	-0.0110 (6)	0.8018 (7)	0.0001 (3)	0.0236 (10)
O41	-0.2456 (6)	1.0558 (6)	-0.0996 (3)	0.0233 (10)
O43	-0.2059 (6)	0.7307 (6)	-0.1068 (3)	0.0210 (9)
O42	0.0530 (7)	0.8330 (7)	-0.1816 (4)	0.0310 (12)
H42	0.050 (13)	0.930 (7)	-0.162 (6)	0.047*
P1	0.6381 (2)	-0.3051 (2)	0.63814 (12)	0.0220 (3)
P2	0.8592 (2)	-0.1187 (2)	0.39488 (12)	0.0215 (3)
P3	-0.3082 (2)	0.6280 (2)	0.14389 (12)	0.0180 (3)
P4	-0.1056 (2)	0.8585 (2)	-0.09741 (12)	0.0187 (3)
Pt1	0.54549 (3)	0.09217 (3)	0.549475 (17)	0.01966 (7)
Pt2	0.09528 (3)	0.53533 (3)	0.052197 (16)	0.01543 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.101 (2)	0.087 (2)	0.0691 (17)	-0.0526 (18)	-0.0488 (16)	0.0208 (15)
K2	0.0525 (12)	0.0601 (13)	0.0406 (10)	-0.0346 (11)	0.0048 (9)	-0.0133 (9)
O1	0.030 (3)	0.030 (3)	0.029 (3)	-0.013 (2)	0.000 (2)	-0.010 (2)
O12	0.030 (2)	0.020 (2)	0.021 (2)	-0.011 (2)	-0.0040 (19)	0.0035 (18)
O14	0.032 (3)	0.028 (3)	0.028 (3)	-0.014 (2)	0.007 (2)	0.001 (2)
O11	0.028 (2)	0.021 (2)	0.019 (2)	-0.003 (2)	-0.0023 (19)	0.0031 (18)
O13	0.029 (2)	0.019 (2)	0.021 (2)	-0.006 (2)	-0.0018 (19)	0.0019 (18)
O22	0.019 (2)	0.023 (2)	0.030 (3)	-0.007 (2)	0.0021 (19)	-0.005 (2)
O24	0.029 (3)	0.026 (3)	0.019 (2)	-0.005 (2)	0.0028 (19)	0.0021 (19)
O21	0.022 (2)	0.031 (3)	0.029 (3)	-0.008 (2)	0.0055 (19)	0.007 (2)
O23	0.021 (2)	0.028 (3)	0.025 (2)	-0.005 (2)	0.0031 (19)	-0.006 (2)
O10	0.028 (2)	0.032 (3)	0.020 (2)	-0.018 (2)	-0.0068 (19)	0.0045 (19)
O32	0.018 (2)	0.023 (2)	0.020 (2)	-0.0061 (19)	-0.0014 (17)	-0.0005 (18)
O33	0.018 (2)	0.025 (2)	0.016 (2)	-0.0101 (19)	0.0043 (16)	-0.0019 (17)
O34	0.020 (2)	0.025 (3)	0.038 (3)	-0.006 (2)	-0.003 (2)	-0.001 (2)
O31	0.024 (2)	0.026 (3)	0.019 (2)	-0.013 (2)	0.0055 (18)	-0.0032 (18)
O44	0.021 (2)	0.023 (2)	0.028 (2)	-0.011 (2)	-0.0080 (18)	0.0054 (19)
O41	0.021 (2)	0.017 (2)	0.031 (3)	-0.0073 (19)	-0.0051 (19)	0.0016 (19)
O43	0.022 (2)	0.020 (2)	0.019 (2)	-0.0077 (19)	-0.0011 (17)	0.0025 (17)
O42	0.025 (2)	0.025 (3)	0.034 (3)	-0.007 (2)	0.008 (2)	0.004 (2)
P1	0.0237 (8)	0.0200 (8)	0.0183 (8)	-0.0061 (7)	-0.0002 (6)	0.0009 (6)
P2	0.0194 (8)	0.0218 (9)	0.0189 (8)	-0.0066 (7)	0.0020 (6)	0.0024 (6)
P3	0.0150 (7)	0.0204 (8)	0.0172 (7)	-0.0071 (6)	0.0016 (6)	-0.0005 (6)
P4	0.0167 (7)	0.0176 (8)	0.0190 (7)	-0.0057 (6)	-0.0011 (6)	0.0033 (6)
Pt1	0.02042 (12)	0.01997 (13)	0.01736 (12)	-0.00806 (10)	0.00056 (9)	-0.00042 (9)
Pt2	0.01457 (11)	0.01729 (12)	0.01430 (11)	-0.00707 (9)	-0.00109 (8)	0.00101 (8)

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Geometric parameters (\AA , $^\circ$)

K1—O24 ⁱ	2.739 (6)	O24—P2	1.570 (5)
K1—O31 ⁱⁱ	2.860 (5)	O21—P2	1.489 (5)
K1—O11 ⁱ	2.957 (6)	O23—P2	1.549 (5)
K1—O42 ⁱⁱⁱ	3.047 (6)	O23—Pt1 ^{iv}	2.014 (5)
K1—O22 ^{iv}	3.053 (5)	O10—Pt2	2.132 (5)
K1—O32 ⁱⁱ	3.156 (5)	O32—P3	1.544 (5)
K1—O12 ⁱ	3.222 (6)	O32—Pt2	1.978 (4)
K2—O14	2.735 (6)	O33—P3	1.561 (5)
K2—O34 ^v	2.822 (6)	O33—Pt2 ^{vii}	2.030 (4)
K2—O41 ^v	2.858 (5)	O34—P3	1.564 (5)
K2—O33 ⁱⁱ	2.920 (5)	O31—P3	1.507 (4)
K2—O42 ⁱⁱⁱ	2.990 (6)	O44—P4	1.555 (5)
K2—O43 ^{vi}	3.000 (5)	O44—Pt2	2.005 (5)
K2—O44 ⁱⁱⁱ	3.215 (5)	O41—P4	1.500 (5)
O1—Pt1	2.143 (5)	O43—P4	1.553 (5)
O12—P1	1.549 (5)	O43—Pt2 ^{vii}	2.014 (4)
O12—Pt1	1.992 (4)	O42—P4	1.541 (5)
O14—P1	1.564 (5)	Pt1—O13 ^{iv}	2.010 (4)
O11—P1	1.493 (5)	Pt1—O23 ^{iv}	2.014 (5)
O13—P1	1.549 (5)	Pt1—Pt1 ^{iv}	2.4944 (5)
O13—Pt1 ^{iv}	2.010 (4)	Pt2—O43 ^{vii}	2.014 (4)
O22—P2	1.541 (5)	Pt2—O33 ^{vii}	2.030 (4)
O22—Pt1	1.985 (4)	Pt2—Pt2 ^{vii}	2.4892 (5)
O11—P1—O12	110.2 (3)	O22—Pt1—O23 ^{iv}	179.02 (19)
O11—P1—O13	110.9 (3)	O12—Pt1—O23 ^{iv}	90.37 (19)
O12—P1—O13	111.5 (3)	O13 ^{iv} —Pt1—O23 ^{iv}	89.97 (19)
O11—P1—O14	110.2 (3)	O22—Pt1—O1	85.5 (2)
O12—P1—O14	104.8 (3)	O12—Pt1—O1	88.0 (2)
O13—P1—O14	109.0 (3)	O13 ^{iv} —Pt1—O1	90.0 (2)
O21—P2—O22	111.1 (3)	O23 ^{iv} —Pt1—O1	93.6 (2)
O21—P2—O23	113.2 (3)	O22—Pt1—Pt1 ^{iv}	90.98 (14)
O22—P2—O23	109.6 (3)	O12—Pt1—Pt1 ^{iv}	91.86 (14)
O21—P2—O24	106.9 (3)	O13 ^{iv} —Pt1—Pt1 ^{iv}	90.05 (14)
O22—P2—O24	107.7 (3)	O23 ^{iv} —Pt1—Pt1 ^{iv}	89.91 (15)
O23—P2—O24	108.1 (3)	O1—Pt1—Pt1 ^{iv}	176.49 (14)
O31—P3—O32	108.9 (3)	O32—Pt2—O44	90.05 (19)
O31—P3—O33	109.3 (3)	O32—Pt2—O43 ^{vii}	89.88 (18)
O32—P3—O33	111.8 (3)	O44—Pt2—O43 ^{vii}	178.49 (18)
O31—P3—O34	111.6 (3)	O32—Pt2—O33 ^{vii}	177.57 (17)

O32—P3—O34	106.3 (3)	O44—Pt2—O33 ^{vii}	90.65 (19)
O33—P3—O34	108.9 (3)	O43 ^{vii} —Pt2—O33 ^{vii}	89.36 (18)
O41—P4—O42	110.7 (3)	O32—Pt2—O10	87.17 (19)
O41—P4—O43	109.3 (3)	O44—Pt2—O10	89.05 (19)
O42—P4—O43	110.0 (3)	O43 ^{vii} —Pt2—O10	89.45 (19)
O41—P4—O44	110.6 (3)	O33 ^{vii} —Pt2—O10	90.51 (18)
O42—P4—O44	106.4 (3)	O32—Pt2—Pt2 ^{vii}	91.55 (13)
O43—P4—O44	109.7 (2)	O44—Pt2—Pt2 ^{vii}	90.52 (13)
O22—Pt1—O12	89.21 (19)	O43 ^{vii} —Pt2—Pt2 ^{vii}	90.99 (13)
O22—Pt1—O13 ^{iv}	90.42 (19)	O33 ^{vii} —Pt2—Pt2 ^{vii}	90.77 (13)
O12—Pt1—O13 ^{iv}	178.06 (19)	O10—Pt2—Pt2 ^{vii}	178.65 (14)

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

Hydrogen-bond geometry (\AA , °)

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O1—H1A···O31 ^v	0.83 (6)	1.73 (6)	2.559 (7)	173 (8)
O1—H1B···O21 ^{viii}	0.84 (6)	2.09 (4)	2.860 (7)	153 (8)
O14—H14···O31 ⁱⁱ	0.83 (2)	1.82 (5)	2.548 (7)	146 (9)
O24—H24···O11 ^{ix}	0.84 (7)	1.73 (7)	2.562 (7)	177 (9)
O34—H34···O41 ^x	0.83 (6)	1.74 (3)	2.546 (6)	162 (9)
O10—H10A···O41 ^{xi}	0.86 (2)	1.94 (4)	2.713 (7)	148 (7)
O10—H10B···O11 ^{iv}	0.86 (2)	1.86 (2)	2.694 (6)	164 (6)
O42—H42···O21 ^{xii}	0.84 (7)	2.27 (9)	2.475 (7)	94 (6)

Symmetry codes: (v) $-x, -y+1, -z+1$; (viii) $-x+2, -y, -z+1$; (ii) $-x, -y, -z+1$; (ix) $-x+2, -y-1, -z+1$; (x) $-x-1, -y+2, -z$; (xi) $-x, -y+2, -z$; (iv) $-x+1, -y, -z+1$; (xii) $-x+1, -y+1, -z$.

supplementary materials

Fig. 1

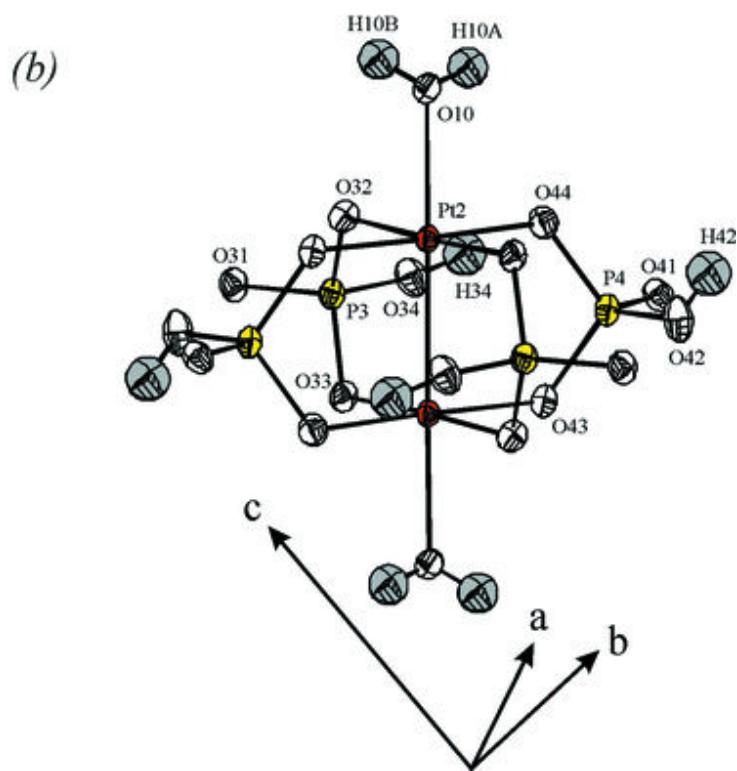
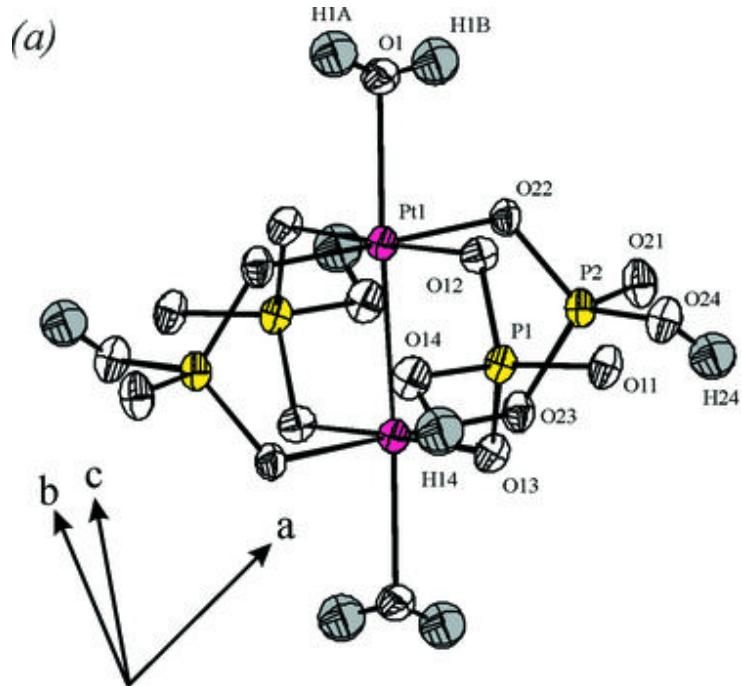
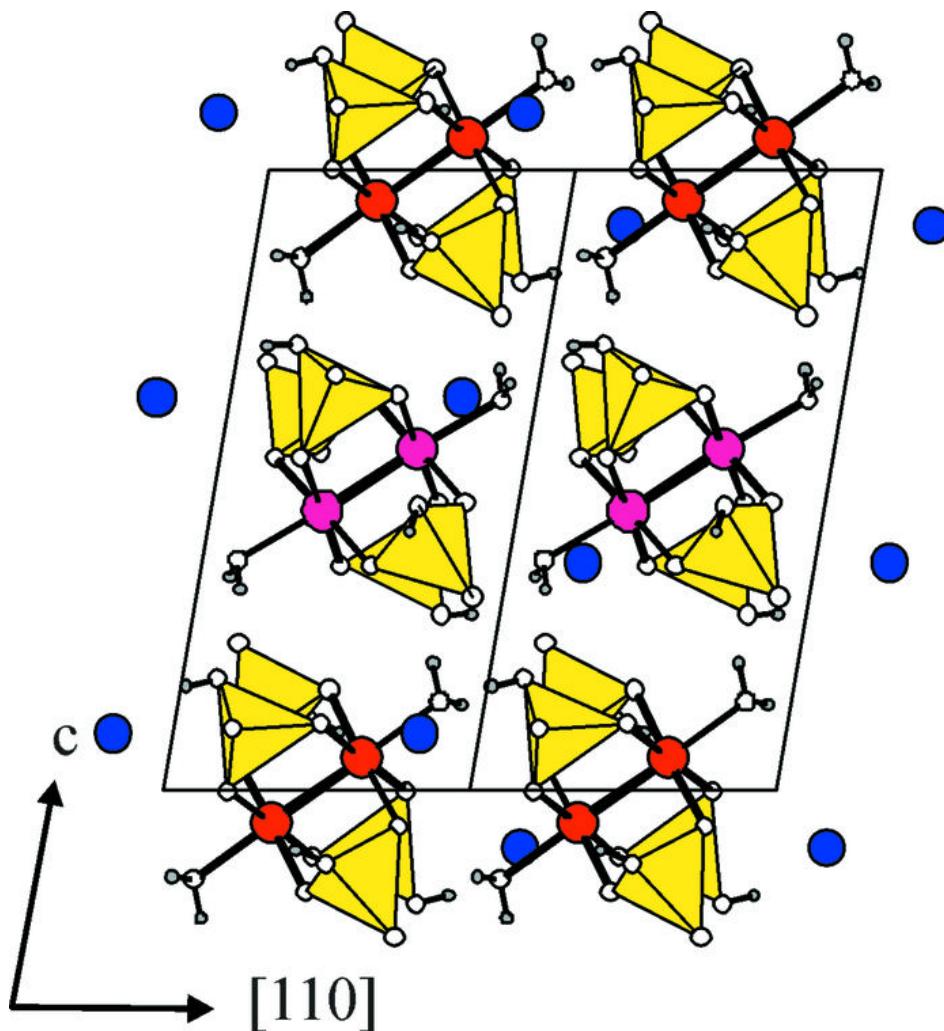


Fig. 2



supplementary materials

Fig. 3

