

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

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

Kosta Panagiotidis and Robert Glaum\*

 Institut für Anorganische Chemie, Rheinische Friedrich-Wilhelms-Universität Bonn,  
 Gerhard-Domagk-Strasse 1, D-53121 Bonn, Germany

Correspondence e-mail: rglau@uni-bonn.de

Received 18 December 2008; accepted 17 February 2009

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

In the crystal structure of the acid platinum phosphate dipotassium di- $\mu$ -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 Pt–Pt distances of 2.4944 (5) and 2.4892 (5) Å. The potassium ions are seven-fold coordinated by hydrogenphosphate groups. In the crystal, O–H...O hydrogen bonds help to establish the packing. The Raman spectrum was recorded.

## Related literature

The structure of the title isotopic sodium compound,  $Na_2[(Pt_2)(HPO_4)_4(H_2O)_2]$ , 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 & Schelokov (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  $In_3(PO_4)_2$  and  $In_2O(PO_4)$ , 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)°
$M_r = 888.32$	$V = 776.32$ (4) Å <sup>3</sup>
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.8852$ (2) Å	Mo $K\alpha$ radiation
$b = 7.9657$ (2) Å	$\mu = 19.05$ mm <sup>-1</sup>
$c = 13.7739$ (4) Å	$T = 123$ K
$\alpha = 82.358$ (1)°	$0.24 \times 0.14 \times 0.08$ mm
$\beta = 81.509$ (1)°	

### Data collection

Nonius Kappa CCD diffractometer	18915 measured reflections
Absorption correction: multi-scan (Blessing, 1995)	5589 independent reflections
$T_{\min} = 0.060$ , $T_{\max} = 0.224$	4077 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.064$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.086$	$\Delta\rho_{\text{max}} = 3.46$ e Å <sup>-3</sup>
$S = 0.98$	$\Delta\rho_{\text{min}} = -2.87$ e Å <sup>-3</sup>
5589 reflections	
259 parameters	
10 restraints	

Table 1

Hydrogen-bond geometry (Å, °).

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

We thank G. Schnakenburg (University of Bonn) for the single-crystal data collection and D. Ernsthäuser (University of Bonn) for the measurement of the Raman spectrum. This work was financially supported by Deutsche Forschungsgemeinschaft (DFG). A noble metal donation by UMICORE AG (Hanau-Wolfgang, Germany) is gratefully acknowledged.

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

## References

- Bancroft, D. P., Cotton, F. A., Falvello, L. R., Han, S. & Schwotzer, W. (1984). *Inorg. Chim. Acta*, **87**, 147–153.
- Baranovskii, I. B. & Schelokov, R. N. (1978). *Russ. J. Inorg. Chem.* **23**, 3–17.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Brauer, G. (1978). In *Handbuch der Präparativen und Anorganischen Chemie*. Stuttgart: Ferdinand Enke.
- Che, C. M., Schaefer, W. P., Gray, H. B., Dickson, M. K., Stein, P. & Roundhill, D. M. (1982). *J. Am. Chem. Soc.* **104**, 4253–4255.
- Cotton, F. A. & Walton, R. A. (1982b). In *Multiple Bonds between Metal Atoms*. New York: Wiley.
- Cotton, F. A., Walton, R. A. & Han, S. (1982a). *Inorg. Chem.* **21**, 1709–1710.
- Dikareva, L. M., Sadikov, G. G., Porai-Koshits, M. A., Baranovskii, I. B., Abdullaev, S. S. & Schelokov, R. N. (1982). *Russ. J. Inorg. Chem.* **27**, 417–423.

- Dikareva, L. M., Zefirov, Yu. V., Zhilyaev, A. N., Baranovskii, I. B. & Porai-Koshits, M. A. (1987). *Russ. J. Inorg. Chem.* **32**, 118–125.
- Dowty, E. (2006). *ATOMS for Windows*. Shape Software, Kingsport, Tennessee, USA.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Hoof, R. W. W. (2004). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Muraveiskaya, G. S., Kukina, G. A., Orlova, V. S., Evstaf'eva, O. N. & Porai-Koshits, M. A. (1976). *Dokl. Akad. Nauk SSSR*, **226**, 596–599.
- Muraveiskaya, G. S., Orlova, V. S. & Evstaf'eva, O. N. (1974). *Russ. J. Inorg. Chem.* **19**, 1030–1035.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Palkina, K. K., Maksimova, S. I., Lavrov, A. V. & Chalisova, N. A. (1978). *Dokl. Akad. Nauk SSSR*, **242**, 829–831.
- Panagiotidis, K., Glaum, R., Schmedt auf der Günne, J., Hoffbauer, W. & Görzel, H. (2005). *Z. Anorg. Allg. Chem.* **631**, 2371–2376.
- Pley, M. & Wickleder, M. S. (2004a). *Z. Anorg. Allg. Chem.* **630**, 1036–1039.
- Pley, M. & Wickleder, M. S. (2004b). *Angew. Chem.* **116**, 4262–4264.
- Pley, M. & Wickleder, M. S. (2005). *Z. Anorg. Allg. Chem.* **631**, 592–595.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Stein, P., Dickson, M. K. & Roundhill, D. M. (1983). *J. Am. Chem. Soc.* **105**, 3489–3494.
- Steiner, T. (2002). *Angew. Chem.* **114**, 50–81.
- Thauern, H. & Glaum, R. (2004). *Z. Anorg. Allg. Chem.* **630**, 2463–2467.
- Wellmann, B. & Liebau, F. (1981). *J. Less Common Met.* **77**, 31–39.
- Woollins, J. D. & Kelly, P. F. (1985). *Coord. Chem. Rev.* **65**, 115–140.

## supporting information

*Acta Cryst.* (2009). E65, i18–i19 [doi:10.1107/S1600536809005601]

## Synthesis, crystal structure and Raman spectrum of $\text{K}_2[(\text{Pt}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$ containing $(\text{Pt}_2)^{6+}$ ions

Kosta Panagiotidis and Robert Glaum

### S1. 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  $\text{Pd}^{\text{II}}\text{P}_2\text{O}_7$  (Panagiotidis *et al.*, 2005) in addition to the already known  $\text{Pd}(\text{PO}_3)_2$  (Palkina *et al.*, 1978). In this context we were also interested in the crystal chemistry of platinum phosphates. Up to now,  $\text{Pt}^{\text{IV}}\text{P}_2\text{O}_7$  (Wellmann & Liebau, 1981) is the only structurally characterized anhydrous phosphate of platinum. Since reactions starting from " $\text{PtO} \times 3\text{H}_2\text{O}$ " and  $\text{P}_4\text{O}_{10}$  did not yield products suitable for closer investigation we tried an alternative synthetic approach by reacting  $\text{K}_2\text{PtCl}_4$  with conc.  $\text{H}_3\text{PO}_4$  (see Experimental). This led to the formation of orange crystals of  $\text{K}_2[(\text{Pt}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$ . The acid phosphate is isotypic to the sodium compound (Cotton *et al.*, 1982a). In contrast to the structure of  $\text{Na}_2[(\text{Pt}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$  no disordered oxygen atoms are observed in the potassium compound. Distances  $d(\text{Pt}—\text{Pt})$  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  $\text{K}_2[(\text{Pt}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$ . The  $(\text{Pt}_2)^{6+}$  binuclear complex was first observed in the crystal structure of  $\text{K}_2[(\text{Pt}_2)(\text{SO}_4)_4(\text{H}_2\text{O})_2]$  (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  $\text{K}_2[(\text{Pt}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]$  two crystallographically equivalent platinum atoms are connected to form  $(\text{Pt}_2)^{6+}$  dinuclear complexes with surrounding  $(\text{HPO}_4)^{2-}$  groups (Fig. 1). The ligating oxygen atoms of the  $(\text{HPO}_4)^{2-}$  ions are arranged in a square-planar coordination around each platinum atom. Distances  $d(\text{Pt}—\text{O})$  range from 1.978 Å to 2.030 Å. Angles  $\langle(\text{O}, \text{Pt}, \text{O})$  are deviating only slightly from  $90^\circ$  and  $180^\circ$ , respectively. Due to their different crystal chemical environment, the oxygen atoms of the hydrogenphosphate anions show significantly different bond lengths  $d(\text{P}—\text{O})$ . Oxygen atoms attached to platinum (coordination number of oxygen c.n.(O) = 2 (P, Pt)) show distances  $d(\text{P}—\text{O}) = 1.55$  Å. Distances  $d(\text{P}—\text{O})$  for those oxygen atoms which are coordinated to  $\text{K}^+$  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  $(\text{HPO}_4)^{2-}$  unit (c.n.(O) = 2, (P, H)), distances  $d(\text{P}—\text{OH})$  around 1.565 Å are observed. The axial ligand positions of the  $\text{Pt}_2$  dumbbells are occupied by water molecules (Fig. 1 & 2). Distances  $d(\text{Pt}—\text{O}) = 2.135$  Å observed for the water ligands are significantly longer than those within the  $[\text{Pt}_2\text{O}_8]$  entity. The hydrogen atoms of  $[\text{HPO}_4]$  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  $[(\text{Pt}_2)(\text{HPO}_4)_4(\text{H}_2\text{O})_2]^{2-}$  units. Interatomic distances  $d(\text{OH} \cdots \text{OP})$  range from 1.73 Å to 2.27 Å. They are in good accordance with those observed for strong hydrogen bonds (Steiner, 2002).

As found for various other compounds containing the  $(\text{Pt}_2)^{6+}$  dinuclear complex, the angle  $\langle(\text{Pt}, \text{Pt}, \text{O})$  between the dumbbell and the axial oxygen atoms deviates only slightly from  $180^\circ$  (Cotton *et al.*, 1982b; Pley & Wickleder *et al.*,

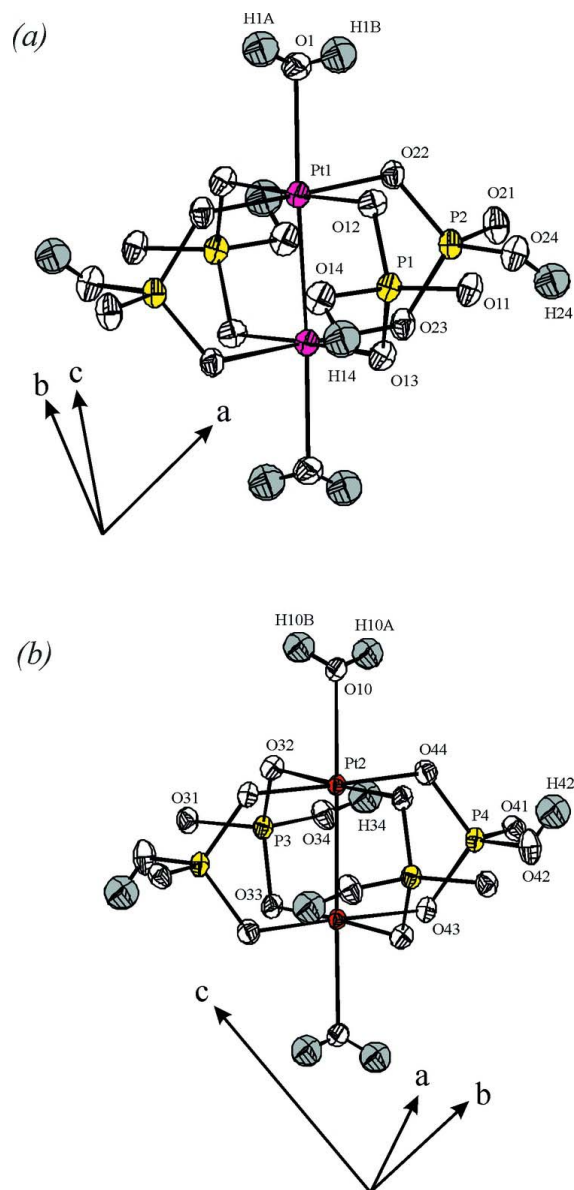
2005). [HPO<sub>4</sub>] tetrahedra in [(Pt<sub>2</sub>)(HPO<sub>4</sub>)(H<sub>2</sub>O)<sub>2</sub>]<sup>2-</sup> show no significant angular distortion. Charge compensation of the anionic [Pt<sup>III</sup><sub>2</sub>(HPO<sub>4</sub>)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>]<sup>2-</sup> unit is achieved by two crystallographically independent K<sup>+</sup> 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 [Pt<sup>III</sup><sub>2</sub>(HPO<sub>4</sub>)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>]<sup>2-</sup> (Fig. 3). An unequivocal assignment of the observed signals is yet impossible. Comparison of the Raman spectrum to those of the In<sub>2</sub><sup>4+</sup> containing indium phosphates In<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> and In<sub>2</sub>O(PO<sub>4</sub>) (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 [(Pt<sup>III</sup><sub>2</sub>)L<sub>4</sub>L'<sub>2</sub>]<sup>n-</sup> (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 K<sub>2</sub>[(Pt<sub>2</sub>)(HPO<sub>4</sub>)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>] 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).

## S2. Experimental

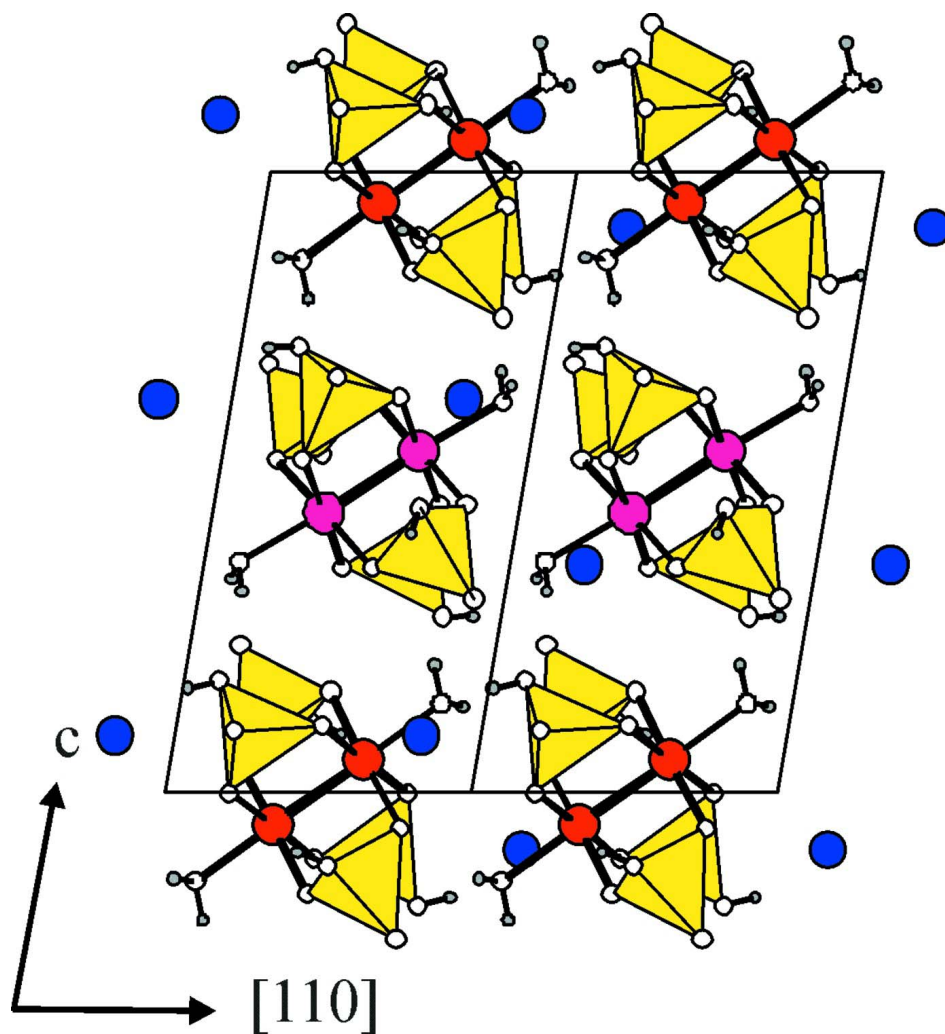
Aiming at the crystallization of "Pt<sub>2</sub>P<sub>2</sub>O<sub>7</sub>" a reaction starting from 150.0 mg K<sub>2</sub>Pt<sup>II</sup>Cl<sub>4</sub>, which were dissolved in water and mixed with 5.0 ml conc. H<sub>3</sub>PO<sub>4</sub>, was performed. After the obtained red solution was kept in a desiccator over P<sub>2</sub>O<sub>5</sub> for two weeks, plate-like, orange crystals of K<sub>2</sub>[(Pt<sub>2</sub>)(HPO<sub>4</sub>)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>] with edge-lengths up to 0.3 mm were deposited besides microcrystalline platinum (eq. 1). The synthesis of K<sub>2</sub>[Pt<sup>III</sup>Cl<sub>4</sub>] was performed according to the procedure given by Brauer (1978).





**Figure 1**

Pa ddle-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).

**Figure 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),  $\text{Pt}_2^{6+}$  (red),  $\text{K}^+$  (violet),  $\text{H}^+$  (blue),  $\text{O}^{2-}$  (white) (Progr. *ATOMS* v.6.2).

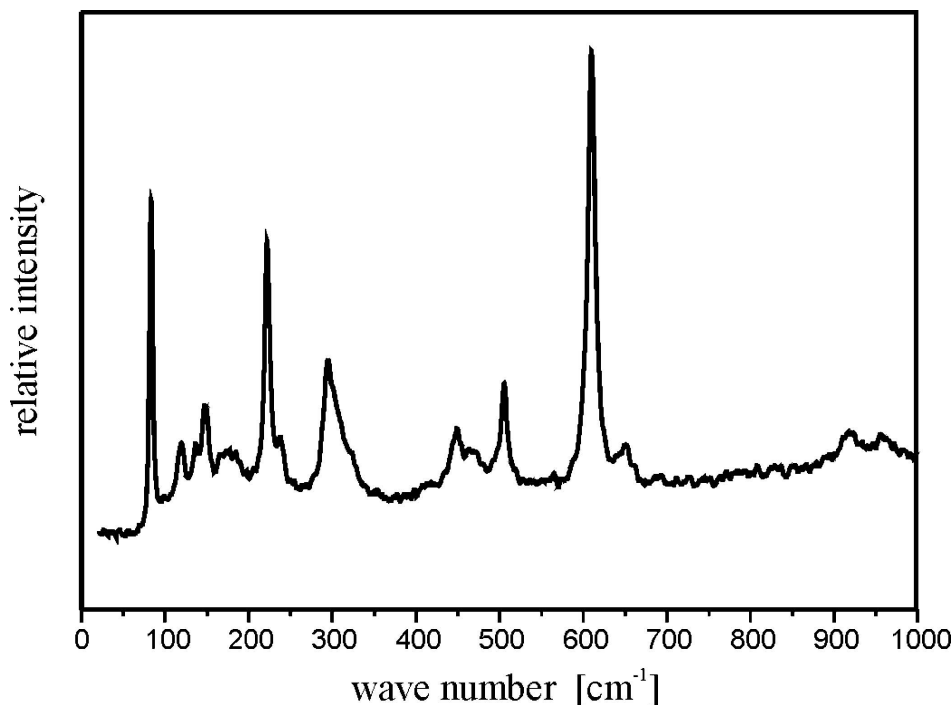


Figure 3

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

**dipotassium di- $\mu$ -hydrogenphosphato-bis[aquaplatinum(III)](*Pt—Pt*)**

*Crystal data*

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

$M_r = 888.32$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

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

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

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

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

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

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

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

$Z = 2$

$F(000) = 812$

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.

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

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5589 reflections

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

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

$T = 123 \text{ K}$

Plate, orange

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

*Data collection*

Nonius Kappa CCD  
diffractometer

Radiation source: Mo $K\alpha$

Graphite monochromator

Detector resolution: 9 pixels  $\text{mm}^{-1}$

CCD rotation images scans

Absorption correction: multi-scan  
(Blessing, 1995)

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

18915 measured reflections

5589 independent reflections

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

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

$\theta_{\max} = 32.5^\circ$ ,  $\theta_{\min} = 1.5^\circ$

$h = -11 \rightarrow 11$

$k = -12 \rightarrow 12$

$l = -19 \rightarrow 20$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.033$   
 $wR(F^2) = 0.086$   
 $S = 0.98$   
 5589 reflections  
 259 parameters  
 10 restraints  
 0 constraints  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0423P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 3.46 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -2.87 \text{ e } \text{\AA}^{-3}$

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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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)

*Geometric parameters (Å, °)*

K1—O24 <sup>i</sup>	2.739 (6)	O24—P2	1.570 (5)
K1—O31 <sup>ii</sup>	2.860 (5)	O21—P2	1.489 (5)
K1—O11 <sup>i</sup>	2.957 (6)	O23—P2	1.549 (5)
K1—O42 <sup>iii</sup>	3.047 (6)	O23—Pt1 <sup>iv</sup>	2.014 (5)
K1—O22 <sup>iv</sup>	3.053 (5)	O10—Pt2	2.132 (5)

K1—O32 <sup>ii</sup>	3.156 (5)	O32—P3	1.544 (5)
K1—O12 <sup>i</sup>	3.222 (6)	O32—Pt2	1.978 (4)
K2—O14	2.735 (6)	O33—P3	1.561 (5)
K2—O34 <sup>v</sup>	2.822 (6)	O33—Pt2 <sup>vii</sup>	2.030 (4)
K2—O41 <sup>v</sup>	2.858 (5)	O34—P3	1.564 (5)
K2—O33 <sup>ii</sup>	2.920 (5)	O31—P3	1.507 (4)
K2—O42 <sup>iii</sup>	2.990 (6)	O44—P4	1.555 (5)
K2—O43 <sup>vi</sup>	3.000 (5)	O44—Pt2	2.005 (5)
K2—O44 <sup>iii</sup>	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 <sup>vii</sup>	2.014 (4)
O12—Pt1	1.992 (4)	O42—P4	1.541 (5)
O14—P1	1.564 (5)	Pt1—O13 <sup>iv</sup>	2.010 (4)
O11—P1	1.493 (5)	Pt1—O23 <sup>iv</sup>	2.014 (5)
O13—P1	1.549 (5)	Pt1—Pt1 <sup>iv</sup>	2.4944 (5)
O13—Pt1 <sup>iv</sup>	2.010 (4)	Pt2—O43 <sup>vii</sup>	2.014 (4)
O22—P2	1.541 (5)	Pt2—O33 <sup>vii</sup>	2.030 (4)
O22—Pt1	1.985 (4)	Pt2—Pt2 <sup>vii</sup>	2.4892 (5)
O11—P1—O12	110.2 (3)	O22—Pt1—O23 <sup>iv</sup>	179.02 (19)
O11—P1—O13	110.9 (3)	O12—Pt1—O23 <sup>iv</sup>	90.37 (19)
O12—P1—O13	111.5 (3)	O13 <sup>iv</sup> —Pt1—O23 <sup>iv</sup>	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 <sup>iv</sup> —Pt1—O1	90.0 (2)
O21—P2—O22	111.1 (3)	O23 <sup>iv</sup> —Pt1—O1	93.6 (2)
O21—P2—O23	113.2 (3)	O22—Pt1—Pt1 <sup>iv</sup>	90.98 (14)
O22—P2—O23	109.6 (3)	O12—Pt1—Pt1 <sup>iv</sup>	91.86 (14)
O21—P2—O24	106.9 (3)	O13 <sup>iv</sup> —Pt1—Pt1 <sup>iv</sup>	90.05 (14)
O22—P2—O24	107.7 (3)	O23 <sup>iv</sup> —Pt1—Pt1 <sup>iv</sup>	89.91 (15)
O23—P2—O24	108.1 (3)	O1—Pt1—Pt1 <sup>iv</sup>	176.49 (14)
O31—P3—O32	108.9 (3)	O32—Pt2—O44	90.05 (19)
O31—P3—O33	109.3 (3)	O32—Pt2—O43 <sup>vii</sup>	89.88 (18)
O32—P3—O33	111.8 (3)	O44—Pt2—O43 <sup>vii</sup>	178.49 (18)
O31—P3—O34	111.6 (3)	O32—Pt2—O33 <sup>vii</sup>	177.57 (17)
O32—P3—O34	106.3 (3)	O44—Pt2—O33 <sup>vii</sup>	90.65 (19)
O33—P3—O34	108.9 (3)	O43 <sup>vii</sup> —Pt2—O33 <sup>vii</sup>	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 <sup>vii</sup> —Pt2—O10	89.45 (19)
O41—P4—O44	110.6 (3)	O33 <sup>vii</sup> —Pt2—O10	90.51 (18)
O42—P4—O44	106.4 (3)	O32—Pt2—Pt2 <sup>vii</sup>	91.55 (13)
O43—P4—O44	109.7 (2)	O44—Pt2—Pt2 <sup>vii</sup>	90.52 (13)
O22—Pt1—O12	89.21 (19)	O43 <sup>vii</sup> —Pt2—Pt2 <sup>vii</sup>	90.99 (13)
O22—Pt1—O13 <sup>iv</sup>	90.42 (19)	O33 <sup>vii</sup> —Pt2—Pt2 <sup>vii</sup>	90.77 (13)
O12—Pt1—O13 <sup>iv</sup>	178.06 (19)	O10—Pt2—Pt2 <sup>vii</sup>	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 (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>
O1—H1 <i>A</i> $\cdots$ O31 <sup>v</sup>	0.83 (6)	1.73 (6)	2.559 (7)	173 (8)
O1—H1 <i>B</i> $\cdots$ O21 <sup>viii</sup>	0.84 (6)	2.09 (4)	2.860 (7)	153 (8)
O14—H14 $\cdots$ O31 <sup>ii</sup>	0.83 (2)	1.82 (5)	2.548 (7)	146 (9)
O24—H24 $\cdots$ O11 <sup>ix</sup>	0.84 (7)	1.73 (7)	2.562 (7)	177 (9)
O34—H34 $\cdots$ O41 <sup>x</sup>	0.83 (6)	1.74 (3)	2.546 (6)	162 (9)
O10—H10 <i>A</i> $\cdots$ O41 <sup>xi</sup>	0.86 (2)	1.94 (4)	2.713 (7)	148 (7)
O10—H10 <i>B</i> $\cdots$ O11 <sup>iv</sup>	0.86 (2)	1.86 (2)	2.694 (6)	164 (6)
O42—H42 $\cdots$ O21 <sup>xii</sup>	0.84 (7)	2.27 (9)	2.475 (7)	94 (6)

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