Tabelle 2. *Os—Sn-Abstände in den $OsSn_6$-Oktaedern (Å)*

Os1—Sn1	2,5604 (13)	Os2—$Sn10^{iv}$	2,5503 (9)
Os1—Sn2	2,5639 (13)	Os2—Sn9	2,5911 (9)
Os1—$Sn3^{i}$	2,5820 (9)	Os2—$Sn9^{ii}$	2,5911 (9)
Os1—$Sn3^{ii}$	2,5820 (9)	Os3—Sn5	2,5580 (13)
Os1—$Sn7^{ii}$	2,6323 (9)	Os3—$Sn9^{v}$	2,5969 (10)
Os1—$Sn7^{i}$	2,6323 (9)	Os3—$Sn9^{vi}$	2,5969 (10)
Os2—Sn8	2,5311 (13)	Os3—$Sn7^{ii}$	2,6010 (10)
Os2—Sn4	2,5439 (13)	Os3—Sn7	2,6010 (10)
Os2—$Sn10^{iii}$	2,5503 (9)	Os3—$Sn6^{vii}$	2,6031 (12)

Symmetriebezeichnungen: (i) $x, y-1, z$; (ii) $x, 1-y, z$; (iii) $x-\frac{1}{2}, \frac{3}{2}-y, z$; (iv) $x-\frac{1}{2}, y-\frac{1}{2}, z$; (v) $\frac{1}{2}+x, \frac{1}{2}+y, 1+z$; (vi) $\frac{1}{2}+x, \frac{1}{2}-y, 1+z$; (vii) $x, y, 1+z$.

Bei der Verfeinerung der Struktur als racemischer Zwilling wurde ein Verhältnis der beiden Individuen von 92 (2):8 (2) berechnet. Deshalb kann man davon ausgehen, daß es sich um ein einzelnes Individuum handelt, das die bestimmte absolute Struktur besitzt. Die erhaltene höchste Restelektronendichte von 6,161 e Å^{-3} befindet sich 0,6 Å neben Os3, die niedrigste Restelektronendichte von −6,305 e Å^{-3} liegt 2,5 Å von O5 entfernt. Weitere Einzelheiten zur Kristallstrukturuntersuchung können auch beim Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, unter Augabe der Hinterlegungsnummer CSD-405047 angefordert werden.

Datensammlung: *CAD*-4 *Software* (Enraf–Nonius, 1994). Gitterverfeinerung: *CAD*-4 *Software*. Datenreduktion: *XCAD4-PC* (Enraf–Nonius, 1994). Strukturlösungsprogramm: *SHELXS*86 (Sheldrick, 1985). Verfeinerungsprogramm: *SHELXL*93 (Sheldrick, 1993). Zeichenprogramme: *ATOMS* (Dowty, 1994) und *ORTEP*II (Johnson, 1976).

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Die Listen der Strukturfaktoren, anisotropen Verschiebungsparameter und vollständigen geometrischen Daten sind bei der IUCr (Aktenzeichen: SK1025) hinterlegt. Kopien sind erhältlich durch: The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Triclinic Room-Temperature Modification of $K_2MgWO_2(PO_4)_2$

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Abstract

$K_2MgWO_2(PO_4)_2$ [dipotassium magnesium tungsten dioxide bis(phosphate); abbreviated formula KMgWP], a new compound crystal-chemically derivable from the well known nonlinear optical crystal $KTiOPO_4$ (KTP), undergoes at least five phase transitions between its melting point ($T_m \simeq 1068$ K) and room temperature. KMgWP's triclinic room-temperature structure (space group $P1$) can be described as a slightly distorted modification of its tetragonal high-temperature phase ($T >$ 782 K), which is isostructural with $K_2NiWO_2(PO_4)_2$ (KNiWP, space group $P4_{1,3}2_12$).

Comment

For nearly twenty years, crystals of $KTiOPO_4$ (potassium titanyl phosphate, KTP, space group $Pna2_1$) have been of great interest in the field of laser optics because of their outstanding nonlinear optical, chemical and mechanical properties. Furthermore, the great variability of KTP's crystal structure has allowed the synthesis of a great number of isotypic compounds, mainly by a simple exchange of either K, Ti or P by crystal-chemically related substituents (Stucky, Phillips & Gier, 1989).

The replacement of the two symmetrically unrelated Ti^{4+} ions in the structure of KTP with two cations of different valencies ($2Ti^{4+} \rightarrow M^{2+} + W^{6+}$) has led to a new family of compounds with the general formula $K_2M^{II}WO_2(PO_4)_2$ (M^{II} = Mg, Ni, Co; $KM^{II}WP$) (Wostrack, 1993). All these compounds, including the recently synthesized new members with M^{II} = Fe, Mn and Cd, are structurally very close to one another, but are not isostructural with KTP (Peuchert & Bohatý, 1995).

For nearly every member, at least one reversible structural phase transition has been detected using thermoanalytical, X-ray powder diffraction and optical methods. The only compound that does not undergo any phase transition within the investigated temperature range ($100 < T < 820$ K) is $K_2NiWO_2(PO_4)_2$ (KNiWP).

The crystal structure of KNiWP was refined in space group $P4_12_12$ and shows a significant metric and structural relationship to KTP (Peuchert, Bohatý & Fröhlich, 1995).

$K_2MgWO_2(PO_4)_2$ (KMgWP) exhibits at least five phase transitions (PT1–PT5) near T_{PT1-5} = 436, 535, 537, 635 and 782 K, respectively. PT1, PT2 and PT3 are the ferroic kind, characterized by polydomain twinning of crystals when they are cooled below the transition points. PT4 and PT5 were detected by anomalous thermal behaviour of the electrical conductivity. Symmetry relationships between the six modifications of KMgWP were determined by temperature-dependent X-ray powder investigations; all low-temperature phases can be described as slightly distorted modifications of a tetragonal prototypic phase which is stable above T_{PT5} = 782 K and is isostructural with KNiWP (Peuchert, 1995).

In order to study the structural background responsible for the phase transitions in KMgWP, a series of X-ray investigations on single crystals and powders at different temperatures have been undertaken. One of these results, *i.e.* the crystal structure of KMgWP's low-temperature modification [T = 293 (2) K], is presented here.

At room temperature, almost all crystals synthesized by spontaneous crystallization of a non-stoichiometric melt were twinned as a result of the phase transitions. Nevertheless, one nearly untwinned fragment suitable for data collection was found in the polycrystalline aggregate using a polarization microscope. The correct choice was confirmed by the successful indexing of 25 reflections distributed over various parts of reciprocal space. At room temperature, KMgWP is triclinic (but pseudo-tetragonal) [space group $P1$; a = 9.141 (2), b = 9.160 (2), c = 10.736 (4) Å; α = 90.28 (2), β = 90.52 (2), γ = 90.11 (2)°]. X-ray powder-diffraction and crystal-optical investigations confirmed the triclinic symmetry. Nearly extinct reflections of type $00l$ with $l \neq 4n$, and $h00$ and $0k0$ with $h,k \neq 2n$ signal pseudosymmetry according to space group $P4_{1,3}2_12$.

The crystal structure of KMgWP consists of [WO_6] and [MgO_6] octahedra alternately linked by the apical O5x (x = 1–8) atoms forming helical chains along [001]. Similar to the [TiO_6] octahedra in $KTiOPO_4$ (Tordjman, Masse & Guitel, 1974), the [WO_6] octahedra are quite distorted, containing three different kinds of bond lengths [Wy—O5x 1.741 (4)–1.758 (4), Wy—O2x 1.957 (4)–1.993 (4) and Wy—O1x 2.069 (4)–2.110 (4) Å; y = 1–4]. In comparison, the [MgO_6] octahedra are less distorted [Mg—O distance range 1.977 (5)–2.158 (5) Å]. The bridging O5x atoms are in *cis* positions relative to the W and Mg atoms, respectively. In contrast, the corresponding titanyl O atoms in the KTP structure are in alternate *cis–trans* positions (Peuchert, Bohatý & Fröhlich, 1995). The octahedral chains are connected *via* slightly distorted [PO_4] tetrahedra [P—O distance range 1.495 (5)–1.598 (4) Å] forming a three-dimensional network (Fig. 1). The bridging O5x atoms do not belong to the phosphate groups.

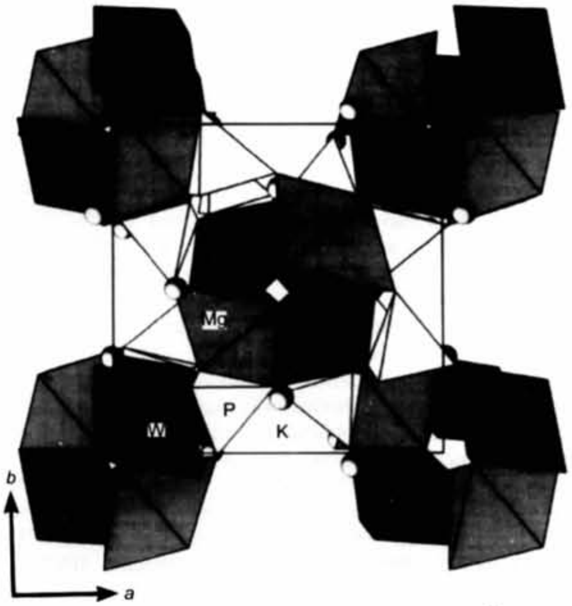

Fig. 1. Perspective view of the title compound along [00$\bar{1}$] (*ATOMS*; Dowty, 1989).

The potassium ions are situated in large prismatic cavities within the oxygen framework. The cavities are connected by common edges around the pseudo-4_1 axes at ($\frac{1}{2}$,0,z) and (0,$\frac{1}{2}$,z) [Fig. 2(a)]. Each cavity contains two positions, $p1$ and $p2$, surrounded in nearly the same manner by eight to nine O atoms; there are in total 13 O atoms [Fig. 2(b)]. A preference in the occupation of $p1$ and $p2$ by the eight K^+ ions in the unit cell cannot

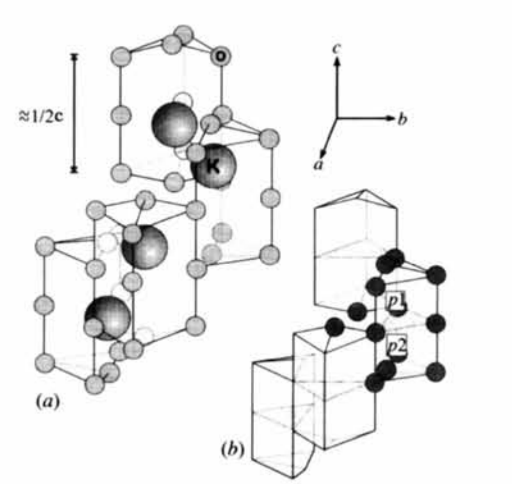

Fig. 2. Schematic representations showing (a) four of the eight potassium ions in the unit cell situated in large prismatic cavities which are connected by common edges along [001], and (b) each cavity containing two positions, $p1$ and $p2$, surrounded by 13 O atoms.

be observed. The bond lengths between potassium and the 13 surrounding O atoms vary between 2.588 (5) and 4.682 (5) Å.

A comparison of the structures of different modifications of KMgWP indicates that K^+ is most affected by the observed phase transitions (Peuchert, 1995). A detailed discussion of these results will be the subject of a future paper.

Experimental

Crystals of KMgWP used for structural investigations were grown from a K_2WO_4–WO_3 flux of eutectic composition (K_2WO_4/WO_3 molar ratio 0.53/0.47). A stoichiometric mixture of KH_2PO_4, MgO and WO_3 (analytical grade) and the tungstate flux (compound/flux molar ratio 2.5/1) was ground, transferred into a platinum crucible and heated to 1120 K. After 1 d at this temperature, the homogenized viscous melt was cooled to 990 K at a constant rate of 0.5 K h^{-1} followed by a more rapid cooling to room temperature (cooling rate 20 K h^{-1}). The experiment led to a nearly glass-free crystallizate consisting of colourless hypidiomorphic KMgWP crystals (a few mm in size) accompanied by an additional unknown colourless crystalline phase. Intensity data were collected on an untwinned KMgWP fragment which was selected using a polarization microscope. Large optically perfect single crystals up to 2 × 3 × 3 cm suitable for physical measurements were grown by a modified Nacken–Kyropoulos technique from a stoichiometric melt (Peuchert, 1995).

Crystal data

$K_2MgWO_2(PO_4)_2$
$M_r = 508.30$
Triclinic
$P1$
$a = 9.141$ (2) Å
$b = 9.160$ (2) Å
$c = 10.736$ (4) Å
$\alpha = 90.28$ (2)°
$\beta = 90.52$ (2)°
$\gamma = 90.11$ (2)°
$V = 898.8$ (4) Å^3
$Z = 4$
$D_x = 3.756$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11.70$–18.11°
$\mu = 14.245$ mm^{-1}
$T = 293$ (2) K
Fragment
0.25 × 0.20 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*SDP*; Frenz, 1978)
$T_{\min} = 0.144$, $T_{\max} = 0.241$
10 336 measured reflections
10 336 independent reflections

10 025 observed reflections [$I > 2\sigma(I)$]
$\theta_{\max} = 31.96$°
$h = -13 \rightarrow 7$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 15$
3 intensity reflections monitored every 120 min; 1 orientation reflection monitored every 250 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.021$
$wR(F^2) = 0.051$
$S = 1.071$
10 336 reflections
578 parameters
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 0.64P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 2.35$ e Å^{-3}
$\Delta\rho_{\min} = -1.99$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0270 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = −0.021 (4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
W1	0.11895 (2)	0.10075 (2)	−0.00645 (2)	0.00534 (4)
W2	0.89408 (2)	0.89398 (2)	0.50913 (2)	0.00543 (5)
W3	0.38145 (2)	0.60938 (2)	0.25272 (2)	0.00530 (4)
W4	0.60651 (2)	0.39477 (2)	0.74482 (2)	0.00539 (4)
Mg1	0.6471 (2)	0.6284 (2)	0.0132 (2)	0.0069 (4)
Mg2	0.3508 (3)	0.3521 (2)	0.4948 (2)	0.0084 (4)
Mg3	0.8626 (2)	0.1266 (2)	0.2414 (2)	0.0072 (4)
Mg4	0.1579 (2)	0.8410 (2)	0.7511 (2)	0.0084 (4)
P1	0.3405 (2)	0.82176 (14)	0.00974 (13)	0.0065 (3)
P2	0.6751 (2)	0.18337 (15)	0.49578 (13)	0.0062 (3)
P3	0.6766 (2)	0.81360 (14)	0.26911 (13)	0.0061 (3)
P4	0.3302 (2)	0.15736 (14)	0.74267 (12)	0.0057 (2)
P5	0.1724 (2)	0.31858 (15)	0.23321 (13)	0.0060 (3)
P6	0.8356 (2)	0.68145 (14)	−0.23994 (13)	0.0060 (2)
P7	0.8254 (2)	0.31200 (15)	−0.01302 (13)	0.0062 (3)
P8	0.1665 (2)	0.65805 (14)	0.49628 (13)	0.0061 (2)
K1	0.9927 (2)	0.7226 (2)	0.0570 (2)	0.0261 (3)
K2	0.0012 (2)	0.3320 (2)	−0.3212 (2)	0.0347 (4)
K3	0.8428 (2)	0.5008 (2)	0.3458 (2)	0.0320 (4)
K4	0.2523 (3)	0.4764 (2)	−0.0626 (2)	0.0336 (4)
K5	0.5055 (2)	0.1869 (2)	0.1948 (2)	0.0327 (4)
K6	0.5174 (2)	0.7637 (3)	0.5753 (2)	0.0430 (5)
K7	0.6640 (2)	0.9988 (2)	−0.1278 (3)	0.0437 (6)
K8	0.2650 (2)	0.00351 (15)	0.4458 (2)	0.0236 (3)
O11	0.2283 (5)	0.2490 (4)	0.1092 (4)	0.0087 (8)
O12	0.7866 (5)	0.7850 (5)	0.6549 (4)	0.0119 (8)
O13	0.2410 (5)	0.7136 (4)	0.3764 (4)	0.0104 (8)
O14	0.7517 (5)	0.2792 (4)	0.8588 (4)	0.0094 (8)
O15	0.2799 (5)	0.7485 (5)	0.1278 (4)	0.0108 (8)
O16	0.7211 (5)	0.2825 (5)	0.6076 (4)	0.0122 (8)
O17	0.2417 (5)	0.2091 (4)	−0.1432 (4)	0.0111 (8)
O18	0.7441 (5)	0.7783 (4)	0.3989 (4)	0.0089 (8)
O21	0.2993 (5)	−0.0112 (4)	0.0235 (4)	0.0086 (7)
O22	0.7293 (5)	0.0263 (4)	0.5370 (4)	0.0106 (8)
O23	0.5090 (5)	0.7803 (4)	0.2873 (4)	0.0090 (7)
O24	0.4914 (5)	0.2149 (4)	0.7691 (4)	0.0097 (8)
O25	0.2070 (5)	0.4870 (4)	0.2162 (4)	0.0087 (7)
O26	0.7715 (5)	0.5267 (4)	0.7168 (4)	0.0106 (8)
O27	−0.0092 (5)	0.2711 (4)	−0.0384 (4)	0.0087 (7)
O28	0.0030 (5)	0.7085 (4)	0.4816 (4)	0.0099 (8)
O31	0.7630 (5)	0.2108 (5)	0.0841 (4)	0.0130 (8)
O32	0.2378 (5)	0.7382 (5)	0.6044 (4)	0.0112 (8)
O33	0.2656 (6)	0.2588 (4)	0.3392 (4)	0.0109 (8)
O34	0.7633 (6)	0.7288 (4)	−0.1204 (4)	0.0112 (8)
O35	0.7387 (5)	0.7096 (5)	0.1738 (4)	0.0110 (8)
O36	0.2655 (5)	0.2311 (4)	0.6279 (4)	0.0097 (8)
O37	0.2570 (6)	0.7566 (4)	0.8996 (4)	0.0126 (9)
O38	0.7556 (5)	0.2306 (4)	0.3803 (4)	0.0107 (8)
O41	0.8159 (5)	0.4714 (4)	0.0214 (4)	0.0106 (8)
O42	0.1688 (5)	0.4943 (4)	0.5068 (4)	0.0104 (8)
O43	0.0102 (5)	0.2985 (4)	0.2486 (4)	0.0106 (8)

O44	−0.0003 (5)	0.6698 (5)	0.7724 (4)	0.0116 (8)
O45	0.6961 (5)	−0.0283 (4)	0.2365 (4)	0.0113 (8)
O46	0.3352 (5)	0.9932 (4)	0.7299 (4)	0.0106 (8)
O47	0.5028 (5)	0.8073 (4)	0.0001 (4)	0.0100 (8)
O48	0.5106 (5)	0.1808 (5)	0.4771 (4)	0.0118 (8)
O51	0.0153 (5)	0.0332 (4)	0.1166 (4)	0.0100 (8)
O52	0.0232 (5)	0.9554 (4)	0.6176 (4)	0.0099 (8)
O53	0.4471 (5)	0.4998 (4)	0.3732 (4)	0.0105 (8)
O54	0.5471 (5)	0.4966 (4)	0.8735 (4)	0.0093 (8)
O55	0.4875 (5)	0.5376 (4)	0.1332 (4)	0.0096 (8)
O56	0.4834 (5)	0.4576 (4)	0.6323 (4)	0.0104 (8)
O57	0.0554 (5)	−0.0188 (4)	−0.1217 (4)	0.0112 (8)
O58	0.9582 (5)	0.9902 (5)	0.3792 (4)	0.0115 (8)

Table 2. *Selected geometric parameters (Å, °)*

W1—O*pq*	1.743 (4)–2.107 (4)	Mg1—O*pq*	2.017 (5)–2.126 (5)
W2—O*pq*	1.741 (4)–2.110 (4)	Mg2—O*pq*	1.978 (5)–2.155 (5)
W3—O*pq*	1.742 (4)–2.084 (4)	Mg3—O*pq*	2.026 (5)–2.124 (5)
W4—O*pq*	1.744 (4)–2.090 (4)	Mg4—O*pq*	1.977 (5)–2.158 (5)
p = 1, 2, 5; *q* = 1–8		*p* = 3–5; *q* = 1–8	
P1—O*pq*	1.495 (5)–1.583 (4)	K1—O*pq*	2.651 (5)–4.561 (5)
P2—O*pq*	1.512 (5)–1.586 (4)	K2—O*pq*	2.649 (5)–4.629 (5)
P3—O*pq*	1.503 (4)–1.576 (5)	K3—O*pq*	2.624 (5)–4.539 (5)
P4—O*pq*	1.510 (4)–1.587 (5)	K4—O*pq*	2.594 (5)–4.682 (5)
P5—O*pq*	1.505 (5)–1.586 (4)	K5—O*pq*	2.655 (5)–4.579 (5)
P6—O*pq*	1.509 (5)–1.598 (4)	K6—O*pq*	2.588 (5)–4.579 (5)
P7—O*pq*	1.507 (4)–1.583 (5)	K7—O*pq*	2.637 (5)–4.309 (5)
P8—O*pq*	1.505 (4)–1.572 (5)	K8—O*pq*	2.609 (4)–4.549 (5)
p = 1–4; *q* = 1–8		*p* = 1–5; *q* = 1–8	
O*pq*—W1—O*rs*	79.8 (2)–100.2 (2) 159.1 (2)–172.5 (2)	O*pq*—Mg1—O*rs*	84.7 (2)–102.8 (2) 165.7 (2)–171.7 (2)
O*pq*—W2—O*rs*	79.2 (2)–102.6 (2) 159.5 (2)–171.7 (2)	O*pq*—Mg2—O*rs*	83.1 (2)–102.2 (2) 166.8 (2)–172.3 (2)
O*pq*—W3—O*rs*	81.7 (2)–98.0 (2) 162.0 (2)–171.9 (2)	O*pq*—Mg3—O*rs*	84.0 (2)–102.3 (2) 165.8 (2)–173.5 (2)
O*pq*—W4—O*rs*	78.5 (2)–100.4 (2) 161.4 (2)–168.2 (2)	O*pq*—Mg4—O*rs*	83.0 (2)–106.5 (2) 167.8 (2)–173.4 (2)
p, *r* = 1, 2, 5; *q*, *s* = 1–8		*p*, *r* = 3–5; *q*, *s* = 1–8	
W*p*—O5*q*—Mg*r*	131.9 (2)–136.1 (2)	O*pq*—P1—O*rs*	105.0 (2)–113.7 (3)
p, *r* = 1–4; *q* = 1–8		O*pq*—P2—O*rs*	103.3 (3)–112.7 (3)
		O*pq*—P3—O*rs*	103.1 (2)–113.7 (3)
		O*pq*—P4—O*rs*	104.5 (3)–113.0 (2)
		O*pq*—P5—O*rs*	103.2 (2)–114.8 (3)
		O*pq*—P6—O*rs*	103.4 (2)–113.0 (3)
		O*pq*—P7—O*rs*	102.1 (2)–113.8 (3)
		O*pq*—P8—O*rs*	104.1 (2)–114.6 (3)
		p, *r* = 1–4; *q*, *s* = 1–8	

Because of pseudosymmetry related to space group $P4_12_12$, the structure solution by direct methods failed. After consideration of the close structural relationship between KMgWP and KNiWP, fractional coordinates of the four symmetry dependent W atoms in KNiWP were used as an input model at the beginning of the refinement using *SHELXL*93 (Sheldrick, 1993). The origin of the unit cell was automatically fixed by the program containing a corresponding routine by Flack & Schwarzenbach (1988). The positions of all K, Mg, P and O atoms were found using difference Fourier maps.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP* (Frenz, 1978). Molecular graphics: *ATOMS* (Dowty, 1989).

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: JZ1148). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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