Data collection

Stoe Siemens AED 2 diffractometer
ω–θ scans
Absorption correction: by integration from crystal shape
$T_{\min} = 0.29$, $T_{\max} = 0.66$
654 measured reflections
250 independent reflections
198 observed reflections [$I > 5\sigma(I)$]

$R_{\text{int}} = 0.034$
$\theta_{\max} = 29.9°$
$h = -1 \rightarrow 13$
$k = -1 \rightarrow 13$
$l = -1 \rightarrow 9$
3 standard reflections
frequency: 360 min
intensity variation: 2.0%

Refinement

Refinement on F
$R = 0.024$
$wR = 0.030$
$S = 1.27$
198 reflections
29 parameters
$w = 1/[\sigma^2(F) + 0.0007F^2]$
$(\Delta/\sigma)_{\max} = 0.004$

$\Delta\rho_{\max} = 0.6$ e Å^{-3}
$\Delta\rho_{\min} = -1.0$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (II)

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

	x	y	z	U_{eq}
Sr	0	1/2	0	0.0103 (4)
Ca	0	1/2	0	0.0103 (4)
Cu	0.3859 (1)	x	0.2267 (2)	0.0134 (3)
O(1)	0.1720 (4)	0.3549 (4)	0.2419 (12)	0.0138 (10)
O(2)	0.0986 (5)	x	0.2785 (19)	0.031 (3)
B	0.2053 (6)	x	0.256 (2)	0.0127 (19)

Table 4. *Selected geometric parameters* (Å, °) *for* (II)

Sr—O(1)	× 4	2.565 (6)	Cu—O(2)		2.938 (12)
Sr—O(1)	× 4	2.631 (6)	B—O(1)	× 2	1.378 (7)
Cu—O(1)	× 2	1.940 (4)	B—O(2)		1.361 (7)
Cu—O(2)	× 2	1.913 (5)			
O(1)—Cu—O(1)		73.45 (15)	O(2)—Cu—O(2)	× 2	86.1 (4)
O(1)—Cu—O(2)	× 2	102.43 (17)	O(1)—B—O(2)		122.7 (5)
O(2)—Cu—O(2)		81.64 (19)	O(1)—B—O(1$^{\text{i}}$)	× 2	114.6 (4)

Symmetry code: (i) y, x, z.

The structural refinement was performed with the *SHELX*76 program package (Sheldrick, 1976). The program *ATOMS* (Dowty, 1989) was used to obtain the structure diagram. Cell parameters were obtained from scanned Guinier X-ray powder photographs, using Cu $K\alpha_1$ radiation with silicon as an internal standard. Data were collected at room temperature and corrected for background, Lorentz and polarization effects.

The synthetic and structural investigations on metal borates are financially supported by the Swedish Natural Science Research Council.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1074). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Octasodium Hydrogen Decatungstogadolinate Triacontahydrate

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Abstract

The decatungstogadolinate anion in the title compound, $Na_8H[GdW_{10}O_{36}].30H_2O$, has approximately D_{4d} symmetry, with Gd—O and W—O distances of 2.39 (2)–2.42 (2) Å and 1.69 (2)–2.34 (2) Å, respectively.

Comment

In our survey of the structures of decatungstolanthanoate anions (Ozeki, Takahashi & Yamase, 1992; Ozeki & Yamase, 1993, 1994*a*,*b*; Sugeta & Yamase, 1993; Yamase, Ozeki & Ueda, 1993; Yamase & Ozeki, 1993), the lanthanide atoms of the $[LnW_{10}O_{36}]^{9-}$ (Ln = Pr, Nd, Sm, Gd, Tb, Dy) anions in their tripotassium tetrasodium salts were found to deform to C_{4v} symmetry from their highest possible symmetry of D_{4d}. This structural distortion originates from the attachment of the K$^+$ cations to the LnO_8 square antiprism in the $[LnW_{10}O_{36}]^{9-}$ anion (Ozeki & Yamase, 1994*a*). We report here the structure of the title compound, (1), and compare it with that of $K_3Na_4H_2[GdW_{10}O_{36}].21H_2O$, (2) (Yamase & Ozeki, 1993) to illustrate the effect of crystal packing on the structures of the decatungstolanthanoate anions.

As shown in Fig. 1, the decatungstogadolinate anion in compound (1) possesses the same metal–oxygen framework structure as found in compound (2). Unlike the $[GdW_{10}O_{36}]^{9-}$ anion in (2), where the Gd—W distances are asymmetric for the two $[W_5O_{18}]^{6-}$

moieties (Yamase & Ozeki, 1993), the Gd···W separations for the two $[W_5O_{18}]^{6-}$ groups in (1) display similar distances: 3.800–3.825 Å [average 3.81 (1) Å] for W1–W4 and 3.795–3.835 Å [average 3.82 (2) Å] for W6–W9. This is because no cationic groups attach to the O atoms in the GdO_8 square antiprism in (1). The Gd—O distances in the title compound are in the range 2.39–2.42 Å [average 2.41 (2) Å], which is slightly shorter than the corresponding distances of 2.37–2.49 Å [average 2.43 (2) Å] found in (2), although the difference is not significant when compared with the standard deviations. The Gd—W distances in (1), which range from 3.795 to 3.835 Å [average 3.81 (1) Å], are also shorter than those in (2), 3.812–3.875 Å [average 3.84 (1) Å]. A similar trend was observed when comparing the structures of the decatungstosamarate anions in $Na_6H_3[SmW_{10}O_{36}].28H_2O$ (Ozeki & Yamase, 1994*b*) and $K_3Na_4H_2[SmW_{10}O_{36}].22H_2O$ (Ozeki & Yamase, 1993). These differences originate in the crystal packing of the compounds. The O atoms coordinating to the lanthanide atoms in the tripotassium tetrasodium salts also coordinate to the K^+ cations in their vicinity. Thus, the electron density at these O atoms is reduced, which results in the elongation of the Ln—O distances in these salts.

Fig. 2 shows the packing diagram of compound (1). Of the ten independent Na^+ cations, Na8–Na10 showed large displacement parameters. Therefore, the site-occupation factors for these atoms were refined and converged to values between 0.5 and 0.7. In the final refinement, these values were fixed at 0.5. The disordered Na^+ cations have four or five O atoms in their coordination sphere with Na—O distances of 2.11–2.66 Å, while all the other Na^+ cations achieve octahedral coordination with Na—O distances of 2.33–2.57 Å.

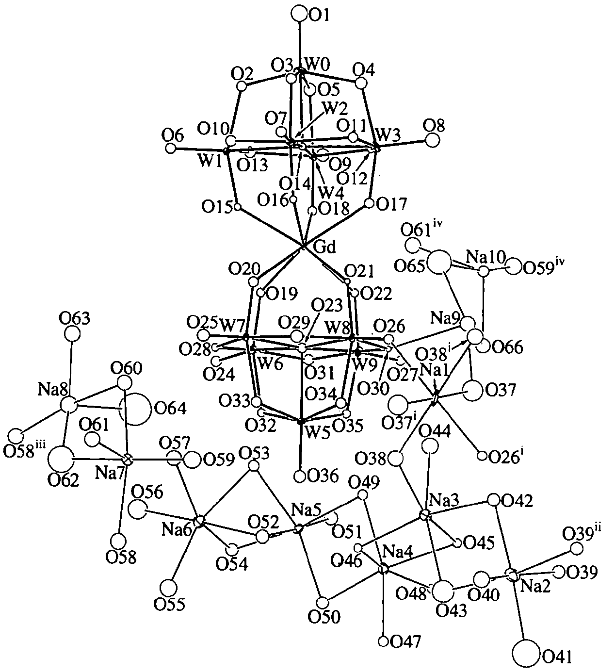

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the decatungstogadolinate anion in compound (1). Displacement ellipsoids are shown at 50% probability levels. Symmetry codes: (i) 1 − *x*, −*y*, 1 − *z*; (ii) 1 − *x*, −1 − *y*, 1 − *z*; (iii) 2 − *x*, −*y*, −*z*; (iv) x, y, 1 + *z*.

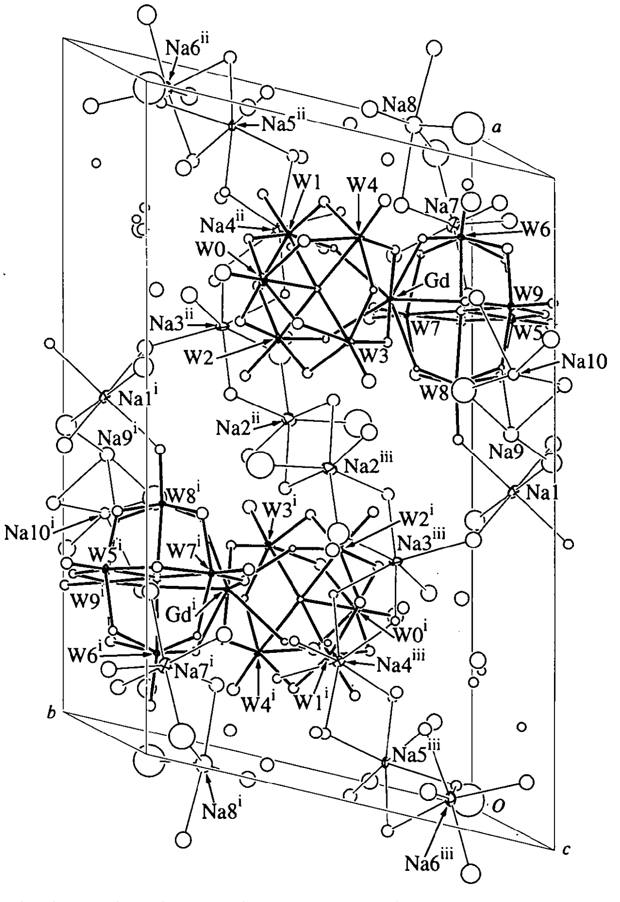

Fig. 2. Packing diagram of compound (1) viewed down the *c** axis. Symmetry codes: (i) 1 − *x*, 1 − *y*, 1 − *z*; (ii) *x*, 1 + *y*, *z*; (iii) 1 − *x*, −*y*, 1 − *z*.

Experimental

8.3 g of $Na_2WO_4.2H_2O$ dissolved in 20 ml of aqueous solution was acidified with CH_3COOH to pH 7.3 and heated to 353 K. 1.15 g of $GdNO_3.4H_2O$ in 2 ml of H_2O was added dropwise and the mixture kept at elevated temperature until the white precipitate dissolved. 0.1 g of NaCl powder was added to the solution. Colourless hexagonal prismatic crystals were obtained after keeping the solution at 277 K for 12 h. The crystals were very efflorescent and all the diffraction measurements were performed at 150 K to minimize the decay of the diffraction intensities.

Crystal data

$Na_8H[GdW_{10}O_{36}].30H_2O$
$M_r = 3354.2$
Triclinic
$P\bar{1}$
$a = 20.908$ (8) Å
$b = 13.088$ (4) Å
$c = 12.884$ (4) Å
$\alpha = 102.65$ (2)°
$\beta = 95.94$ (3)°
$\gamma = 77.05$ (3)°
$V = 3347$ (2) Å^3
$Z = 2$
$D_x = 3.33$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10.0$–12.5°
$\mu = 18.27$ mm^{-1}
$T = 150$ K
Hexagonal prism
0.20 × 0.20 × 0.20 mm
Colourless

Data collection

Rigaku AFC-5*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *DIFABS* (Walker & Stuart, 1983).
$T_{min} = 0.747$, $T_{max} = 1.304$
16 024 measured reflections
15 341 independent reflections

10 351 observed reflections $[I > 3\sigma(I)]$
$\theta_{max} = 27.5°$
$h = -27 \rightarrow 26$
$k = -17 \rightarrow 16$
$l = 0 \rightarrow 16$
3 standard reflections monitored every 100 reflections intensity variation: −2.8%

Refinement

Refinement on F
$R = 0.065$
$wR = 0.086$
$S = 1.72$
10 351 reflections
436 parameters
H atoms not located
$w = 1/[\sigma^2(F) + 0.0009F^2]$

$(\Delta/\sigma)_{max} = 0.05$
$\Delta\rho_{max} = 7.32$ e Å^{-3}
$\Delta\rho_{min} = -4.21$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

U_{iso} for Na8, 9, 10, O*n*; $U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	U_{iso}/U_{eq}
W0	0.74713 (5)	0.71133 (7)	0.98507 (8)	0.0100
W1	0.81561 (5)	0.60190 (7)	0.75571 (7)	0.0066
W2	0.65642 (5)	0.62842 (7)	0.76861 (7)	0.0065
W3	0.68280 (5)	0.49598 (7)	0.96201 (7)	0.0074
W4	0.84227 (5)	0.46784 (7)	0.94893 (7)	0.0060
W5	0.75413 (5)	−0.02013 (7)	0.39513 (8)	0.0064
W6	0.86275 (5)	0.12944 (7)	0.50721 (8)	0.0057
W7	0.72360 (5)	0.24120 (7)	0.39807 (7)	0.0045
W8	0.64305 (5)	0.14976 (7)	0.55037 (8)	0.0058
W9	0.78216 (5)	0.03723 (7)	0.65804 (8)	0.0066
Gd	0.75219 (6)	0.34159 (8)	0.69793 (9)	0.0047
Na1	1/2	0	1/2	0.0187
Na2	0.5255 (6)	−0.4752 (8)	0.3814 (9)	0.0201
Na3	0.6417 (5)	−0.3086 (8)	0.3991 (8)	0.0125
Na4	0.8072 (5)	−0.4548 (8)	0.3611 (8)	0.0120
Na5	0.9423 (5)	−0.3407 (7)	0.3639 (8)	0.0089
Na6	0.9672 (6)	−0.2155 (8)	0.1914 (8)	0.0157
Na7	0.8627 (6)	0.0572 (8)	0.0590 (8)	0.0167
Na8†	0.999 (1)	0.171 (2)	0.146 (2)	0.029 (6)
Na9†	0.595 (1)	0.061 (2)	0.774 (2)	0.021 (5)
Na10†	0.692 (1)	0.097 (2)	0.978 (2)	0.012 (4)
O1	0.747 (1)	0.828 (2)	1.078 (2)	0.026 (5)
O2	0.7993 (8)	0.725 (1)	0.881 (1)	0.009 (3)
O3	0.6730 (9)	0.748 (1)	0.891 (1)	0.011 (4)
O4	0.6955 (9)	0.638 (1)	1.048 (1)	0.013 (4)
O5	0.8212 (9)	0.620 (1)	1.036 (1)	0.015 (4)
O6	0.8639 (9)	0.650 (1)	0.684 (1)	0.010 (3)
O7	0.5868 (9)	0.698 (1)	0.708 (1)	0.011 (4)
O8	0.6344 (10)	0.466 (1)	1.049 (2)	0.019 (4)
O9	0.9092 (9)	0.422 (1)	1.027 (1)	0.015 (4)
O10	0.7262 (9)	0.666 (1)	0.706 (1)	0.011 (4)
O11	0.6186 (9)	0.579 (1)	0.875 (1)	0.010 (3)
O12	0.7716 (8)	0.448 (1)	1.022 (1)	0.005 (3)
O13	0.8800 (9)	0.535 (1)	0.855 (1)	0.011 (4)
O14	0.7482 (8)	0.553 (1)	0.863 (1)	0.005 (3)
O15	0.8080 (8)	0.475 (1)	0.674 (1)	0.005 (3)
O16	0.6688 (8)	0.497 (1)	0.686 (1)	0.005 (3)
O17	0.6917 (8)	0.381 (1)	0.857 (1)	0.009 (3)
O18	0.8331 (8)	0.359 (1)	0.845 (1)	0.008 (3)
O19	0.8510 (8)	0.245 (1)	0.612 (1)	0.007 (3)
O20	0.7273 (9)	0.344 (1)	0.510 (1)	0.010 (3)
O21	0.6549 (8)	0.267 (1)	0.646 (1)	0.003 (3)
O22	0.7765 (8)	0.167 (1)	0.744 (1)	0.007 (3)
O23	0.7530 (9)	0.132 (1)	0.521 (1)	0.009 (3)
O24	0.9425 (9)	0.107 (1)	0.478 (1)	0.011 (4)
O25	0.7011 (9)	0.310 (1)	0.293 (1)	0.014 (4)
O26	0.5613 (9)	0.146 (1)	0.555 (1)	0.010 (3)
O27	0.8045 (8)	−0.047 (1)	0.748 (1)	0.009 (3)
O28	0.8198 (8)	0.205 (1)	0.398 (1)	0.006 (3)
O29	0.6388 (9)	0.225 (1)	0.434 (1)	0.015 (4)
O30	0.6860 (8)	0.058 (1)	0.643 (1)	0.003 (3)
O31	0.8655 (9)	0.037 (1)	0.609 (1)	0.011 (4)
O32	0.8417 (8)	0.000 (1)	0.401 (1)	0.007 (3)
O33	0.7304 (9)	0.090 (1)	0.315 (1)	0.011 (4)
O34	0.6652 (9)	0.017 (1)	0.434 (1)	0.011 (3)
O35	0.7766 (8)	−0.073 (1)	0.520 (1)	0.007 (3)
O36	0.7555 (9)	−0.134 (1)	0.297 (1)	0.013 (4)
O37	0.562 (1)	−0.066 (2)	0.642 (2)	0.040 (6)
O38	0.582 (1)	−0.122 (2)	0.382 (2)	0.024 (5)
O39	0.5755 (9)	−0.554 (1)	0.525 (1)	0.015 (4)
O40	0.452 (1)	−0.405 (2)	0.247 (2)	0.025 (5)
O41	0.541 (2)	−0.662 (3)	0.290 (3)	0.075 (10)
O42	0.5474 (9)	−0.310 (1)	0.487 (1)	0.016 (4)
O43	0.617 (1)	−0.472 (2)	0.286 (2)	0.047 (7)
O44	0.683 (1)	−0.195 (2)	0.561 (2)	0.026 (5)
O45	0.7156 (8)	−0.445 (1)	0.481 (1)	0.007 (3)
O46	0.7250 (8)	−0.335 (1)	0.274 (1)	0.007 (3)
O47	0.7876 (9)	−0.593 (1)	0.221 (1)	0.010 (3)
O48	0.8579 (8)	−0.584 (1)	0.472 (1)	0.008 (3)
O49	0.8462 (9)	−0.300 (1)	0.475 (1)	0.009 (3)
O50	0.9164 (9)	−0.508 (1)	0.269 (1)	0.014 (4)
O51	1.0089 (10)	−0.397 (1)	0.506 (1)	0.017 (4)
O52	0.8726 (10)	−0.274 (1)	0.218 (1)	0.017 (4)
O53	0.9522 (9)	−0.162 (1)	0.385 (1)	0.011 (4)
O54	1.0393 (9)	−0.353 (1)	0.273 (1)	0.015 (4)
O55	0.958 (1)	−0.307 (2)	0.010 (2)	0.028 (5)
O56	1.070 (1)	−0.170 (2)	0.171 (2)	0.037 (6)
O57	0.916 (1)	−0.027 (2)	0.204 (2)	0.022 (4)
O58	0.883 (1)	−0.108 (2)	−0.080 (2)	0.024 (5)
O59	0.762 (1)	0.031 (2)	0.108 (2)	0.029 (5)
O60	0.879 (1)	0.213 (1)	0.198 (2)	0.021 (4)
O61	0.794 (1)	0.183 (2)	−0.038 (2)	0.023 (4)
O62	0.961 (2)	0.088 (3)	0.003 (3)	0.069 (9)
O63	1.012 (1)	0.307 (2)	0.286 (2)	0.025 (5)
O64	1.019 (2)	0.068 (3)	0.294 (3)	0.10 (1)
O65	0.648 (2)	0.195 (2)	0.865 (2)	0.059 (8)
O66	0.6900 (10)	−0.056 (1)	0.848 (2)	0.020 (4)

† Occupation factor = 0.5.

Table 2. *Selected geometric parameters* (Å)

Gd···W1	3.825 (2)	W3—O4	2.01 (2)
Gd···W2	3.800 (2)	W3—O14	2.30 (2)
Gd···W3	3.803 (2)	W4—O9	1.71 (2)
Gd···W4	3.812 (2)	W4—O18	1.76 (2)
Gd···W7	3.829 (2)	W4—O12	1.92 (2)
Gd···W6	3.795 (2)	W4—O13	1.95 (2)
Gd···W8	3.835 (2)	W4—O5	2.04 (2)

Gd···W9	3.818 (2)	W4—O14	2.34 (2)
Gd—O15	2.39 (2)	W5—O36	1.73 (2)
Gd—O16	2.39 (2)	W5—O35	1.87 (2)
Gd—O17	2.42 (2)	W5—O32	1.90 (2)
Gd—O18	2.41 (2)	W5—O33	1.90 (2)
Gd—O19	2.40 (2)	W5—O34	1.90 (2)
Gd—O20	2.42 (2)	W5—O23	2.27 (2)
Gd—O21	2.42 (2)	W6—O24	1.69 (2)
Gd—O22	2.42 (2)	W6—O19	1.78 (2)
W0—O1	1.72 (2)	W6—O28	1.93 (2)
W0—O2	1.87 (2)	W6—O31	1.96 (2)
W0—O5	1.89 (2)	W6—O32	2.04 (2)
W0—O3	1.90 (2)	W6—O23	2.31 (2)
W0—O4	1.93 (2)	W7—O25	1.75 (2)
W0—O14	2.31 (1)	W7—O20	1.76 (2)
W1—O6	1.73 (2)	W7—O29	1.94 (2)
W1—O15	1.79 (1)	W7—O28	1.96 (2)
W1—O13	1.96 (2)	W7—O33	2.02 (2)
W1—O10	1.97 (2)	W7—O23	2.31 (2)
W1—O2	2.01 (2)	W8—O26	1.73 (2)
W1—O14	2.34 (2)	W8—O21	1.79 (1)
W2—O7	1.74 (2)	W8—O30	1.89 (2)
W2—O16	1.79 (1)	W8—O29	1.96 (2)
W2—O11	1.94 (2)	W8—O34	2.03 (2)
W2—O10	1.94 (2)	W8—O23	2.32 (2)
W2—O3	2.02 (2)	W9—O27	1.73 (2)
W2—O14	2.31 (2)	W9—O22	1.80 (2)
W3—O8	1.75 (2)	W9—O31	1.91 (2)
W3—O17	1.77 (2)	W9—O30	1.96 (2)
W3—O11	1.95 (2)	W9—O35	2.04 (2)
W3—O12	1.95 (2)	W9—O23	2.33 (2)

Data collection: *RCRYSTAN* (Rigaku Corporation, 1985). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: AS1132). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 1852–1854

An Mo^V Monophosphate, $Rb_2Mo_2P_2O_{11}$

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Abstract

Molybdenum rubidium phosphate, $Rb_2Mo_2P_2O_{11}$, has been synthesized and the growth of single crystals allowed its structure to be determined. This compound is isotypic with $Tl_2Mo_2P_2O_{11}$ and $K_2Mo_2P_2O_{11}$, and contains a framework built from $Mo_2P_2O_{15}$ units formed by one Mo_2O_{11} group sharing four of its corners with two PO_4 tetrahedra, leading to $[Mo_2P_2O_{13}]_\infty$ chains running along **c**, or to $[Mo_2P_2O_{13}]_\infty$ double layers. The $[Mo_2P_2O_{11}]_\infty$ framework forms two different intersecting tunnels along [001] and $\langle 110 \rangle$ where the Rb^+ ions are located. Although different from $Rb_{1.5}Mo_2P_2O_{11}$, the two structures exhibit some close relationships.

Comment

The synthesis and determination of the structure of the title compound were undertaken in order to test the possibility of replacing potassium by rubidium in the phase $K_2Mo_2P_2O_{11}$ (Gueho, Borel, Grandin, Leclaire & Raveau, 1993). The replacement of K^+ by Cs^+ was only partial and led to $Cs_{1.5}Mo_2P_2O_{11}$ with the same framework structure (Borel, Leclaire, Grandin & Raveau, 1994), whereas the replacement of K^+ by Tl^+ was complete (Guesdon, Borel, Grandin, Leclaire & Raveau, 1993). Attempts to substitute rubidium for potassium allowed $Rb_2Mo_2P_2O_{11}$ to be prepared in the form of a microcrystalline powder (Gueho *et al.*, 1993), but the first single crystals that were obtained were those of the monophosphate $Rb_{1.5}Mo_2P_2O_{11}$ (Borel, Leclaire, Guesdon, Grandin & Raveau, 1994) with a structure which, in contrast to $Cs_{1.5}Mo_2P_2O_{11}$, was different to that of $K_2Mo_2P_2O_{11}$. In the present work, single crystals of $Rb_2Mo_2P_2O_{11}$ were synthesized as a minor phase from a batch of nominal composition $Rb_6Mo_7P_2O_{37}$ heated at 923 K for one day in an evacuated silica ampoule and cooled at 2 K h^{-1} to 823 K. The sample was then quenched to room temperature and reddish purple crystals were extracted.

$Rb_2Mo_2P_2O_{11}$ is isotypic with the 'K_2', '$Cs_{1.5}$' and 'Tl_2' phases; its framework is built from $Mo_2P_2O_{15}$ units formed by one Mo_2O_{11} group sharing four of

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