Crystal Structures of New Pyrovanadates $A_2MnV_2O_7$ ($A = \text{Rb, K}$)

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Dedicated to Dr. Bernard Chevalier on the occasion of his 60th birthday

The new compounds $A_2MnV_2O_7$ ($A = \text{K, Rb}$) with structures related to the melilite-type have been synthesized by a solid state reaction route. The crystal structures of $K_2MnV_2O_7$, $Rb_2MnV_2O_7$ and $KRbMnV_2O_7$ have been determined using X-ray single crystal diffraction data. The compound $K_2MnV_2O_7$ crystallizes with a melilite-type structure with tetragonal unit cell parameters $a = 8.609(3)$ Å, $c = 5.538(4)$ Å and space group $P\bar{4}21m$. The structures of $Rb_2MnV_2O_7$ and $KRbMnV_2O_7$ are derived from the melilite-type structure with space group $P4_2/mnm$ and unit cell parameters $a = 8.577(6)$ Å, $c = 11.809(6)$ Å, and $a = 8.530(6)$ Å, $c = 11.466(5)$ Å, respectively. The three structures consist of $[MnV_2O_7]^2-$ layers perpendicular to the $c$ axis separated by $A^+$ layers. The $[MnV_2O_7]^2-$ layers feature corner-sharing MnO$_4$ tetrahedra and V$_2$O$_7$ pyrovanadate units, the linkage leading to rings of five tetrahedra. The doubling of the $c$ parameter for $Rb_2MnV_2O_7$ or $RbK MnV_2O_7$ is explained by the existence of a mirror plane perpendicular to the $[001]$ direction between two $[MnV_2O_7]^2-$ layers. The $A^+$ alkali cations occupy distorted square antiprisms of oxygen atoms in $K_2MnV_2O_7$ and distorted square prisms of oxygen atoms in $Rb_2MnV_2O_7$ and $RbK MnV_2O_7$.

Key words: Vanadate, Melilite, Crystal Chemistry, Single Crystal X-Ray Diffraction, Oxides

Introduction

Most of the minerals of the melilite group are silicates with the general formula $[\text{Si}_2\text{O}_7]^n$ ($\text{N} = \text{coordination number}$). Among them, one can find the akermanite $\text{Ca}_2\text{MgSi}_2\text{O}_7$ [1], the gehlenite $\text{Ca}_2\text{Al(AlSi)}\text{O}_7$ [2], the okayamalite $\text{Ca}_2\text{B(BSi)}\text{O}_7$ [3] the hardystonite $\text{Ca}_2\text{BeSi}_2\text{O}_7$ [4], the gugiaite $\text{Ca}_2\text{BeSi}_2\text{O}_7$ [5], and the melilite (Ca,Na)$_2$(Mg,Fe,Al)-(AlSi)O$_7$ [6]. Many melilite-type compounds have been synthesized with a wide range of chemical compositions, the general formula becoming $A_2BC_2X_7$ where $A$ is a large cation such as Ca, Sr, Ba, Na, K, Y, lanthanides (La–Er), Pb, Bi; $B$ is a small four coordinated cation such as Be, Mg, Mn, Fe, Co, Cu, Zn, Cd, Al, Ga, Si; $C = \text{Cr, Al, Ga, Si, Ge, B, V, and X = O, F, S, N}$. The melilite-type structure was first determined by Warren (1930) [1]. The structure has a tetragonal symmetry, space group $P4\bar{2}1m$, and consists of $BC_2O_7$ layers parallel to (001) made of corner-sharing $BO_4$ and $CO_4$ tetrahedra. The cations $A$ are lying between these layers in distorted square antiprisms of oxygen atoms. Recently, Tyutyunnik et al. have synthesized $\text{Na}_2\text{ZnV}_2\text{O}_7$, the first vanadate crystallizing in the melilite-type structure [7].

In the past only few vanadate compounds having structures closely related to the melilite-type have been prepared. The compounds $(\text{NH}_4)_2\text{VOV}_2\text{O}_7$ [8], $\text{K}_2\text{VOV}_2\text{O}_7$ [9], $\text{Rb}_2\text{VOV}_2\text{O}_7$ [10], and $\text{KBaCuClV}_2\text{O}_7$ [11] crystallize in the fresnoite-type structure which is very similar to the melilite-type, except that the V$_2$O$_7$ pyrogroeps all point in the same direction and the V$^{4+}$ and Cu$^{2+}$ coordination polyhedra are square pyramids instead of the usual tetrahedra. They all have tetragonal cell parameters with space group $P4bm$ and with $a$ ranging from 8.8581(13) to 8.9229(10) Å and $c$ ranging from 5.215(5) to 5.5640(5) Å. A different variation of the melilite-type structure is observed for $\text{K}_2\text{MgV}_2\text{O}_7$ [12], which crystallizes in space group $P4_2/mnm$ with cell parameters $a = 8.38(2)$ and $c = 11.36(2)$ Å. The main difference between the $\text{K}_2\text{MgV}_2\text{O}_7$ and the melilite-type structures is the doubling of the lattice constant $c$ explained by the existence of a mirror plane perpendicular to the [001] direction between two MgV$_2$O$_7$ layers. This induces a change from $P4\bar{2}1m$ to $P4_2/mnm$ symmetry. This structural variation is only
observed for phosphates and vanadates [12–15]. Notably, $P4_2/m$ and $P4_2/m$m$ are both subgroups of space group $P4/mmm$. No compound of the melilite group crystallizes in this space group.

Crystals of the new phase $K_2MnV_2O_7$ were obtained during fast melting of a vanadate powder with the composition $K MnVO_4$. In a recent paper the crystal structure of $K MnVO_4$ has been solved and shown to be an oxygen-deficient perovskite [16].

The aim of this work is to discuss the structural effects of the partial or complete substitution of $K^+$ for $Rb^+$ on the $A$ positions of the compounds of general formula $A_2MnV_2O_7$, in order to contribute to a better insight into the crystal chemistry of the divanadates $A_2BV_2O_7$.

**Experimental Section**

$K_2MnV_2O_7$ was prepared by solid state reaction from a stoichiometric mixture of $KVO_3$ and $MnO$. $KVO_3$ was obtained by heating a $1:1$ mixture of $K_2CO_3$ and $V_2O_5$ at $550^\circ C$ for $6$ h. The mixture was put in a gold tube which was sealed under vacuum in a silica tube and then heated at $450^\circ C$ for $24$ h and at $500^\circ C$ for $12$ h. After grinding a further heating of the mixture at $500^\circ C$ for $18$ h led to a mixture of three phases: $K_2MnV_2O_7$, $KO_3$, and $MnO$. Different treatments (time and temperature) did not improve the results. As $KVO_3$ is soluble in water, the powder sample was washed to obtain $K_2MnV_2O_7$ as major phase (Fig. 1). Attempts to prepare single crystals of $K_2MnV_2O_7$ by melting the sample powder were unsuccessful. Subsequently, a $1:1$ mixture of $KVO_3$ and $MnO$ was prepared. By fast heating of this starting mixture at $950^\circ C$ followed by slowly decreasing the temperature at the rate of $5^\circ C$ h$^{-1}$ to r.t., a mixture of yellow, orange and green crystals corresponding to $KMnVO_4$, $K_2MnV_2O_7$, and $MnO$, respectively, was obtained.

$Rb_2MnV_2O_7$ powder and crystals were prepared exactly in the same way as $K_2MnV_2O_7$. We used $RbVO_3$ instead of $KVO_3$. $RbVO_3$ was obtained by heating a $1:1$ mixture of $Rb_2CO_3$ and $V_2O_5$ at $700^\circ C$ for $10$ h.

$K RbMnV_2O_7$ was prepared by solid state reaction from a stoichiometric mixture of $KVO_3$, $RbVO_3$ and $MnO$. The mixture was put in a gold tube which was sealed under vacuum in a silica tube and then heated at $500^\circ C$ for $48$ h with intermittent grinding. This led to a mixture of $K RbMnV_2O_7$, $K_1...Rb_2VO_3$, and $MnO$. The heating of this mixture at $950^\circ C$ for $2$ h and the slow cooling at the rate of $5^\circ C$ h$^{-1}$ to $700^\circ C$ and at $10^\circ C$ h$^{-1}$ to r.t. enabled us to obtain several crystals of $K RbMnV_2O_7$.

**X-Ray diffraction measurements**

Crystals of the title compounds suitable for single crystal X-ray diffraction were selected on the basis of the size and the sharpness of the diffraction spots. In the case of $Rb_2MnV_2O_7$, the quality of the single crystals was really poor, and this explains the high internal $R$ value (see Table 1). A similar problem was encountered for $K RbMnV_2O_7$. The data collections were carried out on an Enraf-Nonius Kappa CCD diffractometer using Mo$K_\alpha$ radiation. Data processing and all refinements were performed with the JANA2000 program package [17]. A Gaussian-type absorption correction was applied, and the shapes were determined with the video microscope of the Kappa CCD. Details of data collection are summarized in Table 1.

**Structure refinement**

The extinction conditions observed for $K_2MnV_2O_7$ agree with the space group $P4_2/m$. Most of the atomic positions were found by Direct Methods using $S i r 97$ [18]. With anisotropic displacement parameters, the final residual factors converged to $R(F) = 0.025$ and $wR(F^2) = 0.055$ for $34$ refined parameters, $832$ observed reflections and difference-Fourier residues in the range between $-0.33$ and $+0.32$ e $\AA^{-3}$. The Flack parameter refined to $0.01(4)$.

The lattice of $Rb_2MnV_2O_7$ has tetragonal geometry. The observed systematic extinctions agree with space group $P4_2/m$m$$. The structure was solved by Direct Methods using $S h e l x s-97$ [19], which revealed the heavy atom positions. Several difference-Fourier syntheses allowed us to localize the oxygen atom positions. This refinement led to the residual factors $R(F) = 0.042$ and $wR(F^2) = 0.088$ for $36$ refined parameters, $455$ observed reflections and difference-Fourier residues in the range between $-1.06$ and $+1.43$ e $\AA^{-3}$.

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Table 1. Crystallographic data and structure refinement for K₂MnV₂O₇, Rb₂MnV₂O₇, and RbKMnV₂O₇.

Table 2. Atom positions and isotopic displacement parameters for K₂MnV₂O₇, Rb₂MnV₂O₇, and KRbMnV₂O₇.
refinement of the homologous compound Rb2MnV2O7. The atomic positions of the latter were used as a starting model for the refinement. The rubidium atom in the 4f position was replaced by a potassium atom to get the correct formula K2MnV2O7. At this stage the refinement led to residual factors of R(F) = 0.194 and ωR(F^2) = 0.430 with large negative displacement parameters for Rb (0.067 Å^2) and K, respectively. Attempts to solve the structure with fully ordered sites failed; therefore the structure was refined with introduction of K/Rb disorder on the 4g and 4f positions. With anisotropic displacement parameters, the final residual factors converged to R(F) = 0.044 and ωR(F^2) = 0.100 for 37 refined parameters, 540 observed reflections and difference-Fourier residues in the range between −0.76 and +0.85 e Å^−3. The refined atomic positions and anisotropic displacement parameters (ADPs) of the different phases are given in Tables 2 and 3, respectively.

Further details may be obtained from: Fachinformationsszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen (Germany), by quoting the Registry No’s. CSD–417890 (Rb2MnV2O7), CSD–417891 (KRbMnV2O7), and CSD–417892 (K2MnV2O7).

**Results and Discussion**

The crystal structure of K2MnV2O7 is isotypic to the melilite-type structure consisting of alternating MnV2O7 and K layers (Fig. 2a). The MnV2O7 lay-
ers contain corner-sharing MnO$_4$ tetrahedra and V$_2$O$_7$ pyrovanadate units (two corner-sharing VO$_4$ tetrahedra) that form rings of five tetrahedra (Fig. 2b). The eight-coordinated cations K are positioned between these layers. The interatomic distances for the MnO$_4$, VO$_4$ and KO$_8$ polyhedra are listed in Table 4. The MnO$_4$ tetrahedra contain four regular Mn–O bonds of 2.028(2) Å very close to the value of 2.04 Å estimated from the effective ionic radii of Mn$^{2+}$ and O$^2$ [20]. A comparable Mn–O distance has been reported for the analogous silicate Sr$_2$MnSi$_2$O$_7$ [21]. The large O–Mn–O angles of 121.05(7)$^\circ$ indicate that the MnO$_4$ tetrahedron is flattened along the [001] direction. Two neighboring VO$_4$ units share an O2 atom to form the pyrovanadate unit [V$_2$O$_7$]$^4-$.

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Table 4. Interatomic distances (Å), angles (deg) and bond valence sums (BVS)$^a$ for K$_2$MnV$_2$O$_7$, Rb$_2$MnV$_2$O$_7$, and KRbMnV$_2$O$_7$.

$^a$ BV = $e^{(r_0-r)/b}$ with the following parameters [23]: $b = 0.37$ and $r_0$ (Mn$^{II}$–O) = 1.790, $r_0$ (V$^{IV}$–O) = 1.803, $r_0$ (K–O) = 2.132 and $r_0$ (RB–O) = 2.263 Å.
2.909 Å consistent with the Shannon Table ($d_{K-O} = 2.93$ Å). The calculations of the bond valence sums (BVS) for Mn$^{2+}$, V$^{5+}$, K$^{+}$ confirm the charge balance (i.e., BVS = 2.10, 5.14 and 1.05 for Mn$^{2+}$, V$^{5+}$ and K$^{+}$, respectively) [22, 23].

Projection views onto the (010) and (001) planes of the Rb$_2$MnV$_2$O$_7$ structure are displayed in Fig. 4. The unit cell parameter $c$ of the Rb$_2$MnV$_2$O$_7$ structure is larger than that of melilite K$_2$MnV$_2$O$_7$ by a factor of approximately two. The addition of a symmetry element to the space group $P\bar{4}2_1m$ leads to space group $P4_2/mnm$. As a result, the unit cell of the rubidium manganese vanadate contains two layers of tetrahedral [MnV$_2$O$_7$]$^{2-}$, $T$ and $T'$ (Fig. 4, left), which are related by the mirror plane, instead of only one layer $T$ in K$_2$MnV$_2$O$_7$ (Fig. 2a). The MnV$_2$O$_7$ layers are similar to those observed for K$_2$MnV$_2$O$_7$, consisting of corner-sharing MnO$_4$ and VO$_4$ tetrahedra, the latter forming V$_2$O$_7$ pyrogroups. The Rb$^+$ cation layers are situated between the [MnV$_2$O$_7$]$^{2-}$ layers. As expected, when rubidium is substituted for potassium a slight increase of the interlayer space is observed with $c/2 = 5.905$ Å for Rb$_2$MnV$_2$O$_7$ and $c = 5.538$ Å for K$_2$MnV$_2$O$_7$. The main difference between the two compounds is found in the interlayer alkali cation environment. Indeed, in K$_2$MnV$_2$O$_7$ the potassium atoms occupy only one site (4e) with a square antiprismic coordination (Fig. 3), whereas in Rb$_2$MnV$_2$O$_7$ the rubidium atoms occupy two different sites (4g and 4f for Rb1 and Rb2, respectively) with a distorted square prismatic coordination (Fig. 5). The Rb1 polyhedron is more distorted than that of Rb2. The distances Rb–O range from 2.791(5) to 3.201(5) Å and from 2.958(4) to 3.037(5) Å giving an average distance of 3.02 and 3.00 Å and a bond valence sum of 1.16 and 1.10 for Rb1 and Rb2, respectively. This is in agreement with the sum of ionic radii of 3.03 Å and the charge balance of +1. The short distance Rb1–O3 of 2.791(5) Å is attributed to the fact that the O3 position is shared by the Rb1O$_8$ and VO$_4$ polyhedra. Indeed, in order to obtain an acceptable Rb–O3 distance the V–O3 distance is compressed along the [001] direction to a value of 1.646(2) Å. This steric strain is reflected by the relatively large anisotropic displacement parameters ($U_{11} = U_{22} = 0.041$ Å$^2$) for the O3 position and ($U_{11} = U_{22} = 0.0409$ Å$^2$) for the Rb1 position (Table 3).

As in K$_2$MnV$_2$O$_7$, in Rb$_2$MnV$_2$O$_7$ the MnO$_4$ and V$_2$O$_7$ units form rings of five tetrahedra (Fig. 6) with no significant changes in the V–O and Mn–O distances and in the V–O2–V and V–O1–Mn bridging angles (Table 4). The bond valence sums (BVS) of 2.06 and 5.14 are in agreement with the values expected for Mn$^{2+}$ and V$^{5+}$, respectively [22, 23].
RbKMnV₂O₇ is isostructural to Rb₂MnV₂O₇. The small difference between the two compounds is the appearance of K/Rb disorder in the Rb1 and Rb2 positions which induces a more relaxed structure. This is mainly reflected by the lower values of the anisotropic displacement parameters of all atoms in Rb₂KMnV₂O₇ (Table 3). We can also see that the MnO₄ tetrahedra are less flattened in the [001] direction (dₐₐₐₐ ≈ 2.036 Å and O1–Mn–O1 = 115.63°) than in Rb₂MnV₂O₇ (dₐₐₐₐ ≈ 2.035 Å and O1–Mn–O1 = 117.4°) or in K₂MnV₂O₇ (dₐₐₐₐ ≈ 2.028 Å and O1–Mn–O1 ≈ 121.05°) (Table 4). Bindi et al. reported the observation that the greater the size of the tetrahedral cations with respect to the cations A, the greater the structural misfit leading to the incommensurate structure is found [24]. We can then assume that the structural transition from the melilite-type (space group P₄₂/m) to the closely related structure of Rb₂MnV₂O₇ and KRBmMnV₂O₇ (space group P₄₃/mmm) proceeds by an analogous mechanism. When the size of the tetrahedral cations for B and/or C decreases (with the same cation A) or the size of the cations A increases (no change in B and C sizes) a phase transition occurs. For example, in K₂MnV₂O₇ when Mg²⁺ (rion = 0.57 Å [20]) is substituted for Mn²⁺ (0.66 Å), Zn²⁺ (0.60 Å) and P⁵⁺ (0.17 Å) for Mn²⁺ (0.66 Å) and V⁵⁺ (0.355 Å) or Rb⁺ (1.61 Å) for K⁺ (1.51 Å), this structural transition is observed (see Table 1). The same behavior is observed for Na₂ZnV₂O₇ when phosphorus is substituted for vanadium, the size of the other cations being almost constant (Table 5).

These results indicate that there is no change in the dimension of the sheets of tetrahedra and accordingly no significant change in the lattice parameter a (Table 1).

The mixed potassium rubidium divanadate RbKMnV₂O₇ is isostructural to Rb₂MnV₂O₇. The


