

Kannanite, a new mineral from Kannan Mountain, Japan

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Kannanite, a new Ca-dominant member of the ardennite series, was obtained from Kannan Mountain, Ozu, Ehime Prefecture, Japan. Kannanite occurs in fine quartz veins crossing the hematite-rich regions of an iron-manganese ore included in the metachert. These veins are pale orange in color, while the kannanite itself is brownish-orange to orange, with a thin section exhibiting weak pleochroism. The mineral was found in grain sizes ranging from several to 15 μm and has a Mohs hardness of 6 and a calculated density of 3.43 g cm^{-3} . The mean refractive index obtained from the Gladstone–Dale relationship for this mineral is 1.788. The empirical formula for kannanite is $(\text{Ca}_{3.60}\text{Mn}_{0.40}^{2+})_{\Sigma 4}(\text{Al}_{3.00}\text{Mn}_{1.31}^{3+}\text{Fe}_{0.69}^{3+}\text{Mg}_{0.71}\text{Mn}_{0.19}^{2+}\text{Ni}_{0.06}\text{Cu}_{0.05})_{\Sigma 6}[(\text{V}_{0.70}^{5+}\text{Si}_{0.16}\text{As}_{0.14})_{\Sigma 1}\text{O}_{3.84}(\text{OH})_{0.16}](\text{SiO}_4)_2(\text{Si}_3\text{O}_{10})(\text{OH})_6$, while the simplified formula is $\text{Ca}_4[(\text{Al}, \text{Mn}^{3+}, \text{Fe}^{3+})_5\text{Mg}](\text{VO}_4)(\text{SiO}_4)_2(\text{Si}_3\text{O}_{10})(\text{OH})_6$. Kannanite has an orthorhombic structure with a space group $Pnmm$ and unit cell parameters $a = 8.8802(14) \text{ \AA}$, $b = 5.9919(13) \text{ \AA}$, $c = 18.882(3) \text{ \AA}$ and $V = 1004.7(3) \text{ \AA}^3$. Kannanite is considered to be formed by the activities of metamorphic fluid accompanied with the Sanbagawa metamorphism.

Keywords: Kannanite, Ardennite, New mineral, Ehime Prefecture, Japan

INTRODUCTION

To date, two ardennite-series minerals, ardennite-(As) $\text{Mn}_4^{2+}(\text{Al}_5\text{Mg})(\text{AsO}_4)(\text{SiO}_4)_2(\text{Si}_3\text{O}_{10})(\text{OH})_6$ and ardennite-(V) $\text{Mn}_4^{2+}(\text{Al}_5\text{Mg})(\text{VO}_4)(\text{SiO}_4)_2(\text{Si}_3\text{O}_{10})(\text{OH})_6$, have been identified (Barresi et al., 2007). They occur as rock-forming minerals in oxidized Mn–Al-rich metasediments over a wide range of pressure–temperature conditions (e.g., Pasero et al., 1994). The general formula for ardennite-series minerals can be written as $\text{A}_4^{2+}(\text{M}_3^{3+}\text{M}^{2+})(\text{T}^{5+}\text{O}_4)(\text{T}^{4+}\text{O}_4)_2(\text{T}_3^{4+}\text{O}_{10})(\text{OH})_6$ (e.g., Donnay and Allmann, 1968; Nagashima and Armbruster, 2010). These minerals have orthorhombic symmetry with a space group $Pnmm$ and consist of chains of edge-sharing MO_6 octahedra running along the b -axis, linked to TO_4 and T_3O_{10} tetrahedral groups via AO_7 polyhedra (Fig. 1). There are two A sites for A^{2+} cations, which are primarily occupied by Mn^{2+} . Among the three octahedral sites, the M1 and M2 sites are occupied only by trivalent cations, while the M3 site is shared by both trivalent and divalent cations, with $\text{M}_{0.5}^{3+}\text{M}_{0.5}^{2+}$ as the ideal occupancy. Si exclusively oc-

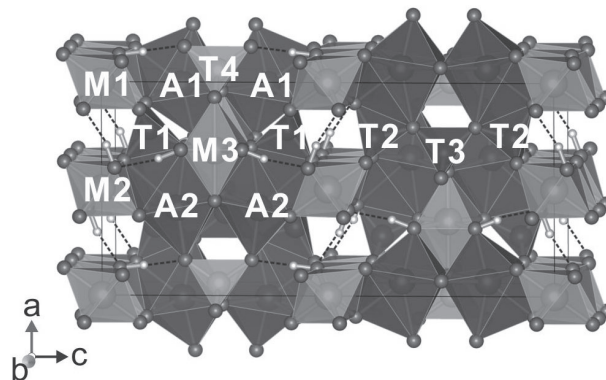


Figure 1. Structure of ardennite-series minerals obtained using VESTA program (Momma and Izumi, 2011).

cupies the T1, T2, and T3 sites, and pentavalent cations such as As and V dominate at the T4 site, with lesser amounts of Si also at this site (Nagashima and Armbruster, 2010). Although the c periodicity of ardennite-series minerals is approximately 19 $\text{\AA}$, anomalous ardennite with a double c periodicity (approximately 37 $\text{\AA}$) has been reported by Pasero and Reinecke (1991). This material was later determined to represent a new mineral called lavoisierite, which has structural features between those

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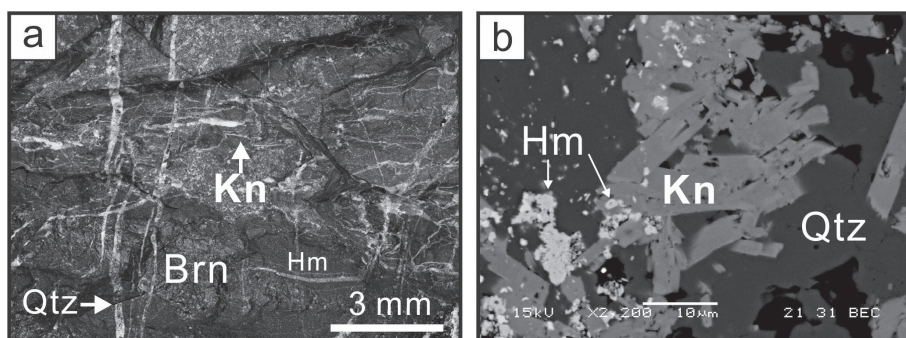


Figure 2. (a) Photographic image of quartz vein containing kannanite in iron-manganese ore sample, and (b) backscattered electron image of kannanite. Legend: Brn, braunite; Hm, hematite; Kn, kannanite; Qtz, quartz. Color version is available online from <https://doi.org/10.2465/jmps.180103>.

of ardennite and sursassite (Orlandi et al., 2013).

During a mineralogical survey of the iron-manganese deposit on Kannan Mountain in Ehime Prefecture, Japan, our own group collected an ardennite-like mineral from an outcrop. Subsequent studies revealed that the mineral is a new phase of Ca-dominant member of the ardennite series. This mineral was named kannanite after the location where it was found, and both the mineral and name have been approved by the International Mineralogical Association, Commission on New Minerals, Nomenclature and Classification (2015-100). A holotype specimen has been deposited in the collection of the National Museum of Nature and Science, Japan (NSM M-44527).

OCCURRENCE AND GENERAL APPEARANCE

The Ozu district is located in the western part of Ehime Prefecture on Shikoku Island, Japan. The geology of this district primarily consists of a Jurassic accretionary complex of the Northern Chichibu terrane, Sanbagawa Metamorphic Complex, Miocene dyke, and Quaternary sediments. Banno et al. (2010) divided the Sanbagawa Metamorphic Complex within the Ozu district into six units according to geological features: the Ozu, Nakaidani, Fukuoka, Kannan, Uchiko, and Iyo units.

Kannan Mountain (33°31'02N 132°37'58"E) is located in the southern part of the Ozu district, and it is mainly comprised of metamorphic rocks of the Kannan unit. The Kannan unit corresponds to Mikabu Greestone and consists of metamorphosed basaltic tuff and lava, metagabbro, metadolerite, and metachert. Pumpellyite and actinolite with minor amounts of epidote commonly occur in the mafic rocks, and thus this unit was evidently exposed to metamorphism in the pumpellyite-actinolite facies (e.g., Sakakibara et al., 1998). The metachert is distributed over the southeast side of the mountain and contains small-scale iron-manganese deposits. Our samples were collected from an outcrop of these deposits which occur as a layer of iron-manganese ore in the metachert with a thickness of approximately 50 cm. The ores

have a reddish-black coloration and are fragile due to the development of cracks. The ore specimens consist mainly of quartz and hematite, in association with braunite. A fine-grained hematite is scattered throughout the ore, with occasional lenticular aggregates of braunite with thicknesses of several mm to 5 cm. Apatite occurs as very minor constituent mineral in the ore. The ore sample is penetrated by numerous quartz veins with widths ranging from several μm to 2 cm. Although the majority of these quartz veins is either not accompanied by other minerals or occurs together with piemontite, veins containing orange-colored minerals were found in a part of the specimen (Fig. 2a). These minerals are kannanite and ardennite-(V), and kannanite occurs in fine quartz veins crossing the hematite-rich part. Minor piemontite and hematite also occur in the quartz vein including kannanite and ardennite-(V). The quartz vein with these ardennite-series minerals is also crossed by another pure quartz vein. Kannanite and ardennite-(V) are considered to be formed by the activities of metamorphic fluid accompanied with the Sanbagawa metamorphism.

PHYSICAL AND OPTICAL PROPERTIES

The quartz vein containing kannanite is pale orange in color (Fig. 2a). Under microscope, kannanite in thin sections exhibits weak pleochroism from orange to brownish orange. The mineral has a vitreous luster, and fluorescence was not observed under short- and long-wavelength ultraviolet light. The grain size is typically several μm and rarely up to 15 μm . The crystals are fragmented into anhedral grains in most cases, and tabular or columnar shapes were rarely seen during scanning electron microscopy observations (Fig. 2b). In either case, many voids were observed in the crystals. The Mohs hardness of the specimen is 6, as determined by rubbing the standard powder on a thin section. The cleavage plane for the sample could not be determined due to the small grain sizes, although it is expected to be perfectly along the $\{010\}$ plane by analogy with ardennite-series minerals. The density of this mineral as

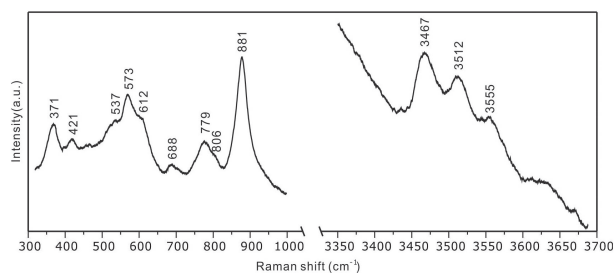


Figure 3. Raman spectra of kannanite.

calculated based on the empirical formula and powder unit cell data is 3.43 g cm^{-3} . Although the optical properties were not measured owing to the small grain size, the mean refractive index obtained from the Gladstone–Dale relationship (Mandarino, 1981) using the empirical formula and calculated density is 1.788.

RAMAN SPECTROSCOPY

A Raman spectroscopic analysis was performed using a 514.5 nm argon laser at a power setting of 50 mW in conjunction with a HORIBA HR320 spectrometer. Peak positions were calibrated using silicon, and the resulting Raman spectra are shown in Figure 3, in which peaks are evident at 300–1000 and 3300–3700 cm^{-1} but not in the range of 1000–3000 cm^{-1} . Based on the Fourier transform infrared (FT-IR) analysis of ardenite-(As) by Frost et al. (2014), the peaks may be assigned as follows: 371 cm^{-1} to ν_2 symmetric bending vibrations, 421 cm^{-1} to ν_4 out-of-plane bending modes for VO_4 units, 779–881 cm^{-1} to VO_4 antisymmetric and symmetric stretching vibrations, and 3400–3600 cm^{-1} to O–H stretching vibrations.

CHEMICAL COMPOSITION

Chemical analyses were carried out using a JEOL JXA-8230 electron microprobe (WDS mode, 15 kV, 20 nA, 1 μm beam diameter) at the Centre for Instrumental Analysis, Yamaguchi University. The ZAF method was employed for data correction. A JEOL JSM-5600 electron microprobe (EDS mode, 15 kV, 0.4 nA, 1 μm beam diameter) was also used for chemical analysis. The presence of OH groups was confirmed by Raman spectroscopy while the H_2O content was calculated based on stoichiometry because it could not be directly measured due to the paucity of material. Table 1 summarizes the WDS analysis ($n = 12$) of kannanite from a single vein. The $\text{Mn}^{2+}/\text{Mn}^{3+}$ ratio and total H_2O wt% are calculated by the stoichiometry as 28 anions and 16 cations ($\text{Si} + \text{V} + \text{As} = 6$ and others = 10). The total iron was estimated as Fe_2O_3 . The total wt% obtained from these calculations was slightly less than 100, suggesting electron beam dam-

Table 1. Mean chemical composition data ($n = 12$) for kannanite obtained from single vein by electron microprobe analysis

	wt% Mean (Min.-Max)	Standards		
V_2O_5	6.04 (5.49–6.53)	$\text{Ca}_3(\text{VO}_4)_2$	Ca	3.60
As_2O_5	1.52 (1.38–1.78)	GaAs	Mn^{2+}	0.40
SiO_2	29.37 (28.73–29.84)	CaSiO_3	ΣA	4
Al_2O_3	14.40 (13.85–15.13)	Al_2O_3		
Fe_2O_3	5.18 (4.83–5.44)	Fe_2O_3	Al	3.00
MnO	12.65 (11.86–13.99)	MnO	Mn^{3+}	1.31
CaO	19.02 (17.34–19.68)	CaSiO_3	Mg	0.71
MgO	2.70 (2.57–2.82)	MgO	Fe^{3+}	0.69
CuO	0.34 (0.23–0.46)	Cu	Mn^{2+}	0.19
NiO	0.40 (0.30–0.51)	NiO	Ni	0.06
Total	91.62		Cu	0.05
MnO*	3.89		ΣM	6
Mn_2O_3^*	9.74			
H_2O^*	5.22		V	0.70
Total	97.83		As	0.14
			Si	0.16
			ΣT4	1
			Si	5
			O	21.84
			OH	6.16

* Calculated from stoichiometry (28 anions and 16 cations).

age or some effect of voids hidden under the beam spot. The empirical formula for kannanite is $(\text{Ca}_{3.60}\text{Mn}_{0.40}^{2+})_{\Sigma 4}(\text{Al}_{3.00}\text{Mn}_{1.31}^{3+}\text{Fe}_{0.69}^{3+}\text{Mg}_{0.71}\text{Mn}_{0.19}^{2+}\text{Ni}_{0.06}\text{Cu}_{0.05})_{\Sigma 6}[(\text{V}_{0.70}\text{Si}_{0.16}\text{As}_{0.14})_{\Sigma 1}\text{O}_{3.84}(\text{OH})_{0.16}](\text{SiO}_4)_2(\text{Si}_3\text{O}_{10})(\text{OH})_6$. The simplified formula is $\text{Ca}_4[(\text{Al}, \text{Mn}^{3+}, \text{Fe}^{3+})_5\text{Mg}](\text{VO}_4)(\text{SiO}_4)_2(\text{Si}_3\text{O}_{10})(\text{OH})_6$.

The chemical composition of the ardenite-series minerals assessed in this study varied among veins, and their chemical variation is shown in Figure 4. Cations at the T4 sites are evidently not correlated with the $\text{Ca}/\text{A}(\text{Ca} + \text{Mn}^{2+})$ ratio. The V content always exceeds 0.5 apfu, while As and Si are found to be present at only minor concentrations at the T4 sites (Fig. 4a). Although the Al content decreases with increasing the Ca ratio, Al is the dominant trivalent cation at the M sites (Fig. 4b). Mn^{3+} is the second most common trivalent cation at the M sites, and its value increases somewhat with increasing the Ca ratio. Fe^{3+} is present at similar levels to Mn^{3+} at the Ca ratios below 0.5 but exhibits essentially no correlation with the Ca ratio. $\text{Mn}^{3+} + \text{Fe}^{3+}$ content correlates with the Ca ratio, and it continuously varies from 1 to 2 apfu with increasing the Ca ratio (Fig. 4b). Mg is the dominant divalent cation at the M sites (Fig. 4c) and always has a content greater than 0.5 apfu. Although the correlation between Mg content and the Ca ratio is very weak, Mg

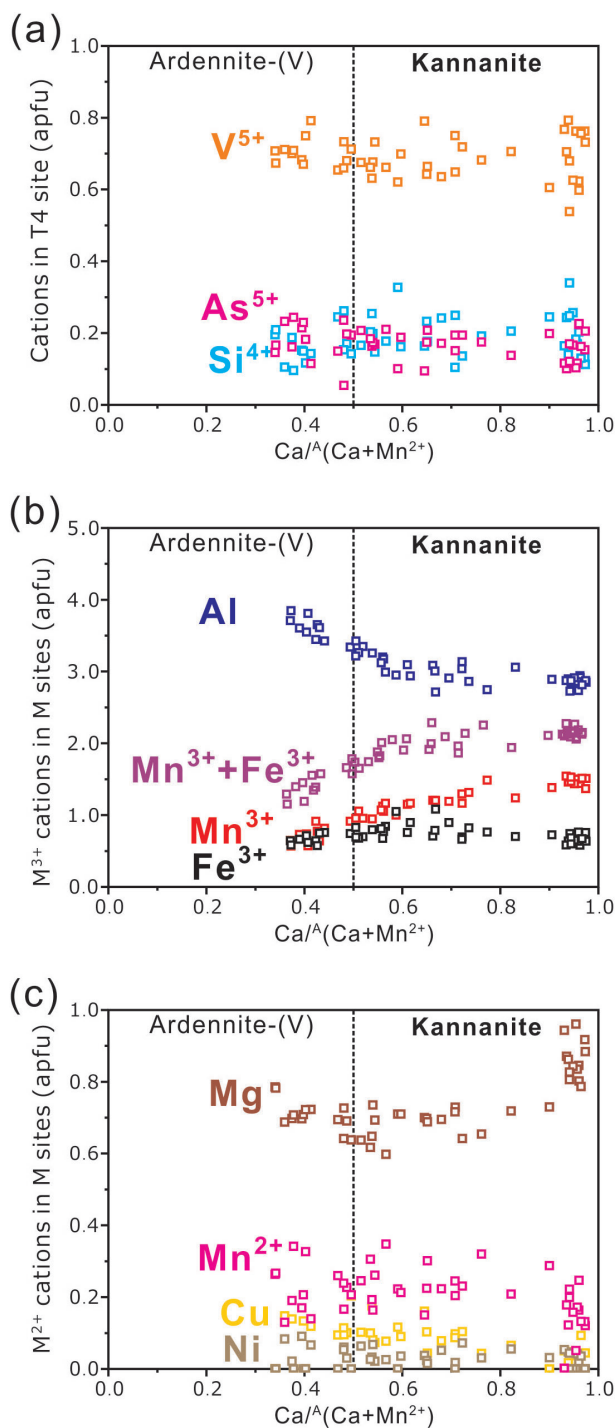


Figure 4. Compositional variations of the ardennite-series minerals of (a) cations in T4 site, (b) M^{3+} cations in M sites, and M^{2+} cations in M sites on $\text{Ca}/\text{A}(\text{Ca} + \text{Mn}^{2+})$.

content is particularly high for high Ca ratios. Mn^{2+} is the second most common divalent cation at the M sites and shows no relationship with the Ca ratio. Cu and Ni, both of which are relatively small divalent cations, exhibit no correlations with the Ca ratio.

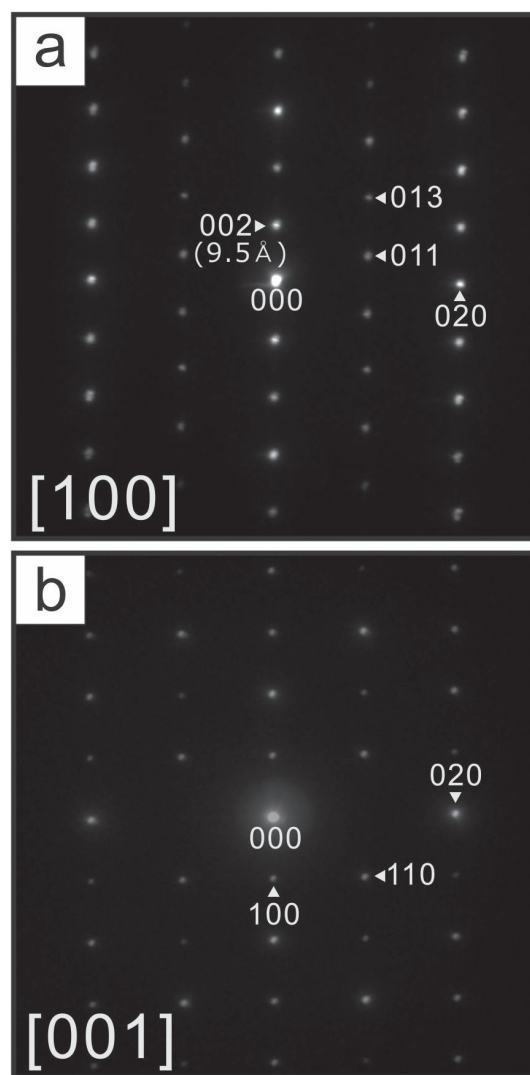


Figure 5. Selected area electron diffraction patterns for kannanite: (a) [100], and (b) [001] directions.

CRYSTALLOGRAPHY

Kannanite is present within the quartz vein in the ore sample as aggregates of fine grains up to 15 μm in size, and this small grain size precluded single-crystal X-ray analysis. Instead, the crystal structure was examined by transmission electron microscopy (TEM) and powder X-ray diffraction (XRD).

The c periodicity and the space group of kannanite were confirmed by TEM (JEOL-2010F, operating at 200 kV) prior to XRD analysis. Samples were prepared by Ar milling using a JEOL Ion-Slicer (EM-09100IS). Figure 5 shows selected area electron diffraction patterns obtained along the [100] and [001] directions. The [100] pattern shows $00l$ reflection spots at $l = 2n$, and no streaking is apparent. The c period calculated from the 002 reflection

Table 2. X-ray powder diffraction data for kannanite

I/I_0	hkl	$d_{\text{obs.}} (\text{\AA})$	$d_{\text{calc.}} (\text{\AA})$	I/I_0	hkl	$d_{\text{obs.}} (\text{\AA})$	$d_{\text{calc.}} (\text{\AA})$
16	100	8.889	8.802	13	220	2.486	2.484
11	101	8.024	8.036	17	222	2.401	2.402
7	103	5.134	5.135	25	117	2.370	2.370
18	110	4.967	4.967	19	216, 008	2.359	2.360, 2.3603
8	111	4.790	4.804	37	207	2.306	2.305
34	004	4.720	4.721	25	401	2.205	2.205
12	200	4.439	4.440	13	026	2.170	2.170
15	112	4.392	4.396	16	403, 321	2.093	2.094, 2.093
20	201	4.327	4.322	32	208	2.083	2.084
19	202	4.017	4.018	23	225	2.074	2.075
19	113	3.899	3.899	10	109	2.042	2.042
17	203	3.627	3.628	9	316	2.030	2.029
9	114	3.422	3.422	5	404	2.009	2.009
37	204	3.234	3.234	4	127	1.956	1.955
11	015	3.200	3.195	5	131	1.938	1.938
6	213	3.102	3.104	6	405	1.915	1.914
100	115, 020	3.001	3.006, 2.996	4	209	1.897	1.897
30	300	2.963	2.960	16	028	1.854	1.854
18	301	2.930	2.924	12	325	1.840	1.839
13	205	2.876	2.877	6	1110	1.765	1.765
26	022	2.857	2.856	6	502	1.746	1.745
35	121	2.809	2.807	7	2010	1.737	1.738
66	122	2.715	2.719	7	135	1.731	1.732
67	116, 310	2.658	2.658, 2.6539	5	407	1.714	1.714
22	311	2.628	2.628	14	511	1.694	1.696
22	206	2.568	2.568	19	408	1.617	1.617
47	024	2.531	2.530	8	0012	1.574	1.574

$$a = 8.8802(14), b = 5.9919(13), c = 18.882(3) \text{ \AA}, V = 1004.7(3) \text{ \AA}^3$$

is approximately 19 Å, corresponding to that of ardenite-series minerals. The extinction rules for the [100] and [001] directions are $k + l = \text{odd}$, $k = \text{odd}$, and $l = \text{odd}$ for the $0kl$, $0k0$, and $00l$ reflections, respectively, which are also in agreement with the $Pnmm$ space group for ardenite-series minerals. These data confirm that kannanite is a member of the ardenite series.

Subsequent to the above analysis, an 80 μm^2 sample of the quartz vein containing kannanite that had just been analyzed was crushed and pelletized using a diamond anvil. The obtained sample was placed on Kapton tape and XRD data were collected using a synchrotron X-ray source on the NE1 beam line at the Photon Factory Advanced Ring (PF-AR) institute, KEK, Japan. This source provided a 30 μm diameter collimated beam of monochromatized X-rays ($\lambda = 0.4183 \text{ \AA}$). The XRD spectra were collected by the Debye-Scherrer method, recorded via an imaging plate detector, and then converted to conventional one-dimensional profiles using the IPAnalyzer and PDindexer software packages (Seto et al. 2010). The resulting data

excluding quartz reflections are summarized in Table 2. The seven strongest lines in the powder XRD pattern [d in $\text{\AA}(I/I_0)(hkl)$] were 3.234(37)(204), 3.001(100)(115, 020), 2.809(35)(121), 2.715(66)(122), 2.658(67)(116, 310), 2.531(47)(024), and 2.306(37)(207). Based on these data, kannanite can be indexed to the orthorhombic $Pnmm$ space group, and the unit cell parameters as refined from the powder data are $a = 8.8802(14) \text{ \AA}$, $b = 5.9919(13) \text{ \AA}$, $c = 18.882(3) \text{ \AA}$ and $V = 1004.7(3) \text{ \AA}^3$. The $a:b:c$ ratio calculated from the above unit cell parameters is 1.48:1:3.15.

DISCUSSION

Ardennite-series minerals are classified in terms of cations in A, M, and T4 site (Fig. 1). The T4 site is preferentially occupied by As or V, and kannanite is V-dominant species (Fig. 4a). There are two A sites for A^{2+} cations and kannanite is characterized by Ca dominance (Table 1). In three M sites, the M1 and M2 sites are occupied only by trivalent cations, and the M3 site is shared by

both trivalent and divalent cations (Fig. 1). In kannanite, the half of M3 site is occupied by Mg and small amount of Mn^{2+} (Fig. 4c), and other half in M3 and M1 and M2 sites should be filled by the trivalent cations. The order of volume in the M sites is $\text{M3} > \text{M1} > \text{M2}$ in the case of ardennite-(As) (Nagashima and Armbruster, 2010). If kannanite structure also shares its characteristics, larger cations such as Mn^{3+} and Fe^{3+} included in kannanite may prefer M3 and M1 sites. However, the single crystal XRD study could not be determined due to the small size in this study. The order of amount of the trivalent cations in kannanite is $\text{Al} > \text{Mn}^{3+} > \text{Fe}^{3+}$, and Al is the dominant compared to $\text{Mn}^{3+} + \text{Fe}^{3+}$ (Fig. 4b). Therefore, the simplified formula of kannanite is represented as $\text{Ca}_4[(\text{Al}, \text{Mn}^{3+}, \text{Fe}^{3+})_5\text{Mg}](\text{VO}_4)(\text{SiO}_4)_2(\text{Si}_3\text{O}_{10})(\text{OH})_6$, which is distinguishable from ardennite-(V) $\text{Mn}_4^{2+}(\text{Al}_5\text{Mg})(\text{VO}_4)(\text{SiO}_4)_2(\text{Si}_3\text{O}_{10})(\text{OH})_6$. A follow-up crystallographic study for site assignment is expected in order to better understand the crystal chemistry of ardennite-series minerals in the future.

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SUPPLEMENTARY MATERIAL

Color version of Figures 2 is available online from <https://doi.org/10.2465/jmps.180103>.

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