

Ammonioleucite, a new mineral from Tatarazawa, Fujioka, Japan

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ABSTRACT

Ammonioleucite, ideally $(\text{NH}_4)\text{AlSi}_2\text{O}_6$, occurs as white surface layers replacing analcime crystals in green dolomitized crystalline schist of the Sanbagawa metamorphic belt, Tatarazawa, Fujioka, Gunma Prefecture, Japan.

Ammonioleucite is tetragonal, space group $I4_1/a$, $a = 13.214(1)$, $c = 13.713(2)$ Å; $c/a = 1.0378$; $V = 2394.4$ Å³; $Z = 16$. The strongest X-ray powder data are [d in Å, (I/I_0) , (hkl)]: 5.53(50)(112), 5.43(100)(211), 3.43(40)(004), 3.30(80)(400), 2.955(20)(420), 2.859(20)(323). The mineral is uniaxial positive, $n = 1.518(2)$, $\epsilon - \omega$ = very small; single crystals are very small and twinned; color white, translucent; luster resinous to vitreous. Hardness was not determined owing to very fine grain size. $D_{\text{calc}} = 2.24$ g/cm³, $D_{\text{meas}} = 2.29(5)$ g/cm³.

Chemical analysis of a mechanically separated sample, after reducing analcime and dolomite and normalizing to 100%, gave SiO₂, 62.66; Al₂O₃, 22.43; (NH₄)₂O, 8.70; K₂O, 4.43; H₂O, 1.78; total 100 wt%, corresponding to $[(\text{NH}_4)_{0.68}\text{K}_{0.19}]_{20.87}\text{Al}_{0.89}\text{Si}_{2.12}\text{O}_6$, based on six oxygen atoms. Differential thermal analysis shows one exothermic peak at 485°C; thermogravimetry shows weight loss between 415 and 550°C. The new mineral is named for the chemical composition and relationship to leucite.

INTRODUCTION

During a recent mineralogical study of a quarry at Tatarazawa, Fujioka, Gunma Prefecture, Japan, an unknown white material coating analcime crystals was discovered by one of the authors (HH) lining cavities in a hydrothermally altered greenschist. Subsequent chemical, infrared, and X-ray studies have shown this white material to be a new mineral, the ammonium analogue of leucite, which may represent the first natural occurrence of the synthetic ion-exchange compound $(\text{NH}_4)\text{AlSi}_2\text{O}_6$ (Barrer, 1950; Barrer et al., 1953).

The new mineral is named ammonioleucite for its chemical composition and relation to leucite. Only two other ammonium aluminosilicates have been found naturally. They are buddingtonite (NH_4 -sanidine) (Erd et al., 1964) and tobelite (NH_4 -muscovite) (Higashi, 1982). The name and mineral have been approved by the IMA Commission on New Minerals and Mineral Names. Type material has been deposited at the National Science Museum, Tokyo.

OCCURRENCE AND GEOLOGY

The quarry at Tatarazawa, which is operated by the Tomioka Kogyo Company, is located about 100 km north

of Tokyo, in the northwest area of the Kanto Mountains of the Main Island. The Median tectonic line, which is the largest in Japan, passes through this region and is considered the most prominent geologic feature of the area. Fracture zones located in the general region, and specifically in the quarry, may belong to the Median tectonic system. This theory has found increasing support in recent years. Fracture zones are widely distributed in this region, and contacts of crystalline schists of the Sanbagawa metamorphic rocks with Tertiary sandstones are common.

Because of the increasing interest in these geologic features, a research group consisting mainly of high school teachers has been surveying the geology of this region (Kaburagawa Research Group, 1985). One member of this group, T. Marubashi, discovered samples of dawsonite and nordstrandite in the sandstones at Tatarazawa (Kizaki et al., 1981). Later, one of the authors (HH) discovered a series of interesting minerals at the quarry, including the new mineral described herein.

Specifically, the quarry exposes a white arkose sandstone which rarely contains fossils of bivalves. The contact zone within the quarry is composed of a crystalline schist that has been dolomitized within several meters on each side of the contact zone. Significant carbonitization has been noted in the contact zone.

The veinlets, fractures, and cavities in the hydrothermally altered schist contain abundant crystals of dolomite

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in addition to analcime, ammonioleucite, and their mixtures. Associated minerals identified within the quarry include calcite, quartz, siderite, chromian aluminohydrocalcite, chromian dawsonite, vaesite, violarite, millerite, pyrite, and chalcopyrite.

At one location in the quarry, a spring containing hydrogen sulfide occurred. Opalized dawsonite (or aluminohydrocalcite) was identified here but has since been buried and is not accessible now. The area surrounding this quarry contains a number of mineral springs including Inoda mineral spring about 2 km from the quarry. These springs are considered to be traces of the hydrothermal activity of this region. The spring water of the Isobe hot spring about 17 km northwest of the quarry has been determined to contain about 19.6 ppm of ammonium ion (Geological Survey of Japan, 1957).

MORPHOLOGY

No single measurable ammonioleucite crystal was observed during this study. However, the crystals of analcime, which have been partially or wholly replaced by ammonium ion exchange, are trapezohedral in habit. Forms present are $n\{211\}$, $a\{100\}$, and rarely $e\{210\}$. The $e\{210\}$ form has not been observed in analcime before. A typical group of partially altered analcime crystals is shown in Figure 1.

X-RAY CRYSTALLOGRAPHY

Powder-diffraction data for ammonioleucite was obtained using a Rigaku RAD-1A powder diffractometer, Ni-filtered $\text{CuK}\alpha$ radiation with an external standard of Si. The data for ammonioleucite and those for leucite (Faust, 1963) are listed in Table 1. The diffraction patterns of both minerals resemble each other, and ammonioleucite is presumed to be isostructural with leucite (Mazzi et al., 1976). One of the characteristic differences between ammonioleucite and leucite is the relative intensity of 211 and 400 reflections: for ammonioleucite their relative intensities are 100 and 80, respectively, whereas for leucite they are 80 and 100.

An X-ray single-crystal study using a Weissenberg camera revealed all crystals to be twinned. The two individuals of the twin have parallel a (or b) axes, whereas the remaining two axes are not parallel, and such twinning is observed in leucite (Korekawa, 1969). These individual segments of the twins give close but distinct spots on the X-ray photographs. The grain of ammonioleucite consists of many minute single crystals that are related by the above twin orientation. On the basis of diffraction photographs, ammonioleucite was found to be tetragonal with space group $I4_1/a$.

The unit-cell parameters of ammonioleucite were calculated from the powder data using the least-squares refinement program APLEM (Appleman and Evans, 1973) and are compared with leucite (Faust, 1963) and synthetic NH_4 -leucite (Barrer et al., 1953) in Table 2. The cell parameters of ammonioleucite are close to those of leucite

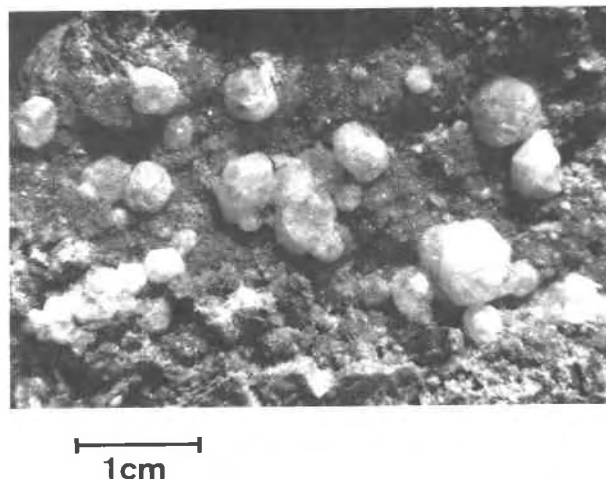


Fig. 1. Ammonioleucite, shown as white surface layers replacing trapezohedral analcime crystals.

and synthetic NH_4 -leucite: c_0 of ammonioleucite lies between c_0 of the latter two phases, whereas a_0 of ammonioleucite is slightly longer than a_0 of both leucite and its synthetic NH_4 analogue.

PHYSICAL AND OPTICAL PROPERTIES

Aggregates of ammonioleucite are white and translucent and have a resinous to vitreous luster. The mineral is uniaxial (+), with $n = 1.518(2)$, and $\epsilon - \omega$ is near zero in white light. The refractive index is very close to those for leucite (1.508) and synthetic NH_4 -leucite (1.524) and is intermediate between the two values.

The density, determined by the floating method for coarse powder, is 2.29(5) g/cm^3 ; the calculated density based on the ideal formula $[(\text{NH}_4)_{0.78}\text{K}_{0.22}]\text{AlSi}_2\text{O}_6$ is 2.24 g/cm^3 . The hardness could not be determined because of the very small crystal aggregate size and the close association with analcime.

CHEMICAL ANALYSIS

A sample of ammonioleucite was carefully separated from associated minerals by mechanical methods. The final purity of the resulting sample was checked by X-ray powder diffractometry. However, complete separation from the associated minerals was not achieved, and the quantitative analysis was performed on a sample containing a small amount of both analcime and dolomite.

The analysis was performed by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) for Si, Al, K, Na, Ca, Fe, and Mg. Organic elementary analysis was used for N, H, and C. The lithium metaborate fusion method (Burman et al., 1978) was used to decompose the sample. A 50-mg quantity of lithium metaborate was added to about 10 mg of sample in a Pt crucible and heated to about 900°C for 30 min. After cooling, the melt was dissolved in hot 1 N nitric acid. The solution was analyzed on a Jarrel-Ash Mk II Plasma Atomcomp ICP spectrom-

Table 1. X-ray powder-diffraction pattern of ammonioleucite compared with pattern of leucite

<i>h k l</i>	Ammonioleucite*			Leucite**		<i>h k l</i>	Ammonioleucite*			Leucite**	
	<i>d_{calc.}</i>	<i>d_{obs.}</i>	<i>I/I₀</i>	<i>d_{obs.}</i>	<i>I/I₀</i>		<i>d_{calc.}</i>	<i>d_{obs.}</i>	<i>I/I₀</i>	<i>d_{obs.}</i>	<i>I/I₀</i>
1 0 1	9.5	9.5	3	9.5	40	7 0 3	1.745	1.745	1		
2 0 0	6.61	6.59	2	6.6	14	3 2 7	1.728	1.729	2	1.726	40
1 1 2	5.53	5.53	50	5.54	50	0 0 8	1.714				
2 1 1	5.43	5.43	100	5.39	80	5 1 6	1.714	1.715	1		
2 0 2	4.76	4.75	5	4.75	40	7 2 3				1.671	20
1 0 3				4.34	20	7 3 2	1.682	1.684	5	1.663	55
2 2 2				3.78	30	5 5 4	1.641	1.641	1		
2 1 3	3.62	3.61	1	3.60	40	7 1 4				1.629	40
3 2 1	3.54	3.54	7	3.52	35	6 4 4				1.605	35
0 0 4	3.43	3.43	40	3.44	85	8 2 0	1.602	1.604	2	1.585	40
4 0 0	3.30	3.30	80	3.27	100	6 5 3	1.587				
1 1 4				3.22	8	3 1 8	1.586	1.588	1		
3 0 3	3.17	3.17	2	3.16	8	6 0 6	1.586				
4 1 1	3.12	3.12	3	3.09	35	4 3 7	1.574	1.574	1		
2 0 4	3.04	3.04	3	3.04	30	7 4 3	1.543				
4 2 0	2.955	2.955	20	2.918	70	6 2 6	1.542	1.544	2	1.533	35
3 2 3	2.859	2.859	20	2.842	70	5 2 7				1.526	35
3 3 2	2.836	2.839	10	2.808	50	3 3 8	1.502				
4 2 2	2.714	2.714	1	2.685	8	7 5 2	1.499	1.501	1		
3 1 4	2.650	2.653	7	2.641	40	4 2 8				1.481	40
4 1 3	2.624	2.624	2	2.602	20	2 1 9	1.475	1.476	1		
2 1 5	2.488	2.489	3	2.487	30	8 2 4				1.439	30
5 1 2	2.424	2.422	3			9 1 2	1.427				
4 0 4	2.379	2.379	7	2.366	65	7 6 1	1.426	1.427	1	1.409	35
4 4 0	2.336			2.308	30	5 4 7				1.414	16
3 0 5	2.328	2.333	2	2.329	30	6 3 7				1.382	35
4 3 3	2.288	2.288	1			7 3 6	1.382	1.382	1	1.373	40
5 0 3				2.270	8	4 1 9	1.376	1.376	1		
1 1 6	2.220	2.218	1			1 1 10	1.357	1.357	1	1.360	30
3 2 5	2.196	2.197	1	2.192	8	8 5 3	1.339	1.337	1		
2 0 6				2.162	30	8 0 6	1.339				
5 3 2	2.160	2.153	5	2.127	50	9 4 1				1.320	16
6 2 0	2.089	2.088	2	2.065	30	7 2 7				1.324	20
5 4 1	2.041	2.042	1	2.012	20	8 6 0	1.321				
6 2 2	1.999	1.999	1	1.980	8	4 3 9	1.320	1.323	1		
1 0 7				1.943	20	7 7 2	1.310	1.310	1		
4 4 4	1.930	1.932	2	1.919	20	3 1 10				1.305	50
5 3 4				1.877	20	9 4 3	1.287				
5 4 3	1.881					6 6 6	1.287	1.288	1		
4 0 6	1.880	1.880	2			9 5 2	1.262	1.262	1		
2 1 7				1.862	20	6 1 9	1.247	1.248	1		
3 3 6	1.843	1.843	1	1.837	20	1 0 11				1.294	50
6 4 0	1.832	1.834	1			2 1 11	1.220				
4 2 6	1.808					10 3 3	1.220	1.220	1		
5 5 2	1.803	1.807	2			7 3 8	1.219				
7 1 2				1.785	20	8 3 7	1.214	1.214	1		

* Present study.

** Faust (1963).

eter. The results of the ICP-AES analysis and the organic elementary analysis are shown in Table 3. N is shown as $(\text{NH}_4)_2\text{O}$, and the H remaining after removing that needed to combine with N is shown as H_2O .

Electron-microprobe analysis of the pure specimen was performed using a JEOL JXA-50A X-ray microanalyzer, and detected only Si, Al, K, N, and a trace of Na. No other element with atomic number greater than 10 was detected. Accordingly, Ca, Fe, Mg, and Na detected by ICP-AES were considered to be associated with dolomite and analcime. The chemical composition of ammonioleucite was calculated by subtracting from the total analysis those chemical elements attributed to the associated minerals. Table 3 shows the analytical results. In the calculation, all Na was attributed to analcime with the composition $(\text{Na}_{0.864}\text{K}_{0.023})\text{Al}_{0.899}\text{Si}_{2.104}\text{O}_6 \cdot 1.01\text{H}_2\text{O}$ (analyzed composi-

tion of associated analcime). Ca, Fe, and Mg were attributed to dolomite $\text{Ca}(\text{Mg},\text{Fe})(\text{CO}_3)_2$; a small amount of CO_2 was neglected.

The calculated water content of 1.8% was neglected in the construction of the chemical formula because the infrared absorption spectrum of a sample dried at 110°C exhibited no absorption peaks due to water; also, the TG-DTA analysis did not show any peaks corresponding to lattice water. Barrer et al. (1953) reported that about 1% water was present in their synthetic NH_4 -leucite as residual water.

Consequently, the chemical formula of ammonioleucite based on six oxygen atoms is



The ideal formula is $(\text{NH}_4,\text{K})\text{AlSi}_2\text{O}_6$.

Table 2. Crystallographic data for ammonioleucite, leucite, and NH_4 -leucite

	Ammonioleucite*	Leucite**	NH_4 -leucite***
Formula unit	$(\text{NH}_4, \text{K})\text{AlSi}_2\text{O}_6$	KAlSi_2O_6	$\text{NH}_4\text{AlSi}_2\text{O}_6$
Crystal system	Tetragonal	Tetragonal	Tetragonal
Space group	$I4_1/a$	$I4_1/a$	—
<i>a</i>	13.214 (1) Å	13.060 Å	13.17 Å
<i>c</i>	13.713 (2)	13.751	13.69
Z	16	16	—

* Present study.
 ** Faust (1963).
 *** Barrer *et al.* (1953).

Detection and characterization of N

In order to confirm the presence of N as a constituent in ammonioleucite, secondary ion mass spectrometry (SIMS) was employed using O_2^+ as primary ions. N was detected in all analyzed points of the sample as N^+ ions with $m/e = 14$, together with Si^{2+} ions and CH_2^+ ions (from C coating) having similar m/e values. Figure 2a shows the result of the SIMS analysis.

Electron-microprobe analysis was also used for the qualitative analysis of N. The interference by higher-order reflections of Al and Si were eliminated by using a pulse-height analyzer. The sample was coated with Ag instead of C because the characteristic X-rays of N are absorbed by C. Figure 2b shows the result of the electron-microprobe analysis. The characteristic X-ray of N ($K\alpha$, $n = 1$) was observed at 31.6 Å.

A small sample of ammonioleucite was treated with

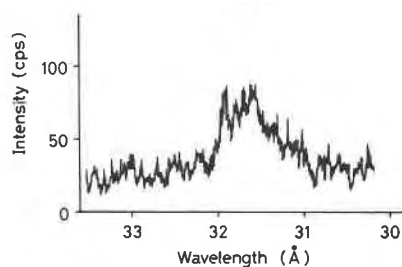
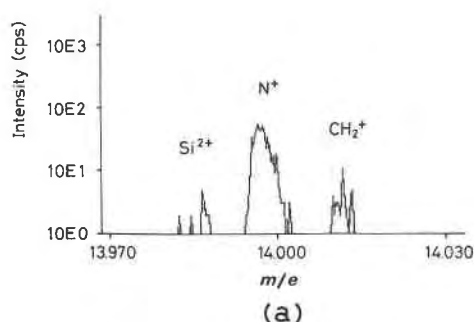


Fig. 2. Results of detection of nitrogen by SIMS (a) and electron microprobe (b).

Table 3. Chemical analysis of ammonioleucite

	wt. % ¹	wt. % ²	No. atoms ³
SiO_2	61.05	62.67	2.12
Al_2O_3	21.86	22.43	0.89
K_2O	4.11	4.43	0.19
Na_2O	0.712	0	—
CaO	0.449	0	—
MgO	0.184	0	—
FeO	0.129	0	—
$(\text{NH}_4)_2\text{O}$	8.01	8.70	0.68
H_2O	2.12	1.77	—
CO_2	2.05	0	—
Total	100.67	(100)	

1 Result of analysis of the sample with a small amount of contamination (See text).

2 After reduction of analcime and dolomite and normalized to 100 wt. %.

3 Based on 6 oxygen atoms.

Nessler's reagent dissolved in dilute hydrochloric acid. A brownish precipitation resulted confirming the presence of ammonium ion.

INFRARED SPECTROMETRY

Infrared absorption spectra were measured from powdered samples using the KBr disk method. The spectrum for ammonioleucite in the range $3800\text{--}330\text{ cm}^{-1}$ is compared with the spectra of leucite, analcime, and buddingtonite (Loughnan *et al.*, 1983) in Figure 3. Ammonioleucite shows characteristic absorptions by ammonium ion at 3250 , 3055 , and 2850 cm^{-1} for N-H stretching and 1435 cm^{-1} for H-N-H bending. Absorption peaks for ammonioleucite correspond to those of buddingtonite. Except for the absorptions by NH_4^+ ion, the absorption

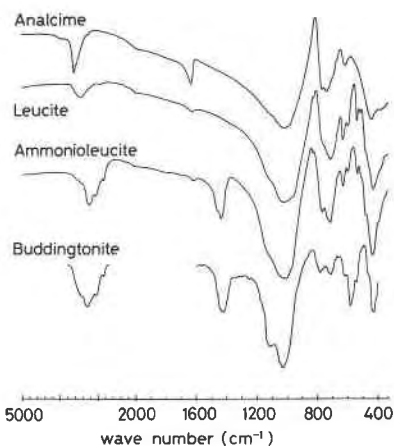


Fig. 3. Infrared absorption spectra of ammonioleucite. Those of analcime, leucite, and buddingtonite are also given for comparison (that for buddingtonite from Loughnan *et al.*, 1983).

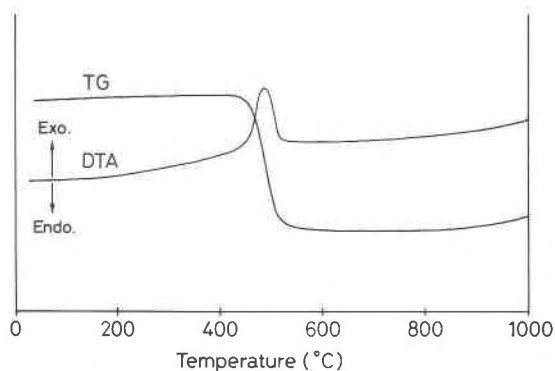


Fig. 4. DTA and TG curves of ammonioleucite.

spectra of ammonioleucite and leucite are very similar in shape and reflect the similar structures of both minerals.

Analcime, which contains zeolitic water and has a structure similar to leucite (Ferraris et al., 1972; Mazzi and Galli, 1978), shows characteristic absorption for water at 3625 cm^{-1} . Ammonioleucite, however, does not show this absorption and therefore does not contain any zeolitic or constitutional water.

THERMAL ANALYSIS

Differential thermal analysis (DTA) and thermogravimetry (TG) were performed on a 6.75-mg sample of powdered ammonioleucite from 25 to 1000°C at a heating rate of $5^{\circ}\text{C}/\text{min}$. The results of the DTA and TG are shown in Figure 4. The DTA curve shows only one exothermic peak at 485°C , and the TG curve indicates only one stage of weight loss between 415 and 550°C . This weight loss was attributed to the decomposition of ammonium ion to $\text{NH}_3 + \frac{1}{2}\text{H}_2\text{O}$. No other reaction peak was observed in the analyzed temperature range. An X-ray diffraction pattern of the heated specimen at 1000°C shows only an amorphous structure.

According to Erd et al. (1964), the DTA peak for the loss of ammonium ion in buddingtonite is about 608°C . Loughnan et al. (1983) reported that ammonium ion in buddingtonite is expelled slowly between 370 and 625°C . The evolution of ammonium from ammonioleucite is more rapid and occurs at lower temperatures than that from buddingtonite.

DISCUSSION

Ammonioleucite is closely related to leucite as shown by the similarity of X-ray powder pattern, optical properties, and infrared absorption spectrum. Data for synthetic NH_4 -leucite, reported by Barrer et al. (1953), also corresponds well to ammonioleucite. The ammonioleucite sample of this study contains a small amount of K. This would indicate the possibility of ion exchange between K and NH_4 . Since the ionic radii of both K and NH_4 ions are similar, 1.33 and 1.43 \AA , respectively, the leucite-type framework structure may be preserved in the K- NH_4 substitution.

In nature, ammonium aluminosilicates are very rare, and to date only two other minerals have been discovered. They are buddingtonite, having the K-feldspar (sanidine)-type framework, reported by Erd et al. (1964), and tobelite, having the muscovite-type framework, reported by Higashi (1982). Ammonioleucite reported in this study is the third ammonium aluminosilicate found in nature.

An experimental study by one of the authors (MY) showed that when analcime reacted with ammonium chloride or potassium chloride solutions at 150°C for 8 d, it changed to NH_4 -leucite or leucite, respectively. Barrer (1950) reported that analcime changed to leucite by treating with potassium chloride solution. Since the natural ammonioleucite of this study occurs as thin coatings on the surface of analcime crystals, the formation mechanism of ammonioleucite at the present locality is presumed to be related to the low-temperature hydrothermal reaction of the solution containing both ammonium and potassium ions with analcime.

Although experimental studies have shown that leucite is converted to analcime (Gupta and Fyfe, 1975) and analcime to leucite, natural leucite after analcime has not been reported. This study has shown that ammonioleucite has formed from natural analcime.

Edgar (1984) reported that the Al:Si ratio in analcime is more variable than in leucite and that the variations in analcime compositions reflect the mode of occurrence. At this locality, ammonioleucite and analcime have the same Al:Si ratio (14:34), based on 96 oxygen atoms. There appears to be no change in the Al:Si ratio by the ion-exchange conversion of analcime to leucite. Since a variable Al:Si ratio is unusual for leucite, this ratio can be useful in identifying natural leucite after analcime.

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