

ÅSKAGENITE-(Nd), $Mn^{2+}NdAl_2Fe^{3+}(Si_2O_7)(SiO_4)O_2$, A NEW MINERAL OF THE EPIDOTE SUPERGROUP¹

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A new epidote-supergroup mineral åskagenite-(Nd) was discovered in specimens from a granite pegmatite outcropped at the Åskagen deposit near the town of Filipstad, Värmland, Sweden, in association with potassic feldspar, quartz, bastnäsite, thorite, the Nd-dominant analogue of allanite-(Ce), brookite, gadolinite-(Y) and allopheane. The new mineral forms coarse prismatic and flattened crystals up to 1×4 cm in size. Åskagenite-(Nd) is black, with resinous lustre and brown streak, translucent in thin fragments; brittle, Mohs' hardness 6, fracture conchoidal. $D_{\text{meas}} = 3.737(5) \text{ g/cm}^3$ (for a metamict sample); $D_{\text{calc}} = 4.375 \text{ g/cm}^3$ (for a sample heated at 600°C during 1 h in nitrogen). Åskagenite-(Nd) is optically isotropic, $n = 1.712(2)$. IR spectrum is given. Chemical composition (electron microprobe, water determined by Alimarin method, $Fe^{2+}:Fe^{3+}$ by Mössbauer data, valency of Mn by XANES spectroscopic data, wt.%): CaO 0.27, Y_2O_3 2.27, La_2O_3 0.44, Ce_2O_3 7.99, Pr_2O_3 1.76, Nd_2O_3 11.21, Sm_2O_3 3.01, Yb_2O_3 0.21, ThO_2 0.72, MnO 7.98, FeO 7.75, Fe_2O_3 9.16, Al_2O_3 15.85, SiO_2 29.51, H_2O 0.55, total; 98.75. Crystal-chemical formula: $(Mn_{0.69}^{2+}Fe_{0.26}^{2+}Ca_{0.03})_{\Sigma 0.98}(Nd_{0.41}Ce_{0.30}Y_{0.12}Sm_{0.10}Pr_{0.07}La_{0.02}Yb_{0.01}Th_{0.02})_{\Sigma 1.05}(Al_{0.90}Fe_{0.10}^{3+})_{\Sigma 1.00}Al_{1.00}(Fe_{0.60}^{3+}Fe_{0.40}^{2+})_{\Sigma 1.00}Si_{2.90}O_{11}[O_{0.63}(OH)_{0.37}]_{\Sigma 1.00}$. Strong lines of X-ray powder-diffraction pattern of a heated sample d , Å (l ; hkl) are: 3.50(46; -211), 3.22(50; -212, 201), 2.897 (100; -301), 2.850(73; 020), 2.687(73; 120), 2.121(48; -403), 1.630(59; 124). Unit-cell parameters are: $a = 8.78(1) \text{ Å}$, $b = 5.710(6) \text{ Å}$, $c = 10.02(1) \text{ Å}$, $\beta = 114.6(2)^\circ$; $V = 456.7(8) \text{ Å}^3$, $Z = 2$; space group $P2_1/m$. Type material is deposited in the collections of the Technische Universität, Bergakademie Freiberg, Germany; inventory numbers are 82194 and 82218.

3 tables, 5 figures, 14 references.

Keywords: åskagenite-(Nd), epidote supergroup, pegmatite, Åskagen.

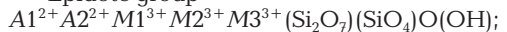
Introduction

According to the recommended nomenclature of epidote-group minerals that was approved recently by the IMA CNMNC, epidote group unites monoclinic (space group $P2_1/m$) minerals with the general crystal-chemical formula is $A_1A_2M_1M_2M_3(Si_2O_7)(SiO_4)\emptyset_4\emptyset_{10}$, where $A_1 = Ca, Mn^{2+}$; $A_2 = Ca, REE, Sr, Pb$; $M_1 = Al, Fe^{3+}, V^{3+}, Mn^{3+}, Cr^{3+}$; $M_2 = Al, Fe^{3+}$; $M_3 = Al, Fe^{3+}, V^{3+}, Mn^{3+}, Cr^{3+}, Mg, Fe^{2+}, Mn^{2+}$; $\emptyset_4 = O, F$; $\emptyset_{10} = OH$ and sometimes subordinate O (Armbruster *et al.*, 2006).

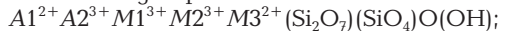
Subsequently, in accordance with new rules of the standartization of mineral group hierarchies (the proposal 09-A-bis), epidote group was renamed into epidote supergroup (Mills *et al.*, 2009), whereas clinzoisite (epidote), allanite and dollaseite subgroups were renamed into corresponding groups.

General formulae for members of these groups are:

Epidote group –



Allanite group –



Dollaseite group –



Åskagenite-(Nd), a new epidote-supergroup mineral described in the present article, is the first representative of a new group whose general formula can be written as follows: $A_1^{2+}A_2^{3+}M_1^{3+}M_2^{3+}M_3^{3+}(Si_2O_7)(SiO_4)O_2$.

The name was given after the locality and reflects the predominance of Nd over other *REE* in the site A_2 .

Type material is deposited in the collections of the Technische Universität, Bergakademie Freiberg, Germany. Inventory numbers are 82194 (holotype) and 82218 (cotype).

¹ – Åskagenite-(Nd) is approved by the IMA Commission on New Minerals, Nomenclature and Classification on December 1, 2009, IMA no. 2009-073.

Occurrence, general appearance and physical properties

Åskagenite-(Nd) was discovered in specimens collected at the beginning of 20th century in a small (5×20 m) working at the Åskagen quarry, near the former mining settlement Tor skebäck located 12 km ENE of Filipstad town, Värmlands län, Sweden (Wilke, 1997). The Åskagen quarry exploited for quartz from 1882 to 1916 is located within leptite zone developed along the eastern coast of the lake Yngen.

The working, where the new mineral was found, crosses a pegmatite body. In the latter, in association with åskagenite-(Nd) the following minerals are identified: potassic feldspar, quartz, bastnäsite, thorite, the Nd-dominant analogue of allanite-(Ce), brookite, gadolinite-(Y), as well as late allopheane.

The new mineral forms coarse prismatic and flattened crystals up to 1×4 cm in size, with orthogonal cross-section (Fig. 1). Åskagenite-(Nd) is black, translucent in thin fragments, with resinous lustre and brown streak. It is brittle, with conchoidal fracture. Mohs' hardness is 6. Cleavage is not observed. Measured density is $D_{\text{meas}} = 3.737(5) \text{ g/cm}^3$ (for a metamict sample); $D_{\text{calc}} = 4.375 \text{ g/cm}^3$ (for a sample heated at 600°C during 1 h in nitrogen).

Åskagenite-(Nd) is weakly radioactive that is the cause of its metamict state. As a result, the mineral is optically isotropic, $n = 1.712(2)$. Optical properties of åskagenite-(Nd) heated at 600°C during 1 h in nitrogen could not be deter-

mined due to the small size of single crystals and the presence of submicroscopic pores and cracks.

Chemical composition

Chemical composition of åskagenite-(Nd) was investigated by electron microprobe, with the scanning electron microscope Tescan Vega II XMU with the EDS mode spectrometer INCAX-sight at the accelerating voltage of 20 kV, electron beam current of 0.5 nA and electron beam diameter of 0.157 μm . The beam was rested on the area 16×16 μm . Four local analyses have been carried out. Mean values of the contents of constituents and their ranges are given in Table 1.

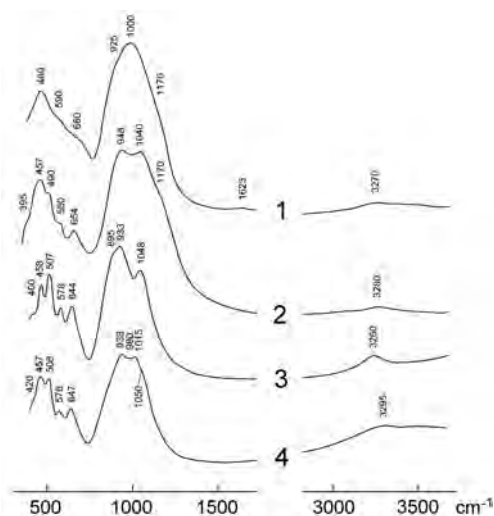
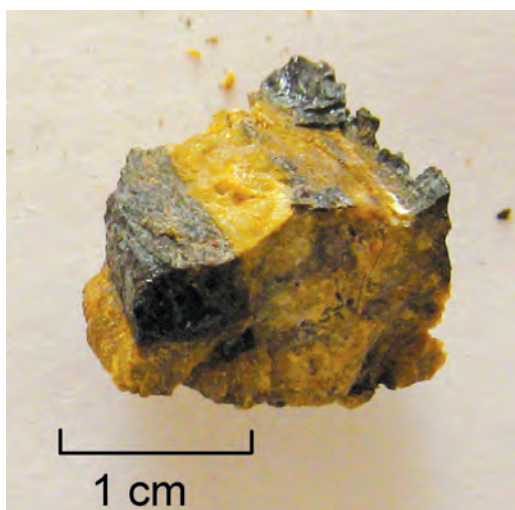
H₂O was determined by the Alimarin method (Penfield method modified for small samples with selective sorption of H₂O on Mg(ClO₄)₂ from gaseous products obtained by heating the mineral in oxygen at 1060°C). CO₂ was not analysed because of the absence of bands corresponding to C-O vibrations in the IR spectrum.

The empirical formula calculated on 13 oxygen atoms, taking into account the valencies of Fe and Mn (by Mössbauer and XANES data) is: $(\text{Mn}_{0.69}^{2+}\text{Fe}_{0.26}^{2+}\text{Ca}_{0.03})_{\Sigma 0.98}(\text{Nd}_{0.41}\text{Ce}_{0.30}\text{Y}_{0.12}\text{Sm}_{0.10}\text{Pr}_{0.07}\text{La}_{0.02}\text{Yb}_{0.01}\text{Th}_{0.02})_{\Sigma 1.05}(\text{Al}_{1.90}\text{Fe}_{0.70}^{3+}\text{Fe}_{0.40}^{2+})_{\Sigma 2.98}\text{Si}_{2.99}\text{O}_{12.63}(\text{OH})_{0.37}$. The simplified formula of åskagenite-(Nd) is $\text{Mn}^{2+}\text{NdAl}_2\text{Fe}^{3+}(\text{Si}_2\text{O}_7)(\text{SiO}_4)\text{O}_2$.

Gladstone-Dale compatibility is $1 - (K_p/K_c) = 0.012$ (superior) for initial (metamict) åskagenite-(Nd), with measured density.

Fig. 1. Crystal of åskagenite-(Nd) in pegmatite.

Fig. 2. IR spectra of initially metamict åskagenite-(Nd) (1), åskagenite-(Nd) heated at 600°C during 1 h in nitrogen (2), Nd-rich allanite-(Ce) from Khaldzan Buragtag, Mongolia (3) and Nd-rich allanite-(Y) from Åskagen, Sweden (4).



XANES, IR and Mössbauer data. The valency of Mn and Fe

In order to obtain IR spectra, mineral samples have been mixed with anhydrous KBr, pelletized, and analysed using a Specord 75 IR spectrophotometer. IR spectrum of pure KBr-disk was subtracted from the overall spectrum. Polystyrene and gaseous NH_3 were used as frequency standards; the precision of frequency measurement is $\pm 1\text{ cm}^{-1}$; the mean resolution for the region $400 - 1600\text{ cm}^{-1}$ is 0.8 cm^{-1} .

The IR spectrum of åskagenite-(Nd) (Figure 2, curve 1) contains two strong bands with maxima at 469 and 1000 cm^{-1} and several shoulders. The absence of distinct band maxima in the range $550 - 900\text{ cm}^{-1}$ is a generic property of the majority of metamict silicates. A very weak band at 1623 cm^{-1} indicates the presence of trace amounts of H_2O . The IR spectrum of åskagenite-(Nd) heated at 600°C , during 1 h in nitrogen (Figure 2, curve 2) is similar to the spectra of allanite group minerals (Figure 2, curves 3 and 4). A very weak band at 3280 cm^{-1} corresponds to stretching vibrations of OH groups.

Mössbauer spectrum has been obtained using the modified WISSEL spectrometer, with registration simultaneously in 512 channels and signal accumulation during 308 hours at the temperature of 293 K .

In the Mössbauer spectrum of åskagenite-(Nd) (Figure 3) two doublets are present. The doublet with an isomer shift of 0.2869 mm/s , quadrupole splitting of 0.9139 mm/s and relative area of 51.5% corresponds to Fe^{3+} . The doublet with isomer shift of 0.9402 mm/s , quadrupole splitting of 2.1830 mm/s and relative area of 48.5% corresponds to Fe^{2+} . Thus the $Fe^{3+}:Fe^{2+}$ ratio in åskagenite-(Nd) is $51.5:48.5$.

Fine structure of the X-ray absorption spectrum near edge structure (XANES) is very sensitive to the electronic state of an absorbing atom,

Table 1. Analytical data for åskagenite-(Nd)

Comp.	wt.%	Range	Probe standard
CaO	0.27	0.00–1.43	Wollastonite
Y_2O_3	2.27	2.02–2.82	Y
La_2O_3	0.44	0.40–0.49	$LaPO_4$
Ce_2O_3	7.99	7.83–8.15	$CePO_4$
Pr_2O_3	1.76	1.47–1.90	$PrPO_4$
Nd_2O_3	11.21	11.06–11.45	$NdPO_4$
Sm_2O_3	3.01	2.82–3.33	$SmPO_4$
Yb_2O_3	0.28	0.00–0.48	$YbPO_4$
ThO_2	0.72	0.58–0.77	ThO_2
MnO*	7.98	7.79–8.48	Mn
FeO**	7.75	15.85–16.13***	Fe
$Fe_2O_3^{**}$	9.16		
Al_2O_3	15.85	15.60–15.97	Al_2O_3
SiO_2	29.51	29.32–29.90	SiO_2
H_2O	0.55		
Total	98.75		

Note: * – According to XANES data, all Mn is bivalent.
 ** – Total iron content analysed as FeO is $16.00\text{ wt.}\%$ and divided over FeO and Fe_2O_3 according to the results of Mössbauer spectroscopy.

*** – For total iron measured as FeO.

as well as to its local environment. In order to determine the state of Mn in åskagenite-(Nd), its XANES spectrum was compared with X-ray spectra of standard samples.

XANES spectra were obtained on the X-ray beamline of the synchrotron radiation source ANKA (SUL-X Beamline) in the Synchrotron Radiation Laboratory for Environmental Studies, Karlsruhe Institute of Technology, Campus North. The samples have been prepared as pellets (with cellulose as matrix stuff) and as raw fragments of the mineral using fluorescence-mode and transmission-mode data-logging, in the range of energies from 6530 to 6630 eV , the

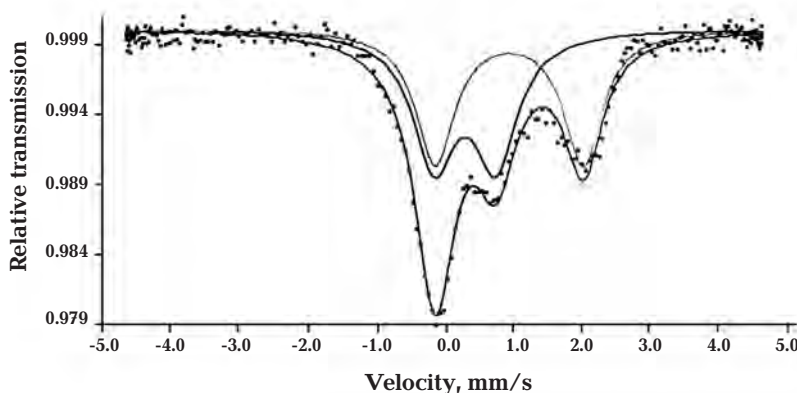


Fig. 3. Mössbauer spectrum of åskagenite-(Nd).

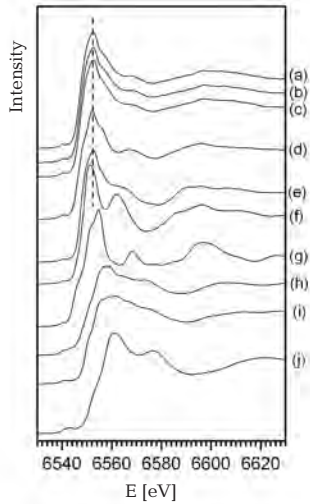


Fig. 4. Mn K-edge XANES spectra of åskagenite-(Nd) (a – c, three measurements), $Mn^{2+}SiO_3$ (d), $Mn^{2+}SO_4 \cdot H_2O$ (e), $Mn^{2+}CO_3$ (f), MnO (g), Mn_2O_3 (h), $Mn^{3+}PO_4$ (i) and MnO_2 (j).

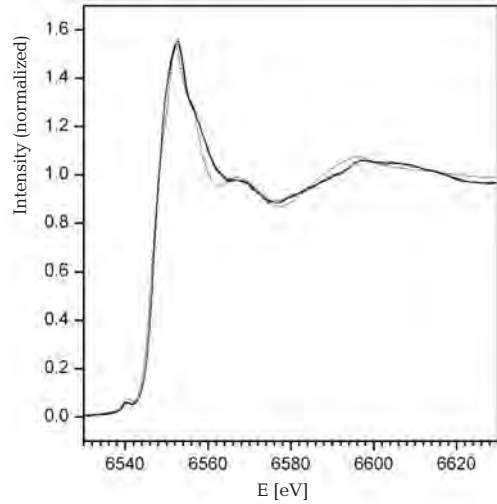


Fig. 5. Comparison of Mn K-edge XANES spectra of åskagenite-(Nd) (solid lines) and $Mn^{2+}SiO_3$ (dashed line).

Table 2. X-ray powder-diffraction data for åskagenite-(Nd) heated at 600°C during 1 hour in nitrogen

I_{meas} %	d_{meas} Å	d_{calc} Å	hkl
23	9.12	9.11	001
12	8.01	7.98	100
16	5.05	5.05	101
18	4.63	4.64	110
17	4.00	3.99	200
46	3.50	3.48	-211
50	3.22	3.23	-212
		3.20	201
100	2.897	2.900	-301
73	2.850	2.855	020
73	2.687	2.688	120
52	2.606	2.611	-303
38	2.534	2.526	202
23	2.394	2.393	-221
22	2.394	2.322	220
15	2.300	2.299	113
37	2.15*	2.145	-401
48	2.121	2.122	-403
27	2.038	2.042	203
		2.034	-321
25	1.979	1.983	104
35	1.876	1.873	114
46	1.644	1.646	-306
59	1.630	1.629	124
15	1.580	1.582	-316
14	1.546	1.548	330
29	1.456	1.457	304
		1.455	-334
33	1.404	1.405	-612, 140
		1.404	-141

Note: * – broad reflection.

beam sizes at the sample surface being $100 \times 200 \mu m$. In the working range of energies, other components except Mn do not absorb X-ray irradiation. The detectable contribution of the Nd L-series becomes apparent only above 6700 eV.

The Mn K-edge spectrum of åskagenite-(Nd) with the main resonance line at 6553 eV is very close to the spectrum of $Mn^{2+}SiO_3$ (Mn^{2+} in anhydrous silicate matrix), and little different from spectra of compounds with Mn^{2+} in other matrices. It is strongly different from spectra of compounds with Mn^{3+} and Mn^{4+} (Figures 4 and 5). Thus we conclude that all manganese in åskagenite-(Nd) is divalent.

X-ray diffraction

Åskagenite-(Nd) is metamict, amorphous. Its X-ray powder-diffraction pattern contains only a broad halo with the maximum near 3 Å.

After heating in a nitrogen atmosphere (heating rate 5°C per minute up to 600°C, then one hour at a constant temperature of 600°C, and thereafter with rapid cooling to room temperature) åskagenite-(Nd) gives a distinct X-ray powder diffraction pattern. X-ray powder diffraction data were collected using a PANalytical X'Pert PRO diffractometer with Soller slit and X'Cellerator detector. The data (in Å for $CuK\alpha_1$) are given in Table 2. Heated sample is monoclinic, space group $P2_1/m$; refined unit-cell parameters are: $a = 8.78(1)$ Å, $b = 5.710(6)$ Å, $c = 10.02(1)$ Å, $\beta = 114.6(2)^\circ$; $V = 456.7(8)$ Å³, $Z = 2$.

Table 3. Comparative data for åskagenite-(Nd) and some related epidote supergroup minerals (all monoclinic, $P2_1/m, Z = 2$)

Mineral	Åskagenite-(Nd)	Epidote-(Sr)	Epidote-(Pb)	Allanite-(Ce)	Allanite-(La)	Mangani- androsite-(Ce)	Mangani- androsite-(La)
Simplified formula	$Mn^{2+}NdAl_2Fe^{3+}(Si_2O_7)(SiO_4)O_2$	$CaSrAl_2Fe^{3+}(Si_2O_7)(SiO_4)O(OH)$	$CaPbAl_2Fe^{3+}(Si_2O_7)(SiO_4)O(OH)$	$CaCeAl_2Fe^{2+}(Si_2O_7)(SiO_4)O(OH)$	$CaLaAl_2Fe^{2+}(Si_2O_7)(SiO_4)O(OH)$	$Mn^{2+}CeMn^{3+}AlFe^{2+}(Si_2O_7)(SiO_4)O(OH)$	$Mn^{2+}LaMn^{3+}AlFe^{2+}(Si_2O_7)(SiO_4)O(OH)$
Unit cell data							
<i>a</i> , Å	8.78	8.928	8.958	8.932	8.914	8.901	8.896
<i>b</i> , Å	5.71	5.652	5.665	5.770	5.726	5.738	5.706
<i>c</i> , Å	10.02	10.244	10.304	10.1575	10.132	10.068	10.083
β , °	114.6	114.46	114.4	114.69	114.87	113.425	113.88
<i>V</i> , Å ³	456.7	470.5	476.2	475.6	469.1	471.81	468.0
Strongest reflections	3.50–46	3.50–42	3.49–50	3.53–43	3.506–20	3.514–41	3.504–40
of the X-ray powder pattern	2.897–100	2.92–100	2.81–100	2.886–28	2.901–100	2.896–100	2.897–100
<i>d</i>, Å – <i>I</i>, %	2.850–73	2.83–32	2.71–40	2.714–66	2.692–60	2.713–39	2.707–60
	2.687–73	2.72–41	2.60–50	2.627–41	2.611–50	2.707–39	2.615–60
	2.606–52	2.61–42	2.18–40	2.182–36	2.174–25	2.6225–53	2.178–60
	2.121–48	2.58–49	1.90–40	2.158–23			2.145–60
	1.630–59						
Density, g/cm³	3.737 (meas)*; 4.375 (calc)		4.03 (meas); 4.03 (calc)	3.5–4.2 (meas); 4.11 (calc)	3.93 (meas); 3.94 (calc)	4.21 (calc)	>4.03 (meas); 4.21 (calc)
Optical data:							
Refraction indices	Isotropic, mean refraction index is 1.712*	$\alpha = 1.737$ $\beta = 1.780$ $\gamma = 1.792$	$\alpha = 1.788$ $\beta = 1.81$ $\gamma = 1.830$	Mean 1.54–1.72* $\alpha = 1.69$ –1.79 $\beta = 1.70$ –1.815 $\gamma = 1.71$ –1.83	$\alpha = 1.755$ $\beta = 1.760$ $\gamma = 1.765$	Mean refraction index is 1.80 (calc)	Mean refraction index is 1.877 (calc)
Optical sign, 2V	No data	(-) 62	(-) 50	(-) or (+) 40–123	(+/-) 90	(+) 80.6	No data
Sources	This work	Minakawa <i>et al.</i> , 2008	Dollase, 1971; Dunn, 1985; Holtstam, Langhof, 1994	Vlasov, 1966; Dollase, 1971; Deer <i>et al.</i> , 1986	Orlandi, Pasero, 2006	Cenki-Tok <i>et al.</i> , 2006	Bonazzi <i>et al.</i> , (1996)

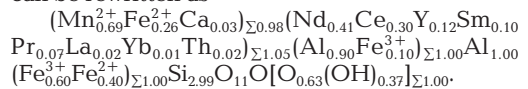
Note: * – Data for metamict samples.

Discussion

The distribution of ionic species between various sites is determined by ionic radii. In particular, Al^{3+} ($r = 0.535$ Å) preferentially occupies the M2 and M1 sites, whereas Fe^{3+} ($r = 0.645$ Å) present in amounts 1 apfu concentrates in the M3 site. In order to determine site population in an epidote-group mineral, one must know valencies of species-determining components.

Taking into account that the valencies of Fe and Mn in åskagenite-(Nd) have been determined by non-destructive methods and following the accepted rules for the assignment of ionic species to the various key sites (Armbruster *et al.*,

2006), the empirical formula of åskagenite-(Nd) can be rewritten as



In Table 3, comparative data for åskagenite-(Nd) and some related epidote group minerals are given. Åskagenite-(Nd) has the smallest unit cell volume among epidote group minerals, even smaller than those of manganiandrosite-(Ce) and manganiandrosite-(La), both of which contain Mn^{2+} in the A1 site. As compared with manganiandrosite-(Ce) and manganiandrosite-(La), in åskagenite-(Nd), Fe^{3+} prevails over the larger cation Fe^{2+} and Mn^{2+} is partially substituted by

the smaller cation Fe^{2+} . Probably, this is the main cause of the lowering of unit cell dimensions.

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