

Article

Fenitization at the Lovozero Alkaline Massif, NW Russia: Composition and Evolution of Fluids

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Abstract: The 360–370 Ma old Lovozero massif (NW Russia) is a layered nepheline syenitic-foidolitic pluton. Despite its huge size (650 km²), the massif is surrounded by a narrow fenite aureole, and the most intensive fenitization is associated with pegmatites and hydrothermal veins that have intruded into the wall rocks. We studied petrography, petrochemistry, mineralogy and fluid inclusions along a profile crossing the direct contact of the Lovozero massif with country Archean gneiss. We found that the fluid responsible for fenitization was a heterogeneous mixture of two coexisting phases, an aqueous fluid with salinity 8.6–15.1 eq. wt.% NaCl and a methane fluid. The coexistence of these two fluids indicates immiscibility conditions at (or below) CH₄–H₂O solvus. The aqueous fluid affected both the endocontact alkaline rocks and country gneiss. In the endocontact, intense autometasomatic alterations of the early crystallized minerals occurred, for example, the natrolitization of nepheline and sodalite. Besides, the aqueous fluid transported Na₂O, K₂O, as well as P₂O₅, TiO₂, H₂O, F, Cl and S into the exocontact. These components were precipitated in the immediate vicinity of the massif contact, and the salinity of the aqueous fluid decreased to 0.53–3.06 eq. wt.% NaCl. We assume that there are two reasons for a narrow fenite aureole in the Lovozero massif: intense autometasomatic alterations and a decrease in the permeability of country rocks due to fluid immiscibility.

Keywords: fenite; fluid inclusions; Lovozero massif; methane



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1. Introduction

Fenitization is the metasomatism of wall rocks under the influence of fluids released from crystallizing carbonatite or alkaline intrusions [1,2]. Fenites are named after the Fen complex (southern Norway), where these rocks were first described in 1921 by Brøgger [1]. Initially, fenites were defined as rocks of originally granitic composition that had been metasomatically altered towards an alkali-syenitic composition by solutions sourced from ijolite–melteigite magma. Now the term ‘fenite’ has a more general meaning and includes a wide spectrum of alkaline alteration products developed near alkaline and carbonatite intrusions. The mineralogical and textural diversities of fenites are determined by a large number of parameters, such as compositions of alkaline melt and fluid, protolith mineralogy, temperature, and pressure [3–8].

Fenites are of great economic importance because they often contain elevated concentrations of rare earth elements (REE) as well as high field strength elements (HFSE). The reason is that fluids exsolved from the crystallizing intrusions of alkaline rocks and carbonatites contain Cl[−], F[−], SO₄^{2−}, PO₄^{3−} or CO₃^{2−} anions, which can form complexes with REE and HFSE [9–12]. As a result, the solubility of these elements increases significantly, and they can be transported into fenite aureoles and precipitated there [7,13,14].

Generally, fenites associated with alkaline and carbonatite intrusions differ from each other in volume. Indeed, compared to carbonatites, alkaline intrusions produce comparatively narrow fenite aureoles. For example, fenitization extends 1–2 km from the

central Sokli (Finland) carbonatite intrusion [15], whereas fenitization surrounding large nepheline-syenite intrusions such as Khibiny (NW Russia) and Ilímaussaq (Greenland) does not typically exceed 100–120 m from the intrusion [16–19]. The reason for such differences is the high solubility of water in alkaline melts [20]. Aqueous fluid gradually accumulates during magmatic crystallization, and at the late- to post-magmatic stage, this fluid causes intense autometasomatic reactions. As a result of such reactions, secondary assemblages of minerals are formed at the expense of primary magmatic minerals [7]. Typical examples of autometasomatic reactions are the replacement of nepheline and sodalite by natrolite and gonnardite [21,22]. In other words, the main volume of fluids is spent on autometasomatic reactions occurring within the boundaries of the alkaline intrusion.

An excellent example of a case where a huge alkaline massif is surrounded by a surprisingly narrow fenite aureole is the Lovozero massif, Kola Peninsula, Russia. This massif was emplaced 360–370 Ma ago [23–25] into Archean gneiss and gneissose granite and has a size of 20×30 km at the top, and about 12×16 km at a 5 km depth [26]. According to Arzamastsev and colleagues [27], the calculated volume of the Lovozero massif is 1600 ± 250 km³. Depending on the initial water content in the melt, the volume of water fluid released during crystallization of the massif could reach 100 km³. Considering such a huge volume of fluid, one should assume the presence of a wide fenite aureole. However, fenitization in the Lovozero massif is associated mainly with alkaline pegmatites and hydrothermal veins that have intruded into the exocontact [17,28], while at the contact of alkaline rocks and country rocks, the width of the fenite aureole does not exceed several meters. Thus, a relatively wide fenitization aureoles are observed only in areas where pegmatites and hydrothermal veins are present.

In this article, we present the results of petrological, geochemical and fluid inclusion studies of the contact influence of alkaline rocks of the Lovozero massif on country Archean gneiss. In order to study the direct effect of the alkaline melt on the surrounding rocks, samples for this research were taken from an area where pegmatites and hydrothermal veins are absent. Based on the results of the fluid inclusions study, as well as the chemical compositions of minerals and rocks, we made conclusions about the sequence of metasomatic alterations of the country rocks and the evolution of the fenitizing fluids.

2. Geological Background and Previous Research

The Lovozero layered intrusion [26,29] is located on the Kola Peninsula, Russia (Figure 1) and covers an area of 650 km². The main rock types of the Lovozero massif are foid syenites and foidolites, which compose three main units (or complexes; Figure 1a–c). The most voluminous (77% of massif's volume), the so-called Layered complex, comprises of numerous subhorizontal layers (or rhythms) [26,29,30]. Each rhythm is a sequence of rocks: foidolite (urtite or ijolite; bottom of the rhythm)—leucocratic nepheline syenite (foyaite)—meso/melanocratic nepheline syenite (lujavrite). In some rhythms foidolite is absent and such rhythms consist only of foyaite (bottom of the rhythm) and lujavrite. The contact between underlying lujavrite and overlying urtite or foyaite is sharp. All rhythms of the Layered complex are grouped into seven series (I–VII from top to bottom). In each series, the urtite layers are additionally indicated by Arabian numerals. Figure 1b,c shows only some of the urtite horizons, namely I-4, II-4, II-7, III-1, III-10, III-14, IV-1, IV-2.

The second, so-called Eudialyte complex (18% of massif's volume; thickness varies from 0.1 to 0.8 km), overlies the Layered complex (Figure 1c) and consists of lujavrite enriched in eudialyte-group minerals (EGM), so-called eudialyte lujavrite. In addition to eudialyte lujavrite, this complex also contains lenses and sheet-like bodies of foyaite, as well as fine-grained/porphyritic nepheline syenites (Figure 1b).

The Poikilitic complex (5% of massif's volume) consists of leucocratic feldspathoid syenites, in which grains of feldspathoids are poikilitically incorporated into large crystals of alkali feldspar. These rocks form lenses, or irregularly shaped bodies, which are located in both the Layered and Eudialyte complexes. In the Poikilitic complex, the following two main groups of rocks are recognized: uneven-grained nepheline syenite and poikilitic

foid syenite. These rocks are connected by gradual transitions but differ in the content of poikilitic feldspar crystals. A large number of xenoliths of Devonian volcanoclastic rocks [31,32], both unaltered and intensely metasomatized, are found among the rocks of the Layered and the Eudialyte complexes.

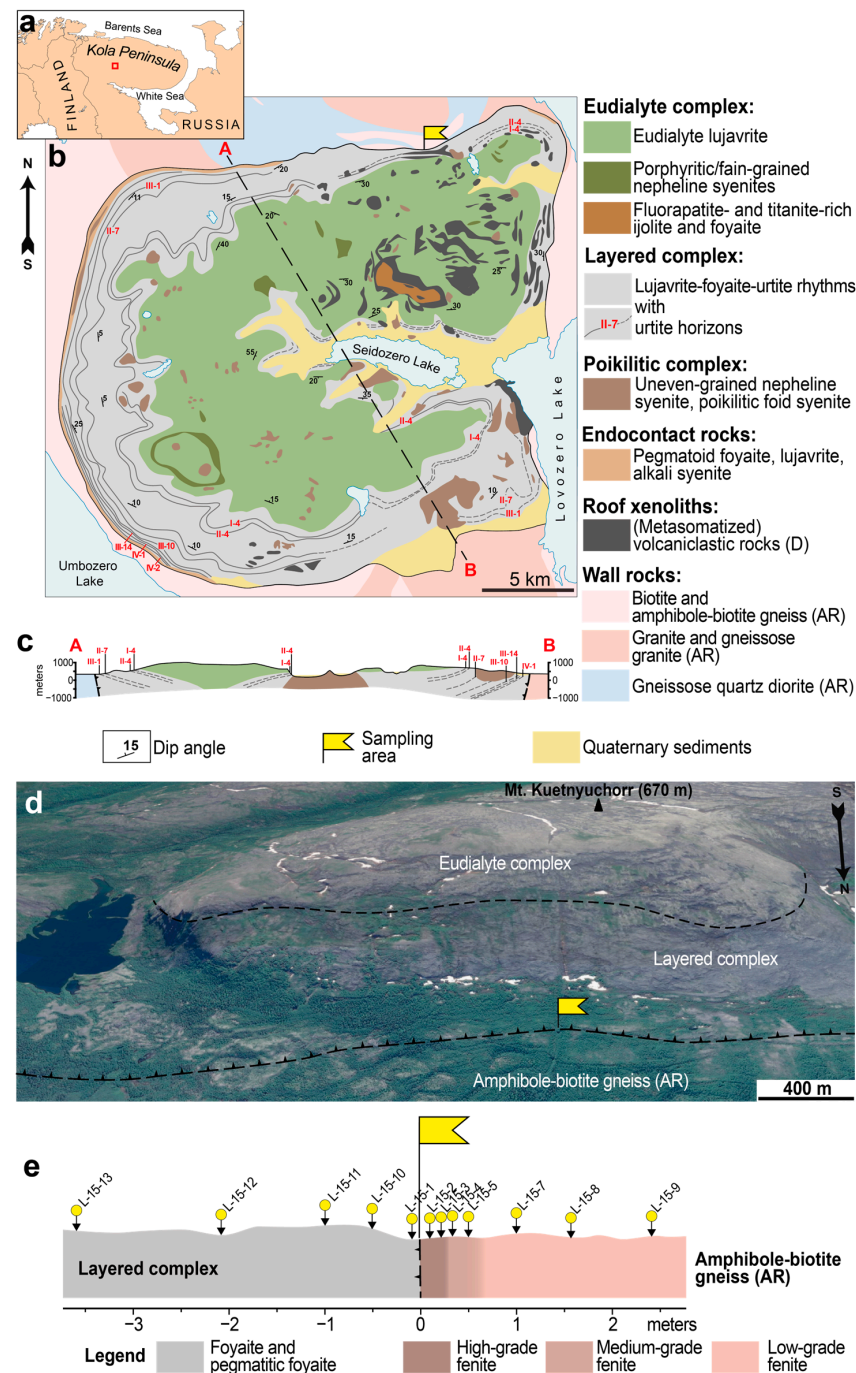


Figure 1. Geological background and sampling points. (a) Location of the Lovozero massif (red square); (b) geological scheme of the Lovozero alkaline massif and (c) cross-section along the line A–B (after [33] with simplifications); (d) satellite image of the northern slope of the Mt. Kuetsnyuchorr. The contact of the Eudialyte and Layered complexes (dashed line), the contact of the massif with country amphibole-biotite gneiss (dotted line with triangles), and the sampling area (yellow flag) are shown; (e) sampling scheme; yellow circles with arrows show sampling points (see also Table 1).

Table 1. List of studied samples and their brief description.

Sample	Distance from Contact, Meters	Brief Description	Zone
LV-15-13	−3.6	foyaite	Endocontact
LV-15-12	−2.1	foyaite	
LV-15-11	−1.0	pegmatoid foyaite	
LV-15-10	−0.5	pegmatoid foyaite	
LV-15-1	−0.1	foyaite	
LV-15-2	0.1	high-grade fenite	Exocontact
LV-15-3	0.2	high-grade fenite	
LV-15-4	0.3	medium-grade fenite	
LV-15-5	0.5	medium-grade fenite	
LV-15-7	1.0	low-grade fenite	
LV-15-8	1.6	low-grade fenite	
LV-15-9	2.4	low-grade fenite	

The earliest studies of fenites of the Lovozero massif were carried out in the 60s of the last century [34,35]. The following features of the contact zone have been recognized: (1) the fenite aureole at the direct contact between alkaline rocks and Archean gneiss is very narrow; (2) pegmatites and hydrothermal veins surrounded by wide fenitization halos are widespread in the exocontact; (3) pegmatitic foyaite with rare-metal mineralization is widespread in the endocontact. Later, the contact interactions of the Lovozero massif with country rocks were studied by Arzamastsev and colleagues [17,28]. These researchers found that fenitization was associated predominantly with hydrothermal veins and occurred 359 ± 5 Ma ago, i.e., after crystallization of the bulk of the massif. According to Arzamastsev [17], metasomatic alterations of gneiss occurred under the influence of an essentially aqueous fluid that also transported Nb, Ta, Zr, Hf, and REE in complexes with F^- , Cl^- , and SO_4^{2-} to the exocontact zone. In endocontact up to 200 m wide, nepheline syenites have a fine-grained or, conversely, pegmatitic texture and are enriched in calcium-bearing minerals, such as diopside, katophorite, Sr-rich apatite, titanite, pyrochlore-group minerals, and rinkite.

3. Materials and Methods

For this study, 12 rock samples were collected along a profile crossing the contact of the Lovozero massif with surrounding amphibole-biotite gneiss. This cross-section is located at the foot of Mt. Kuetyuchorr in the north of the massif (Figure 1b,d). The sampling scheme is shown in Figure 1e, and a list of samples studied samples and their brief description is shown in Table 1. Fenites were classified based on the intensity of textural or mineralogical change in accordance with the qualitative ‘grade’ classification of V. Morogan [36]:

- (1) *low-grade fenites* (textures and mineralogy inherited from the protolith largely unaltered);
- (2) *medium-grade fenites* (relicts rare, new rock textures develop from fluid injection and by replacement reactions or recrystallization);
- (3) *high-grade fenites* (protolith mineralogy almost fully altered).

Endocontact alkaline rocks are leucocratic nepheline syenites that differ in grain size, so we subdivided these rocks into medium- to coarse-grained foyaite (hereinafter, we refer to this rock as foyaite) and pegmatoid foyaite (Table 1).

The 12 doubly polished thin sections were prepared for fluid inclusion investigations and microprobe analyses. Back-scattered electron (BSE) images were obtained and minerals were diagnosed at the Geological Institute of the Kola Science Center of the Russian Academy of Sciences (GI KSC RAS, Apatity, Russia) using a scanning electron microscope LEO-1450 (Carl Zeiss Microscopy, Oberkochen, Germany) with the energy-dispersive system AZtec Ultimmax 100 (Oxford Instruments, Oxford, UK). The chemical composition of minerals was analyzed with the Cameca MS-46 electron microprobe (Cameca, Gennevilliers, France) operating in the WDS-mode at 22 kV with a beam diameter of 10 μ m, a beam current of 20–40 nA, and counting times of 10 s (for a peak) and 10 s (for background

before and after the peak), with 5–10 counts for every element in each point. Standards are shown in Table A1. The analytical precision (reproducibility) of mineral analyses was 0.2–0.05 wt.% (2 standard deviations) for the major element and approximately 0.01 wt.% for impurities. The systematic errors were within the random errors.

Major elements in rocks (12 samples) were determined by wet chemical analysis at the GI KSC RAS. The accuracy limits for SiO₂, TiO₂, ZrO₂, Fe₂O₃, Al₂O₃, CaO, SrO, MgO, MnO, Na₂O, K₂O, P₂O₅, REE₂O₃, S_{tot}, F, Cl, H₂O are 0.01 wt.%, and for FeO is 0.1 wt.%.

The Raman spectra were recorded with a Horiba Jobin–Yvon LabRAM HR800 spectrometer equipped with an Olympus BX-41 microscope in backscattering geometry (Saint-Petersburg State University). The 514.5 nm Ar + laser line was used for spectra excitation. The spectra were obtained in the range of 100–4200 cm^{−1} at a resolution of 1 cm^{−1} at room temperature. All spectra were processed using the algorithms implemented in the OriginPro 8.1 software package (Originlab Corporation, Northampton, MA, USA).

Heating and freezing experiments with fluid inclusions were carried out using a Linkam THMSG-600 stage with a measuring range from −196 to +600 °C. Cooling was carried out by supplying a stream of liquid nitrogen. The measurement errors of freezing and heating temperature are ±0.1 and ±1 °C, respectively. Temperature changes were controlled using the Linksys 32 software module. Visual control of phase transformations in inclusions was carried out on an Olympus BX53 microscope (Olympus Corp., Tokyo, Japan) equipped with an Olympus 50× long-focus objective and an Olympus EP50 digital camera.

Mineral abbreviations (Table 2) are given in accordance with International Mineralogical Association (IMA)-approved mineral symbols [37].

Table 2. Abbreviations, names and formulae of minerals mentioned in this article.

Abbreviation	Mineral	Formula ¹
Ab	albite	Na(AlSi ₃ O ₈)
Act	actinolite	□Ca ₂ (Mg _{4.5–2.5} Fe ²⁺ _{0.5–2.5})Si ₈ O ₂₂ (OH) ₂
Aeg	aegirine	NaFe ³⁺ Si ₂ O ₆
Aeg-Aug	aegirine-augite	(Ca,Na)(Fe ³⁺ ,Mg,Fe ²⁺)Si ₂ O ₆
Blmp	barytolamprophyllite	(BaK)Ti ₂ Na ₃ Ti(Si ₂ O ₇) ₂ O ₂ (OH) ₂
Di	diopside	CaMgSi ₂ O ₆
EGM	eudialyte-group minerals	N ₁₅ M ₁₆ M ₂₃ M ₃ M ₄ Z ₃ [Si ₂₄ O ₇₃]O' ₄ X ₂ ; where N = Na, Ca, K, Sr, REE, Ba, Mn, H ₃ O ⁺ ; M ₁ = Ca, Mn, REE, Na, Sr, Fe; M ₂ = Fe, Mn, Na, Zr, Ta, Ti, K, Ba, H ₃ O ⁺ ; M _{3,4} = Si, Nb, Ti, W, Na; Z = Zr, Ti, Nb; O' = O, OH [−] , H ₂ O; X = H ₂ O, Cl [−] , F [−] , OH [−] , CO ₃ ^{2−} , SO ₄ ^{2−} ; [38,39]
Fap	fluorapatite	Ca ₅ (PO ₄) ₃ F
Gon	gonnardite	(Na,Ca) ₂ (Si,Al) ₅ O ₁₀ ·3H ₂ O
Hap	hydroxylapatite	Ca ₅ (PO ₄) ₃ OH
Ilm	ilmenite	Fe ²⁺ Ti ⁴⁺ O ₃
Kfs	K-feldspar	K(AlSi ₃ O ₈)
LGM	lovozerite-group minerals	A ₃ B ₃ C ₂ MSi ₆ O ₁₂ O _{6x} (OH) _x ·nH ₂ O; where M = Zr, Ti, Fe ³⁺ , Ca; C = Ca, Mn ²⁺ , Na, □ (vacancy); A = Na, Ca; B = Na, □; 0 ≤ x ≤ 6; n = 0–1; [40]
Marf	magnesio-arfvedsonite	NaNa ₂ (Mg ₄ Fe ³⁺)Si ₈ O ₂₂ (OH) ₂
Nph	nepheline	Na ₃ K(Al ₄ Si ₄ O ₁₆)
Ntr	natrolite	Na ₂ (Si ₃ Al ₂)O ₁₀ ·2H ₂ O
Olig	oligoclase	(Na,Ca)[Al(Si,Al)Si ₂ O ₈]
Phl	phlogopite	KMg ₃ (AlSi ₃ O ₁₀)(OH) ₂
Qz	quartz	SiO ₂
Rct	richterite	Na(NaCa)Mg ₅ Si ₈ O ₂₂ (OH) ₂
Ttn	titanite	CaTi(SiO ₄)O

¹ Formulae are given in accordance with IMA (International Mineralogical Association) list of minerals, with the exception of oligoclase, eudialyte-group minerals, lovozerite-group minerals.

4. Results

4.1. Petrography and Rock Chemistry

Low-grade fenite at a distance of 1–2.4 m from the massif contact consists of oligoclase (up to 1 mm across), quartz (up to 0.8 mm across), phlogopite, and actinolite (Figure 2a,b). Accessory minerals are fluor- and hydroxylapatite, ilmenite, monazite-(Ce), zircon and thorite. Albite replaces quartz and oligoclase and forms thin rims around their grains. At the contact between albite and quartz, there are small (up to 20 μm across) K-feldspar grains (Figure 2b).

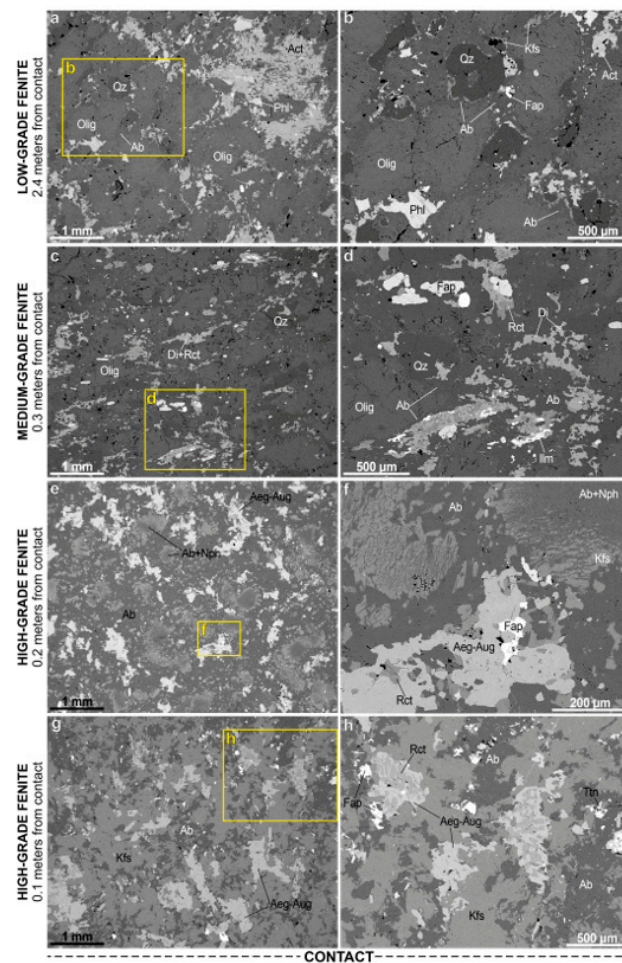


Figure 2. Mineral associations of fenites. (a) Low-grade fenite (sample L-15-9); (b) detailed fragment of Figure 2a; (c) medium-grade fenite (sample L-15-4); (d) detailed fragment of Figure 2c; (e) high-grade fenite (sample L-15-3); (f) detailed fragment of Figure 2e; (g) high-grade fenite (sample L-15-2); (h) detailed fragment of Figure 2g. BSE images. Please see Table 2 for abbreviations.

In medium-grade fenite at a distance of 0.3–0.5 m from the massif contact, phlogopite is absent, and albite intensely replaces quartz and oligoclase (Figure 2c,d). Mafic rock-forming minerals are represented by diopside and richterite in close intergrowths (Figure 2d). Accessory minerals are fluorapatite, ilmenite, monazite-(Ce), and zircon.

In high-grade fenite at a distance of 0.2 m from the massif contact quartz and oligoclase are absent. The main rock-forming minerals are albite (60–65% of the rock volume), potassium feldspar, aegirine-augite, and richterite (Figure 2e,f). Albite forms close intergrowths with K-feldspar, as well as intergrowths with nepheline, surrounded by a K-feldspar rims (Figure 2f). Accessory minerals are fluorapatite, ilmenite, titanite and zircon.

At a distance of 0.1 m from the massif contact, the potassium feldspar is the predominant (70–75 vol.%) mineral in high-grade fenite (Figure 2g,h). Rock-forming minerals are

also albite, nepheline, aegirine-augite, richterite, and accessory minerals are fluorapatite, titanite, ilmenite, lamprophyllite, sodalite, and pyrrhotite.

Foyaite at a distance of 0.1 m from the contact (Figure 3a,b) consists of euhedral to subhedral nepheline and microcline-perthite, needle-like crystals of aegirine-(augite), and anhedral magnesio-arfvedsonite. Accessory minerals are lamprophyllite and barytolamprophyllite, fluorapatite, loparite-(Ce), eudialyte-group minerals, and rhabdophane-(Ce). Nepheline and microcline are intensively replaced by albite and natrolite, and eudialyte-group minerals are pseudomorphized by lovozerite-group minerals.

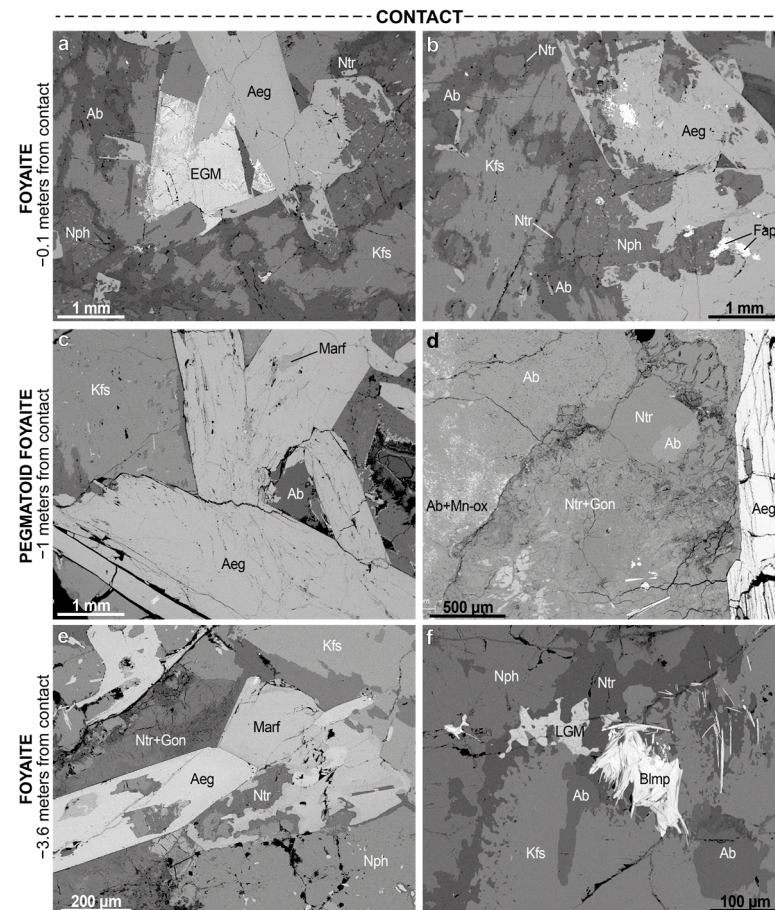


Figure 3. Mineral associations of foyaite and pegmatoid foyaite. (a,b) Foyaite (sample L-15-1). Note the intense alteration of K-feldspar and nepheline by natrolite and albite; (c) pegmatoid foyaite (sample L-15-11); (d) alteration of potassium feldspar by natrolite, albite, gonnardite in pegmatoid foyaite (sample L-15-11); (e) foyaite (sample L-15-13); (f) alteration of potassium feldspar and nepheline by natrolite and albite in foyaite (sample L-15-13). BSE images. See Table 2 for abbreviations.

The main rock-forming minerals of pegmatoid foyaite (Figure 3c,d) are microcline-perthite and aegirine-augite. Microcline-perthite forms are euhedral to subhedral laths (up to 5 cm in length) and is intensively replaced by natrolite, gonnardite, and albite. Aegirine-augite forms both large (up to 4 cm in length) prismatic and small needle-like crystals. Magnesio-arfvedsonite usually forms intergrowths with aegirine-augite.

Foyaite at a distance of 2.1–3.6 m from the contact, like one near the contact, is composed of nepheline, microcline-perthite, aegirine-augite, and magnesio-arfvedsonite (Figure 3e,f). Accessory minerals are lamprophyllite and barytolamprophyllite, fluorapatite, loparite-(Ce), eudialyte-group minerals, and labuntsovite-group minerals. Nepheline and microcline are intensively replaced by albite, natrolite, and gonnardite.

The chemical compositions of foyaites and fenites are presented in Supplementary Table S1, and Figure 4 shows the changes in the content of the main components in the above-mentioned rocks along the studied cross-section.

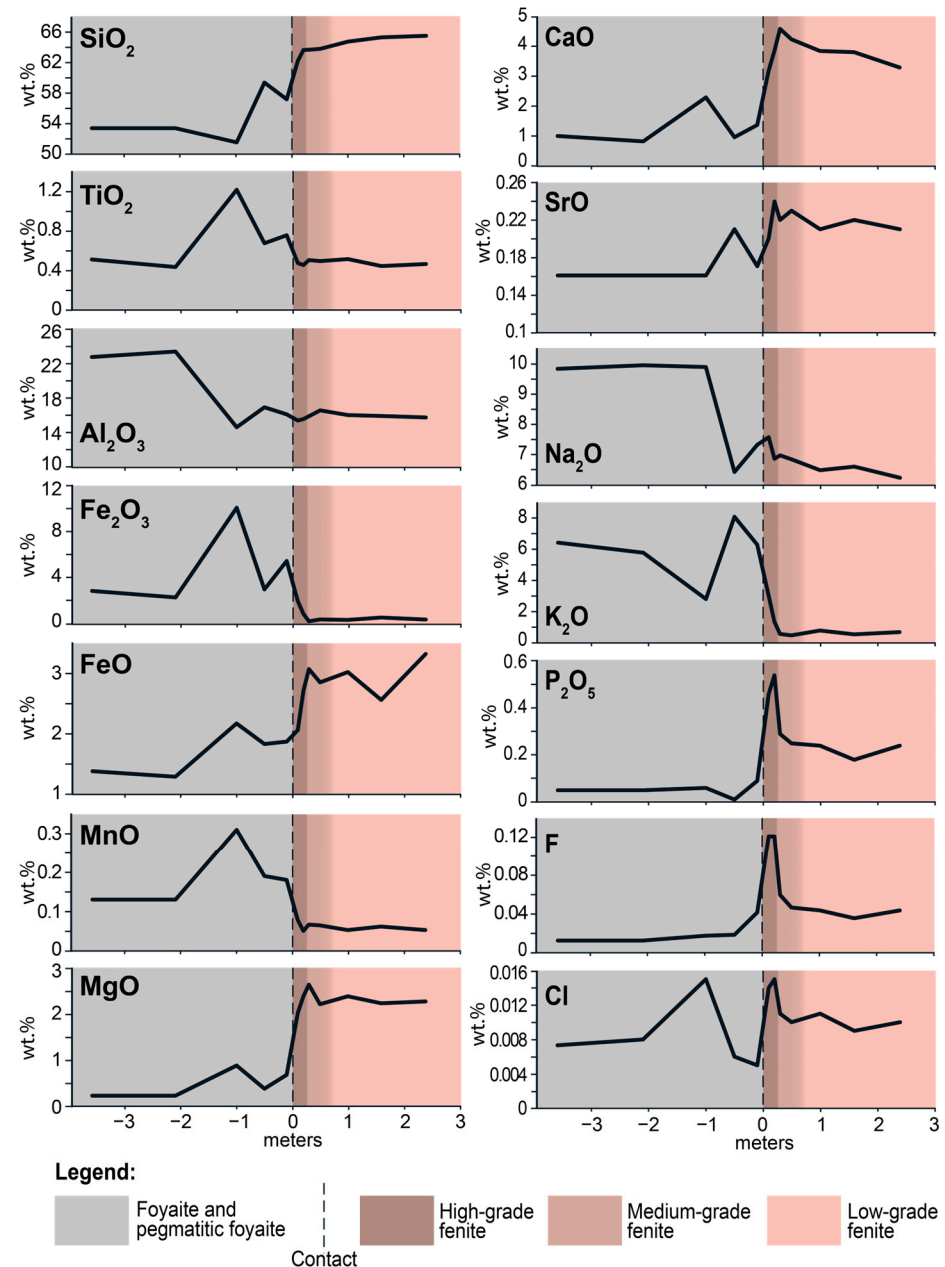


Figure 4. Changes in the contents of the main rock components along the studied cross section.

4.2. Mineral Chemistry

Feldspars are the main rock-forming minerals of all studied samples. Representative chemical compositions of oligoclase, albite, and K-feldspar from foyaites and fenites are shown in Table 3. Oligoclase $\text{Ab}_{73-76}\text{An}_{23-27}\text{Or}_{0-2}$ was found only in low- and medium-grade fenites, while in high-grade fenite it was completely replaced by albite and K-feldspar. Albite from foyaite, pegmatoid foyaite and fenites do not contain calcium, while in low- and medium-grade fenites the calcium content in albite can reach 0.06 apfu (Table 3). Feldspars, with the exception of oligoclase, are characterized by a small admixture of ferric iron (up to 0.02 Fe^{3+} pfu). Barium admixture (up to 0.15 wt.% BaO) is typical of K-feldspar from foyaites.

Table 3. Representative compositions of feldspars.

Sample	L-15-10		L-15-1		L-15-2		L-15-3		L-15-4		L-15-8		L-15-9	
Rock	Pegmatoid Foyaite		Foyaite		High-Grade Fenite		High-Grade Fenite		Medium-Grade Fenite		Low-Grade Fenite		Low-Grade Fenite	
Mineral	Ab	Kfs	Ab	Kfs	Ab	Kfs	Ab	Kfs	Olig	Ab	Olig	Ab	Olig	Ab
SiO ₂	68.29	64.74	66.93	63.54	67.46	63.98	68.96	65.83	61.70	67.37	62.65	68.42	62.33	68.02
Al ₂ O ₃	18.79	18.05	18.51	17.31	18.59	17.45	18.83	17.37	23.80	19.96	23.78	20.56	23.87	19.86
Fe ₂ O ₃	0.08	Bdl	0.19	0.24	0.23	0.06	0.42	0.58	Bdl	0.09	Bdl	0.11	Bdl	0.19
CaO	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl	4.92	0.88	5.20	1.34	5.47	0.82
Na ₂ O	10.86	0.27	11.39	0.36	10.66	0.37	11.17	0.85	8.10	10.88	7.86	10.08	8.39	10.94
K ₂ O	0.14	15.99	0.10	16.04	0.12	16.10	0.14	15.32	0.13	0.14	0.29	0.23	0.29	0.27
BaO	Bdl	0.12	Bdl	0.10	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl
Total	98.16	99.16	97.12	97.58	97.05	97.95	99.52	99.95	98.65	99.31	99.79	100.74	100.35	100.11
Formulae based on O = 8 pfu														
Si	3.03	3.01	3.01	3.02	3.02	3.02	3.02	3.03	2.76	2.97	2.77	2.96	2.75	2.97
Al	0.98	0.99	0.98	0.97	0.98	0.97	0.97	0.94	1.26	1.04	1.24	1.05	1.24	1.02
Fe ³⁺	-	-	0.01	0.01	0.01	-	0.01	0.02	-	-	-	-	-	0.01
Ca	-	-	-	-	-	-	-	-	0.24	0.04	0.25	0.06	0.26	0.04
Na	0.93	0.02	0.99	0.03	0.93	0.03	0.95	0.08	0.70	0.93	0.67	0.85	0.72	0.93
K	0.01	0.95	0.01	0.97	0.01	0.97	0.01	0.90	0.01	0.01	0.02	0.01	0.02	0.01
Total	4.95	4.98	5.00	5.00	4.95	5.00	4.96	4.97	4.96	4.98	4.95	4.94	4.99	4.98
Mol. % end members														
An	-	-	-	-	-	-	-	-	25	5	26	7	26	3
Ab	99	3	99	3	99	4	98	8	74	95	72	92	72	95
Or	1	97	-	96	-	96	-	90	1	-	2	1	2	1
For	-	-	1	1	1	-	1	2	-	-	-	-	-	1
Fab	-	-	-	-	-	-	1	-	-	-	-	-	-	-

Bdl—below detection limit; An—anorthite; Ab—albite; Or—orthoclase; Olig—oligoclase; For—ferriorthoclase (KFe³⁺Si₃O₈); Fab—ferrialbite (NaFe³⁺Si₃O₈).

Phlogopite is found only in low-grade fenite in association with quartz, oligoclase, and actinolite. Table 4 shows a representative composition of phlogopite. Typical impurities are titanium (up to 0.31 Ti pfu), barium (up to 0.01 Ba pfu), vanadium (up to 0.01 V pfu) and manganese (up to 0.01 Mn pfu). Phlogopite also contains up to 0.05 Cl per formula unit.

Table 4. Representative compositions of phlogopite.

Sample	L-15-7	L-15-7	L-15-8	L-15-8	L-15-9	L-15-9
SiO ₂	37.65	39.71	39.17	38.22	37.54	37.97
TiO ₂	4.45	4.91	5.17	5.31	4.92	4.89
Al ₂ O ₃	12.32	13.86	13.16	13.85	12.54	12.05
V ₂ O ₃	Bdl	0.25	0.18	0.15	Bdl	Bdl
FeO	13.96	16.06	16.60	17.65	20.53	20.24
MnO	0.10	0.13	0.09	Bdl	0.11	0.14
MgO	15.12	14.06	13.18	12.10	10.19	12.21
BaO	0.18	0.39	0.27	Bdl	0.30	0.18
Na ₂ O	Bdl	Bdl	0.14	Bdl	Bdl	0.08
K ₂ O	9.61	8.60	9.67	8.36	8.02	9.62
Cl	0.10	0.12	0.15	0.19	0.32	0.35
O=Cl	−0.02	−0.03	−0.03	−0.04	−0.07	−0.08
Total	93.47	98.03	97.74	95.79	94.40	97.64
Formulae based on 7 cations and 22 anions						
Si	2.93	2.95	2.97	2.93	2.97	2.92
Ti	0.26	0.27	0.30	0.31	0.29	0.28
Al	1.13	1.21	1.18	1.25	1.17	1.09
V	-	0.01	0.01	0.01	-	-
Fe ²⁺	0.91	1.00	1.05	1.13	1.36	1.30
Mn	0.01	0.01	0.01	-	0.01	0.01
Mg	1.76	1.56	1.49	1.38	1.20	1.40
Ba	0.01	0.01	0.01	-	0.01	0.01

Table 4. Cont.

Sample	L-15-7	L-15-7	L-15-8	L-15-8	L-15-9	L-15-9
Na	-	-	0.02	-	-	0.01
K	0.96	0.81	0.94	0.82	0.81	0.94
Cl	0.01	0.02	0.02	0.02	0.04	0.05

Bdl—Below detection limit.

Amphiboles are rock-forming minerals of foyaites and fenites, but the composition of amphiboles regularly changes. Low-grade fenite contains Ca-amphibole (actinolite), medium-grade fenite contains Na-Ca-amphibole (richterite), high-grade fenite are characterized by richterite and Na-amphibole (magnesio-arfvedsonite), and only magnesio-arfvedsonite is found in foyaites (Table 5). Thus, in the sequence low-grade fenite → medium-grade fenite → high-grade fenite → foyaites, the sodium content consistently increases (Figure 5a). In addition, amphiboles from low- and medium-grade fenite lack fluorine, while amphiboles from high-grade fenite and foyaites contain up to 0.72 F pfu (Figure 5b).

Table 5. Representative compositions of amphiboles.

Sample	L-15-13	L-15-11	L-15-1	L-15-2	L-15-2	L-15-3	L-15-5	L-15-7	L-15-9
Rock	Foyaite	Pegmatoid Foyaite	Foyaite	High-Grade Fenite			Medium-Grade Fenite	Low-Grade Fenite	
Mineral	Marf	Marf	Marf	Marf	Rct	Rct	Rct	Act	Act
SiO ₂	54.29	54.80	52.72	54.09	54.90	56.44	53.53	53.50	52.73
TiO ₂	0.72	0.62	1.78	0.61	0.23	0.50	1.31	0.67	0.84
Al ₂ O ₃	0.65	0.87	1.50	0.37	0.49	0.06	1.04	1.79	2.18
V ₂ O ₃	Bdl	Bdl	0.09	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl
MnO	2.70	2.49	1.86	0.21	0.43	0.38	0.19	0.18	0.25
FeO	17.56	16.94	20.73	15.01	13.78	7.35	14.41	9.33	15.76
MgO	9.09	9.61	7.36	11.18	13.24	18.17	13.00	18.06	13.14
CaO	0.40	1.03	1.14	1.01	3.73	5.48	7.99	10.63	11.23
Na ₂ O	8.89	9.05	9.43	8.99	7.59	7.28	5.44	2.25	0.84
K ₂ O	2.33	2.22	1.40	1.91	1.73	1.39	0.53	0.41	0.33
F	0.90	1.10	1.01	1.30	1.11	1.60	Bdl	Bdl	Bdl
Cl	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl	0.02	0.08
O=F,Cl	−0.38	−0.46	−0.43	−0.55	−0.47	−0.67	0.00	−0.01	−0.02
Total	97.15	98.28	98.59	94.13	96.76	97.96	97.44	96.83	97.36
Formulae based on O + OH + F = 24 apfu and OH = 2 − 2Ti									
Si (T)	8.14	8.09	7.88	8.22	8.12	8.08	7.91	7.68	7.73
Al (T)	-	-	0.12	-	-	-	0.09	0.30	0.27
Ti (T)	-	-	-	-	-	-	-	0.02	-
Total T	8.14	8.09	8.00	8.22	8.12	8.08	8.00	8.00	8.00
Ti (C)	0.08	0.07	0.20	0.07	0.03	0.05	0.15	0.06	0.09
Al (C)	0.11	0.15	0.14	0.07	0.09	0.01	0.09	-	0.11
V (C)	-	-	0.01	-	-	-	-	-	-
Fe ³⁺ (C)	0.64	0.68	0.97	0.52	0.18	-	-	0.21	0.25
Mn (C)	0.34	0.31	0.24	0.03	0.05	0.05	0.02	-	-
Fe ²⁺ (C)	1.56	1.42	1.62	1.39	1.52	0.88	1.78	0.87	1.67
Mg (C)	2.03	2.12	1.64	2.53	2.92	3.88	2.86	3.86	2.87
Total C	4.77	4.74	4.82	4.60	4.79	4.87	4.91	5.00	5.00
Mn (B)	-	-	-	-	-	-	-	0.02	0.03
Fe ²⁺ (B)	-	-	-	-	-	-	-	0.04	0.01
Ca (B)	0.07	0.16	0.18	0.16	0.59	0.84	1.27	1.64	1.77
Na (B)	1.94	1.84	1.82	1.84	1.41	1.16	0.74	0.31	0.20
Total B	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
Na (A)	0.65	0.75	0.92	0.81	0.77	0.86	0.82	0.32	0.04
K (A)	0.45	0.42	0.27	0.37	0.33	0.25	0.10	0.08	0.06
Total A	1.09	1.17	1.18	1.18	1.09	1.11	0.92	0.40	0.10
OH	1.41	1.35	1.12	1.24	1.43	1.17	1.71	1.89	1.80
F	0.43	0.51	0.48	0.63	0.52	0.72	-	-	-
Cl	-	-	-	-	-	-	-	0.01	0.02
O	0.16	0.14	0.40	0.14	0.05	0.11	0.29	0.11	0.18
Total W	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00

Bdl—Below detection limit; T, C, B, A, W—sites in general formula AB₂C₅T₈O₂₂W₂ [41]; See Table 2 for abbreviations.

The composition of *clinopyroxenes*, like amphiboles, regularly changes in the sequence medium-grade fenite → high-grade fenite → foyaite. Diopside is found in low- and medium-grade fenite, aegirine-augite is typical of high-grade fenite, and both aegirine-augite and aegirine are found in foyaite. Representative compositions of clinopyroxenes are presented in Table 6, and Figure 5c shows the ratios of sodium, magnesium and ferrous iron in the composition of all studied clinopyroxenes.

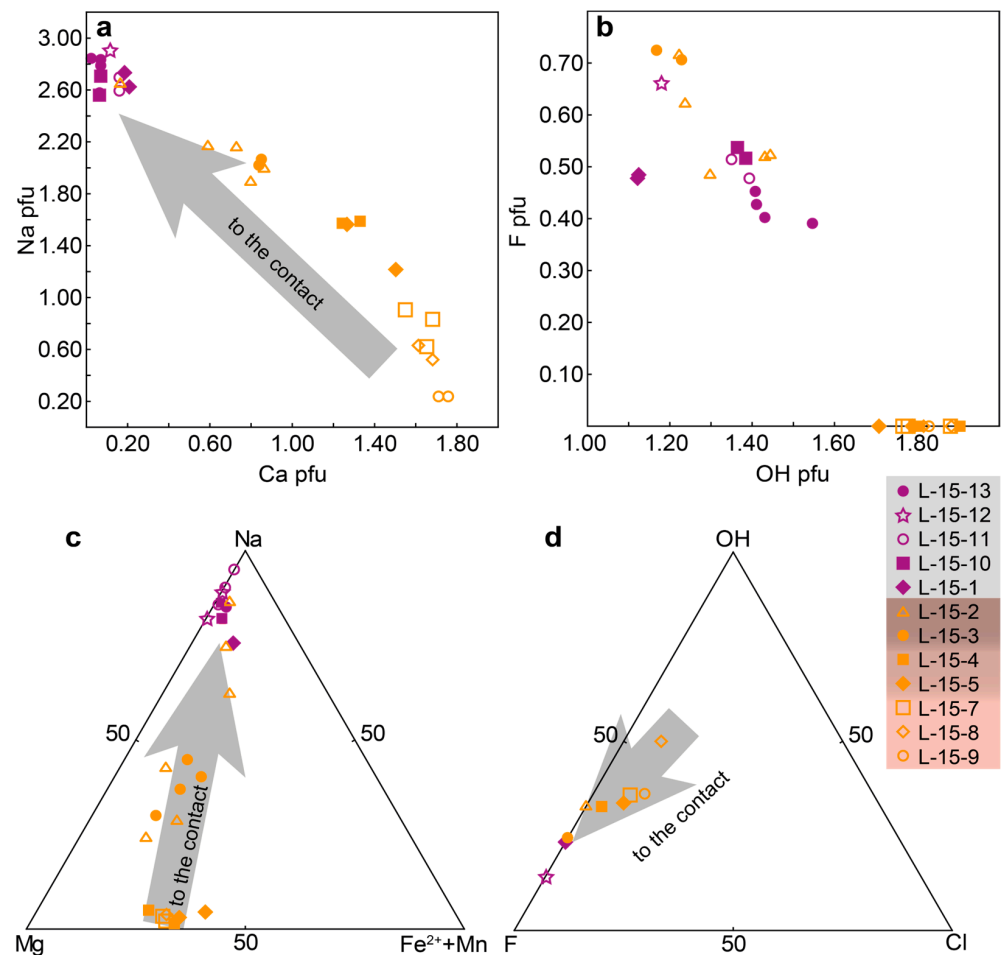


Figure 5. Compositions of rock-forming and accessory minerals. (a) Compositional variation of amphiboles in the Na–Ca system; (b) compositional variation of amphiboles in the F–OH system; (c) compositional variation of clinopyroxenes in the Mg–(Fe²⁺ + Mn)–Na system; (d) compositional variation of apatite-group minerals in the F–Cl–OH system.

Apatite-group minerals are the typical accessory of all studied samples. Apatite-group minerals from foyaite and fenites contain a high content of strontium (up to 0.75 Sr pfu in foyaite and up to 0.24 Sr pfu in high-grade fenite), while in low- and medium-grade fenites, the content of strontium does not exceed 0.01 Sr pfu (Table 7). Relatively high content of rare earth elements was found only in apatite-group minerals from foyaite, while in fenites the content of REE does not exceed 0.05 apfu. In the sequence low-grade fenite → medium-grade fenite → high-grade fenite → foyaite, the fluorine content in apatite-group minerals gradually increases (Table 7 and Figure 5d).

Nepheline is a rock-forming mineral of foyaite; it is also found in high-grade fenite, where it forms close intergrowths with albite, surrounded by a rim of K-feldspar (Figure 2e,f). The chemical composition of nepheline is presented in Table 8. The composition of nepheline from foyaite and high-grade fenite is identical, an admixture of ferric iron is typical (up to 0.02 Fe³⁺ pfu).

Table 6. Representative compositions of clinopyroxenes.

Sample	L-15-12	L-15-11	L-15-10	L-15-2	L-15-3	L-15-4	L-15-5	L-15-7	L-15-8
Rock	Foyaite	Pegmatoid Foyaite	High-Grade Fenite		Medium-Grade Fenite		Low-Grade Fenite		
Mineral	Aeg-Aug	Aeg-Aug	Aeg-Aug	Aeg-Aug	Aeg-Aug	Di	Di	Di	Di
SiO ₂	52.53	53.16	51.26	51.92	52.92	53.30	53.15	53.07	53.91
TiO ₂	1.79	1.70	1.66	1.85	0.89	0.07	Bdl	0.10	0.11
ZrO ₂	0.56	0.62	0.56	0.97	0.08	0.14	Bdl	Bdl	Bdl
Al ₂ O ₃	0.83	0.75	0.93	0.80	0.23	0.33	Bdl	0.22	0.21
FeO	23.50	23.86	26.24	20.54	16.72	9.37	13.10	10.62	10.76
MnO	0.50	0.48	0.52	0.72	0.16	0.36	0.39	0.41	0.38
MgO	3.12	2.32	2.48	4.04	7.08	13.39	10.70	12.95	12.32
CaO	5.63	4.87	5.44	9.55	14.87	21.51	21.15	21.65	22.00
Na ₂ O	11.18	11.71	10.78	8.50	6.00	0.73	0.65	0.34	0.33
Total	99.64	99.46	99.87	98.89	98.93	99.20	99.14	99.36	100.01
Formulae based on 4 cations and O = 6 pfu									
Si	1.95	1.98	1.92	1.97	1.99	2.00	2.04	2.01	2.03
Ti	0.05	0.05	0.05	0.05	0.03	-	-	-	-
Zr	0.01	0.01	0.01	0.02	-	-	-	-	-
Al	0.04	0.03	0.04	0.04	0.01	0.01	-	0.01	0.01
Fe ³⁺	0.73	0.73	0.78	0.50	0.39	0.02	-	-	-
Fe ²⁺	-	0.01	0.04	0.15	0.14	0.27	0.42	0.34	0.34
Mn	0.02	0.02	0.02	0.02	-	0.01	0.01	0.01	0.01
Mg	0.17	0.13	0.14	0.23	0.40	0.75	0.61	0.73	0.69
Ca	0.22	0.19	0.22	0.39	0.60	0.87	0.87	0.88	0.89
Na	0.81	0.85	0.78	0.63	0.44	0.05	0.05	0.02	0.02

Bdl—below detection limit; See Table 2 for abbreviations.

Table 7. Representative compositions of apatite-group minerals.

Sample	L-15-12	L-15-1	L-15-2	L-15-3	L-15-4	L-15-5	L-15-7	L-15-8	L-15-9
Rock	Foyaite	High-Grade Fenite		Medium-Grade Fenite		Low-Grade Fenite			
Mineral	Fap	Fap	Fap	Fap	Fap	Fap	Fap	Fap	Hap
P ₂ O ₅	38.05	37.57	41.29	41.31	41.62	41.02	41.67	41.91	41.16
SiO ₂	0.26	1.26	0.34	0.24	0.23	0.23	0.21	0.28	0.31
Y ₂ O ₃	Bdl	Bdl	Bdl	Bdl	0.07	Bdl	Bdl	Bdl	Bdl
La ₂ O ₃	1.96	2.05	0.23	0.30	Bdl	0.39	0.30	0.34	0.43
Ce ₂ O ₃	3.65	3.88	0.42	0.65	0.07	0.65	0.65	0.72	0.81
Pr ₂ O ₃	0.48	0.62	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl	Bdl
Nd ₂ O ₃	1.27	1.39	0.17	0.22	Bdl	0.21	0.20	0.16	0.35
CaO	36.90	38.79	50.05	51.03	53.81	52.81	53.12	53.06	52.74
SrO	13.40	9.83	4.72	2.92	0.11	0.13	0.17	0.16	Bdl
Na ₂ O	1.15	1.14	0.16	0.20	0.08	0.46	0.32	0.31	Bdl
Cl	Bdl	Bdl	Bdl	Bdl	0.23	0.52	0.58	0.61	0.74
F	2.80	2.50	2.40	2.70	2.30	2.00	2.00	1.50	1.90
O=F ₂ Cl	−1.18	−1.05	−1.01	−1.14	−1.02	−0.96	−0.97	−0.77	−0.97
Total	98.74	97.96	98.78	98.42	97.50	95.46	98.24	98.29	97.47
Formulae based on 13 total anions									
P	3.12	3.04	3.09	3.10	3.09	3.00	3.09	3.07	3.08
Si	0.03	0.12	0.03	0.02	0.02	0.02	0.02	0.02	0.03
La	0.07	0.07	0.01	0.01	-	0.01	0.01	0.01	0.01
Ce	0.13	0.14	0.01	0.02	-	0.02	0.02	0.02	0.03
Pr	0.02	0.02	-	-	-	-	-	-	-
Nd	0.04	0.05	0.01	0.01	-	0.01	0.01	0.01	0.01
Ca	3.83	3.97	4.75	4.85	5.05	4.89	4.98	4.93	4.99
Sr	0.75	0.54	0.24	0.15	0.01	0.01	0.01	0.01	-
Na	0.22	0.21	0.03	0.03	0.01	0.08	0.05	0.05	-
Cl	-	-	-	-	0.03	0.08	0.09	0.09	0.11
F	0.86	0.76	0.67	0.76	0.64	0.56	0.55	0.41	0.53
OH	0.14	0.24	0.33	0.24	0.33	0.36	0.36	0.50	0.36

Bdl—below detection limit; See Table 2 for abbreviations.

Table 8. Representative compositions of nepheline.

Sample	L-15-13	L-15-12	L-15-1	L-15-3	Sample	L-15-13	L-15-12	L-15-1	L-15-3
Rock	Foyaite			High-Grade Fenite	Rock	Foyaite			High-Grade Fenite
SiO ₂	41.45	43.30	42.09	42.59	Formulae based on O = 16 pfu				
Al ₂ O ₃	32.60	32.60	32.70	32.61	Si	4.13	4.22	4.16	4.18
Fe ₂ O ₃	0.17	0.30	0.10	0.14	Al	3.82	3.75	3.81	3.77
Na ₂ O	16.01	15.85	15.83	16.40	Fe ³⁺	0.01	0.02	0.01	0.01
K ₂ O	7.08	6.49	6.88	6.61	Na	3.09	3.00	3.03	3.12
Total	97.31	98.53	97.60	98.35	K	0.90	0.81	0.87	0.83

4.3. Fluid Inclusions

4.3.1. Fluid Inclusions in Quartz: Morphology and Classification

Numerous secondary inclusion trails were found in quartz from low- and medium-grade fenites (Figure 6a). Both intergranular trails forming in healed microcracks cross-cutting grain boundaries and intragranular trails occurring within single grains and not cross-cut grain boundaries were found. The size of the inclusions varies from 1 to 15 µm across. The shapes of the inclusions are round, oval or irregular, some of the inclusions are in the form of negative crystals (Figure 6b).

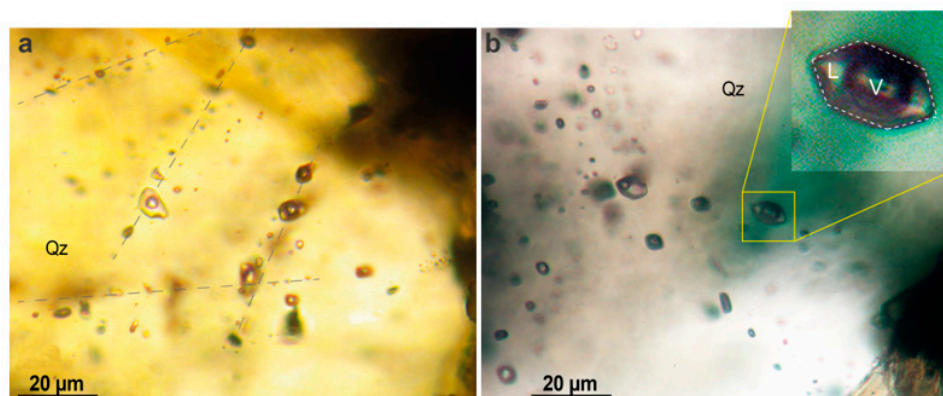


Figure 6. Secondary fluid inclusions in quartz from fenites. (a) intersecting secondary inclusion trails (marked by dotted lines); Dotted lines are positioned at the end of the trail, as seen under the microscope by changing focus level; (b) morphology of inclusions; small rounded inclusions and inclusions having the form of negative crystals are shown. An enlarged photograph of one of the inclusions is shown at the top right. The white dotted lines show the faces of the negative crystal. L—liquid; V—vapor. Samples L-15-5 (a) and L-15-7 (b). Photos in transmitted light.

The classification of fluid inclusion types observed in this study is based on phase proportions at room temperature. Based on the petrographic criteria, the following three types of inclusions were distinguished in this study: LV (liquid-rich 2-phase; liquid > vapor; Figure 7a,b), VL (vapor-rich 2-phase; vapor > liquid; Figure 7c), and V (mono-phase vapor; Figure 7d). Since the shapes of the inclusions are often irregular, liquid–vapor ratios were determined from area ratios of these phases in the photo in transmitted light. According to data from Raman spectroscopy, the vapor phase is usually represented by methane (CH₄). LV-type inclusions, in addition to methane, very rarely also contain carbon dioxide (CO₂) (Figure 7b), while only methane was found in VL- and V-type inclusions (Figure 7c,d).

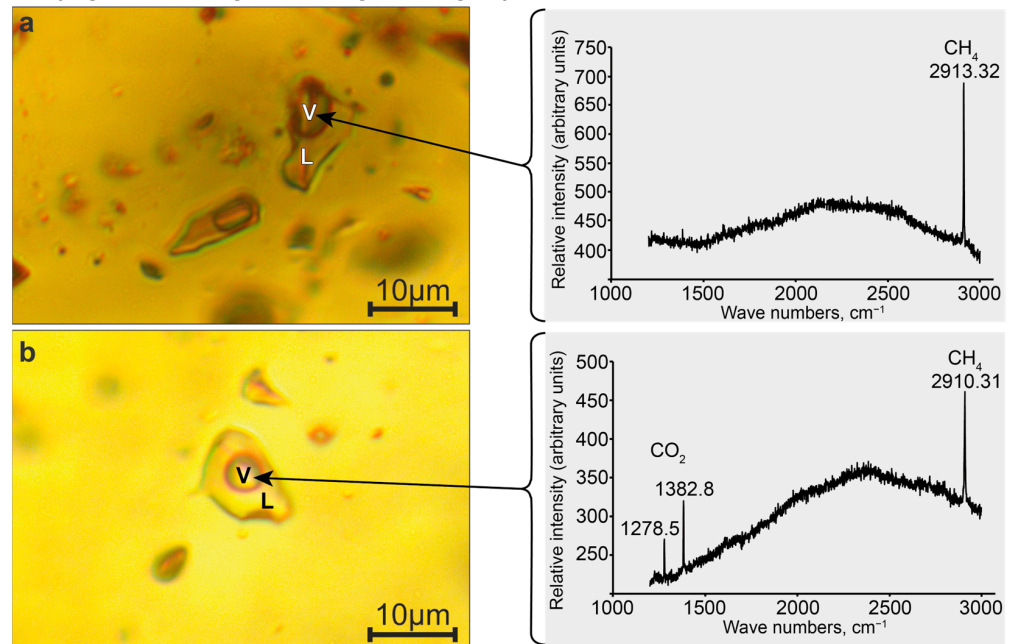
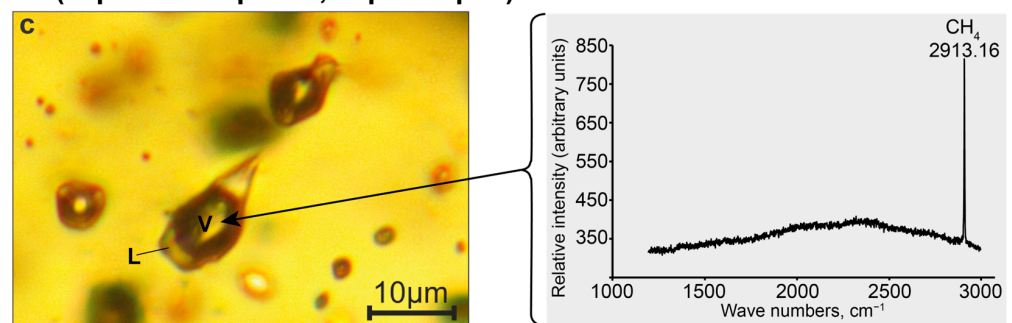
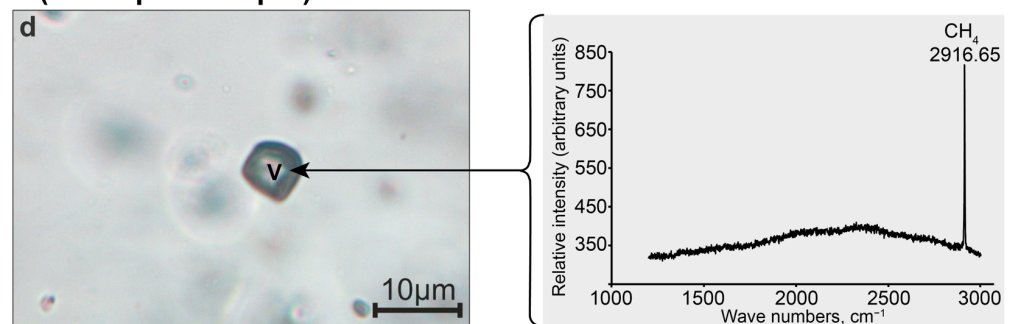
LV (liquid-rich 2-phase; liquid>vapor) inclusions**VL (vapor-rich 2-phase; vapor>liquid) inclusions****V (mono-phase vapor) inclusions**

Figure 7. Three types of fluid inclusions in quartz from fenites. (a) LV-type inclusions (left) and the Raman spectrum of the vapor phase in one of these inclusions (right); V = CH₄; sample L-15-5; (b) LV-type inclusions (left) and the Raman spectrum of the vapor phase in one of these inclusions (right); V = CH₄ + CO₂; sample L-15-5; (c) VL-type inclusions (left) and the Raman spectrum of the vapor phase in one of these inclusions (right); V = CH₄; sample L-15-5; (d) V-type inclusion (left) and the Raman spectrum of the vapor phase in this inclusion (right); V = CH₄; sample L-15-8. L—liquid; V—vapor. Photos in transmitted light.

It is important to note that inclusions of all three types occur along the same healed microfractures. Figure 8 shows two examples of such inclusions, located very close to each other, but having different vapor–liquid ratios.

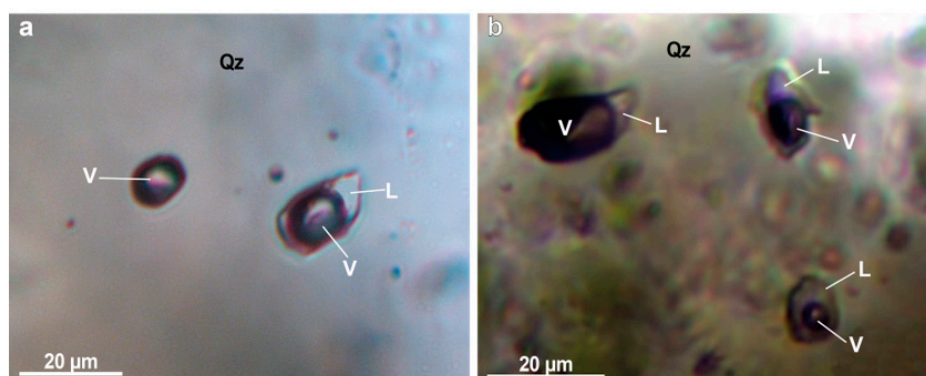


Figure 8. Fluid inclusions in quartz with different vapor-liquid ratios. (a) V-type (left) and VL-type (right) inclusions located along the same healed microfractures; (b) VL-type (top) and LV-type (bottom) inclusions located along the same healed microfractures. Sample L-15-8. L—liquid; V—vapor. Photos in transmitted light.

4.3.2. Fluid Inclusions in Quartz: Methane Densities

According to previous Raman studies [42,43], the peak position of the C–H symmetric stretching band (ν_1) of methane shifts systematically to lower wavenumbers with increasing density at constant temperature. At a given density, the peak shifts to lower wavenumbers with decreasing sample temperature. An equation

$$\rho = p_{00} + p_{10} \times T + p_{01} \times \nu_1 + p_{20} \times T^2 + p_{11} \times T \times \nu_1 + p_{02} \times \nu_1^2$$

(where ν_1 is the Raman peak shift in cm^{-1} , T is the temperature in $^{\circ}\text{C}$, ρ is the density of CH_4 in g/cm^3 , and p_{00} , p_{10} , p_{01} , p_{20} , p_{11} , p_{02} are fitting coefficients) was proposed by Shang and colleagues [42] to describe the relationship among CH_4 ν_1 peak position, density, and temperature. This equation was used to calculate the density of methane in a fluid inclusion from measured CH_4 ν_1 peak position at a fixed sample temperature. In this study, the density of methane was determined in inclusions containing only methane in the vapor phase. The results are presented in Table 9.

Table 9. Methane density calculation results.

Rock	Sample	Inclusion Type	CH_4 ν_1	CH_4 Density, g/cm^3
Medium-grade fenite	L-15-4	V	2914.66	0.137
		V	2914.63	0.138
		V	2914.20	0.156
	L-15-5	V	2914.79	0.131
		VL (see Figure 7c)	2913.16	0.203
		LV (see Figure 7a)	2913.32	0.196
		LV	2914.06	0.163
		LV	2912.36	0.242
Low-grade fenite	L-15-7	V	2917.30	0.040
		V	2917.46	0.031
		VL	2916.81	0.053
	L-15-8	V	2917.11	0.043
		V (see Figure 7d)	2916.65	0.060
		V	2916.76	0.055
		V	2916.72	0.056
		VL	2917.29	0.036
		LV	2917.13	0.042
	L-15-9	VL	2916.34	0.070
		VL	2916.41	0.068
		LV	2916.25	0.073

Fitting parameters: $p_{00} = 13,260.2997734257$; $p_{10} = 0.159909924698094$; $p_{01} = -9.0579957183101$; $p_{20} = -8.23392651807174 \times 10^{-8}$; $p_{11} = -5.47854804748698 \times 10^{-5}$; $p_{02} = 0.00154684331421227$ [42]; $T = 22$ $^{\circ}\text{C}$; L—liquid; V—vapor.

4.3.3. Fluid Inclusions in Quartz: Heating Experiments

Heating experiments were performed to determine the approximate homogenization temperature (T_h) of LV-type and VL-type inclusions. Before conducting heating experiments, the liquid:vapor (L:V) ratio in the inclusions was determined. It has been established that LV-type inclusions are homogenized into liquid ($L + V \rightarrow L$), while VL-type inclusions are homogenized into vapor ($L + V \rightarrow V$). Inclusions with approximately equal contents of liquid and vapor were homogenized into the liquid phase. Homogenization temperatures are shown in Table 10. The homogenization temperature of VL-type inclusions (386–410 °C) is higher than that of LV-type (135–375 °C).

Table 10. Results of experiments on heating and freezing of fluid inclusions in quartz.

Rock	Sample	Liquid:Vapor (L:V) Ratio	Type of Homogenization	T_h , °C	$T_{m(ice)}$, °C	Salinity, wt.% NaCleq
Medium-grade fenite	L-15-4	70:30	$L + V \rightarrow L$	275	−0.5	0.88
		80:20	$L + V \rightarrow L$	231	−1.2	2.07
		85:15	$L + V \rightarrow L$	170		
		30:70	$L + V \rightarrow V$	386		
	L-15-5	50:50	$L + V \rightarrow L$	341		
		20:80	$L + V \rightarrow V$	410		
Low-grade fenite	L-15-7	90:10	$L + V \rightarrow L$	150	−0.3	0.53
		80:20	$L + V \rightarrow L$	182	−1.0	1.74
		60:40	$L + V \rightarrow L$	330		
		60:40	$L + V \rightarrow L$	355		
		50:50	$L + V \rightarrow L$	355		
	L-15-8	50:50	$L + V \rightarrow L$	356	−1.8	3.06
		80:20	$L + V \rightarrow L$	285	−1.5	2.57
		90:10	$L + V \rightarrow L$	135		
		60:40	$L + V \rightarrow L$	375		
		30:70	$L + V \rightarrow V$	403		
		90:10	$L + V \rightarrow L$	169		
		90:10	$L + V \rightarrow L$	177		
		90:10	$L + V \rightarrow L$	135		
	L-15-9	80:20	$L + V \rightarrow L$	280	−1.1	1.91
		60:40	$L + V \rightarrow L$	345	−1.6	2.74
		20:80	$L + V \rightarrow V$	389		

T_h —homogenization temperature; $T_{m(ice)}$ —temperature of the melting point of ice; L—liquid; V—vapor.

4.3.4. Fluid Inclusions in Quartz: Freezing Experiments

Freezing experiments were performed to determine the composition of the liquid in inclusions as well as to determine the salinity of this liquid. Freezing was carried out mainly for liquid-rich LV-type inclusions.

The composition of the liquid can be determined by measuring the temperature of the eutectic, i.e., the temperature of the first melting of the ice. The eutectic temperature was determined for 10 liquid-rich LV-type inclusions. Our measurements showed that the eutectic temperature varies from −20.0 °C to −22.4 °C. The eutectic temperatures for the systems H_2O -NaCl and H_2O -KCl are −21.2 °C and −10.7 °C, respectively, whereas the eutectic for the ternary H_2O -NaCl-KCl is −22.9 °C [44]. Owing to the difficulty in recognizing the first melting during the heating of frozen inclusions, it is unlikely to distinguish between inclusions that begin to melt at −21.2° and those that start to melt at −22.9 °C. Thus, we assume that the composition of the liquid phase of the studied inclusions is H_2O -NaCl-(KCl).

To determine salinity (wt.% NaCl equivalent) the temperatures of the melting point of ice ($T_{m(ice)}$) were recorded (Table 10). The collected data were converted into corresponding

salinity values by using an equation and table for determining the freezing point depression of H₂O–NaCl solutions from the work of R. J. Bodnar [45].

4.3.5. Fluid Inclusions in Nepheline

There are two types of fluid inclusions in nepheline from foyaites: V (mono-phase vapor; Figure 9a) and LV (liquid-rich 2-phase; Figure 9b). It is secondary, trail-bound inclusions, which are disposed along the planar surface of healed microfractures. Mono-phase vapor (V-type) inclusions predominate, while LV-type inclusions are very rare and are found in the same trails as V-type inclusions. V-type inclusions usually have a rounded shape, their size does not exceed 5 µm. LV-type inclusions have a round or oval shape, and their size reaches 15 µm. According to the results of Raman spectroscopy, V-type inclusions consist of methane with a density of 0.089–0.099 g/cm³ (see Section 4.3.2 for the density calculation method). The vapor phase in LV-type inclusions is also represented by methane with a density of 0.08–0.1 g/cm³. The salinity of LV-type inclusions is 8.6–15.1 eq. wt.% NaCl {determined by the melting point of ice ($T_{m(ice)}$)}.

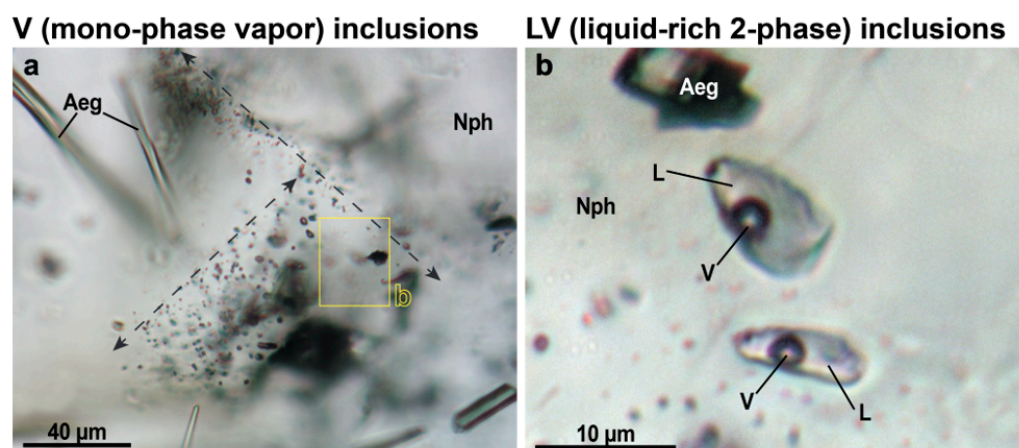


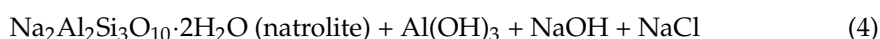
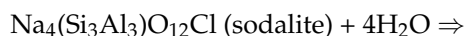
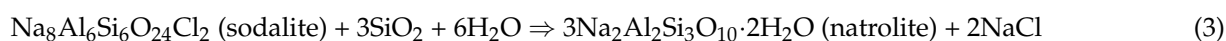
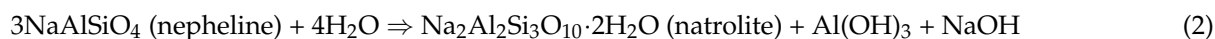
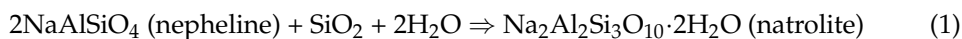
Figure 9. Secondary inclusions in nepheline from foyaites. (a) Intersecting secondary inclusions trails (marked by dotted lines with arrows; these lines are positioned at the end of the trail, as seen under the microscope by changing focus level); (b) morphology of LV-type inclusions; L—liquid; V—vapor. Sample L-15-12. Photos in transmitted light. Please see Table 2 for mineral abbreviations.

5. Discussion

Our studies have shown that nepheline from endocontact foyaites contains secondary fluid inclusions of two types: mono-phase vapor (V = CH₄) and liquid-rich two-phase (LV; L = H₂O; V = CH₄). The salinity of LV-type inclusions is 8.6–15.1 eq. wt.% NaCl, and methane density in V- and LV-type inclusions varies from 0.09 to 0.10 g/cm³. It is important to note that mono-phase vapor (V-type) inclusions absolutely predominate, while LV-type inclusions are very rare and are found along the same healed microfractures as V-type inclusions (Figure 9). Such inclusions are typical of nepheline (and also for K-feldspar, sodalite and eudialyte-group minerals) from the rocks of the internal parts of the Lovozero massif and were previously studied in detail by J. Potter and colleagues [46–48]. J. Potter in work [46] stressed that the rarity of H₂O-rich two-phase inclusions and the coexistence of these inclusions in the same trails as the mono-phase vapor (CH₄) inclusions implies coeval, immiscible trapping of aqueous and methane fluids, at or below the CH₄–H₂O solvus at ~350 °C.

We assume that the reason for the predominance of mono-phase methane inclusions and the rarity of H₂O-rich two-phase inclusions in nepheline is that aqueous fluid is intensively consumed in autometasomatic mineral reactions. Apparently, these reactions occurred before (or simultaneously) with the entrap of fluid inclusions. In the Lovozero massif, post-magmatic autometasomatic alterations of previously crystallized magmatic

minerals are extremely widespread [21,49,50]. For example, rock-forming feldspathoids such as nepheline and sodalite are intensively replaced by zeolites (mainly natrolite) in accordance with the following water-consumed reactions [22,51–53]:



The autometamorphic reactions are in places so intense that urtite is almost completely transformed into natrolite rock [50]. In the studied foyaites, intensive metasomatic alterations are also observed, including natrolitization of nepheline (Figure 3a,b,e,f). Thus, if immiscible aqueous fluid and methane were present in the rock at the post-magmatic stage, then the bulk of the aqueous fluid was consumed in reactions similar to (1)–(4), while methane remained in the fluid. For this reason, methane inclusions predominate in nepheline, while H₂O-rich two-phase inclusions are very rare. Intense autometamorphic reactions are also responsible for the extremely narrow fenite aureole that we observed in this study.

Quartz from low- and medium-grade fenites contains numerous secondary inclusions with very different ratios of vapor and liquid phases: LV (liquid-rich 2-phase; liquid > vapor), VL (vapor-rich 2-phase; vapor > liquid), and V (mono-phase vapor). The coexistence of LV-, VL- and V-type inclusions along the same healed microfractures (Figure 8) provides strong evidence that the fluids were entrapped under immiscible conditions at or below the CH₄–H₂O solvus. In fact, secondary inclusions in quartz have the following characteristics of heterogeneously trapped inclusions: (1) variable phase ratios at room temperature, ranging from gas-dominated to water-dominated compositions and (2) the gas-dominated inclusions homogenize towards the vapor-phase, and the water-dominated inclusions homogenize to liquid phase [54,55].

Thus, the phase composition and mechanism of formation of inclusions in exocontact quartz and inclusions in endocontact nepheline are similar. However, the salinity of liquid-bearing (LV and VL) inclusions in quartz is an order of magnitude lower than the salinity of LV inclusions in nepheline. Such low salinity is the result of the precipitation of hydrothermal solution components in the process of fenitization.

To identify the components added and lost during fenitization of the amphibole-biotite gneiss, we used the method proposed by J. Grant [56,57]. According to this method, the equation for composition–volume relations in metasomatic alteration was written as

$$C_i^A = M^O/M^A(C_i^O + \Delta C_i),$$

where, C_i is the concentration of component “i”.

“O” and “A”—the original and altered rocks, respectively.

M^O —the equivalent mass before alteration.

M^A —the equivalent mass after alteration.

ΔC_i —the change in the concentration of component “i”.

For each component, there is an equation of this form in which M^O/M^A is constant. If it is possible to identify immobile components, for which $\Delta C_i = 0$, M^O/M^A can be obtained by solving the set of simultaneous equations of the form $C_i^A = (M^O/M^A)C_i^O$. This procedure can be executed graphically by plotting the analytical data, in which case the immobile components define a straight line (isocon) through the origin. The isocon

equation is $C^A = (M^O/M^A)C^O$. The slope of the isocon yields the overall change in mass relative to M^O .

Figure 10 is an isocon diagram comparing the chemical composition of the low-grade fenite (sample L-15-9) and high-grade fenite (sample L-15-2). The slope of the isocon was determined from the a priori assumption that Al_2O_3 was immobile. Conclusions that can be drawn from this isocon diagram are that K_2O , Na_2O , P_2O_5 , TiO_2 , H_2O , F , Cl , and S were added in the process of fenitization, while SiO_2 was removed. In addition, iron was oxidized during fenitization. These data are consistent with the results obtained by A. Arzamastsev in the study of fenites associated with hydrothermal veins [17].

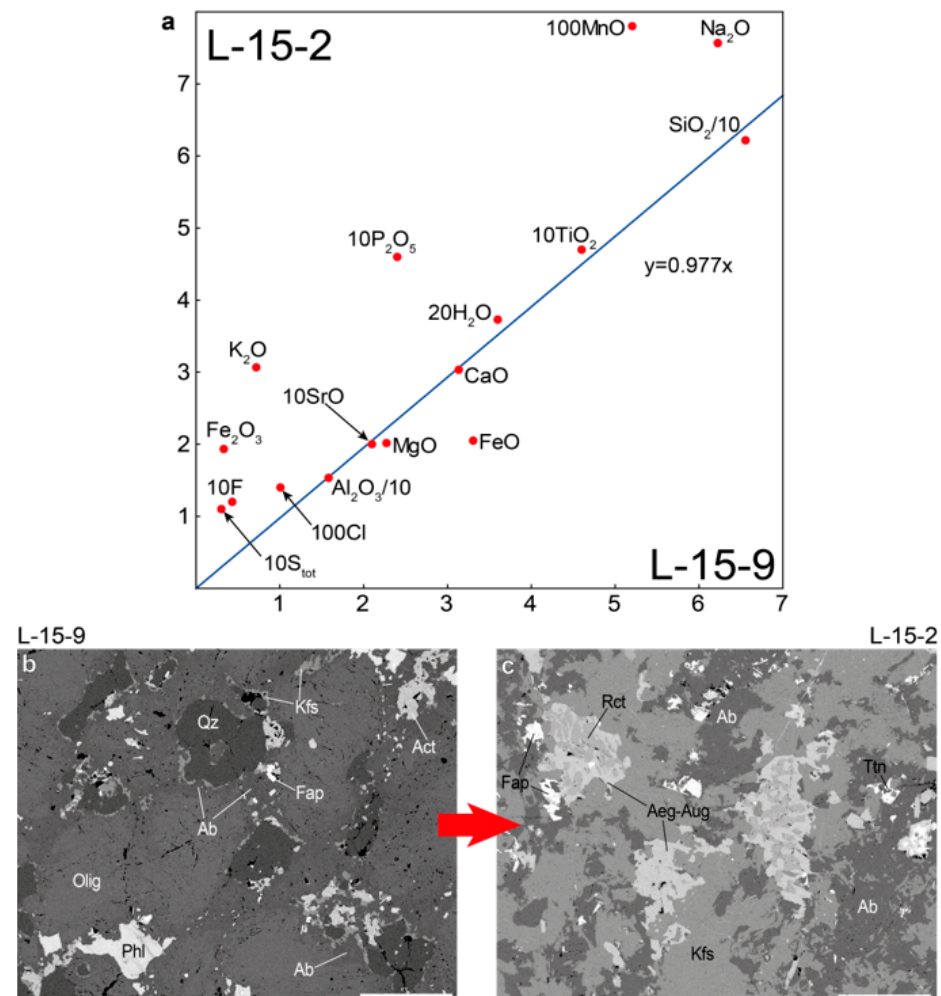


Figure 10. Comparison of low- and high-grade fenites. (a) Isocon diagram comparing the chemistry of low-grade fenite (sample L-15-9) and high-grade fenite (sample L-15-2); In order for the points corresponding to the components to be evenly distributed on the diagram, some values were multiplied by coefficients. For example, $10P_2O_5$ means that the P_2O_5 contents have been multiplied by 10; (b,c) mineral associations of low-grade fenite and high-grade fenite, respectively. The red arrow shows an increase in the intensity of fenitization. Scale bars are 500 microns. BSE-images. See Table 2 for abbreviations.

When the immiscible aqueous and methane fluids infiltrated in amphibole-biotite gneiss, sodium and potassium precipitated from aqueous fluid in the form of albite, nepheline, and K-feldspar. The following zoning is observed in the fenite aureole: at a distance 0–10 cm from the contact there is K-rich fenite, consisting mainly of K-feldspar, and at a distance 10–20 cm from the contact there is Na-rich fenite, consisting mainly of albite and nepheline (Figure 2e,h). According to [58], this zoning may be associated with a

decrease in the NaCl/KCl ratio during fluid-rock interaction at low fluid/rock ratios. The NaCl/KCl ratio decreases as a result of the crystallization of nepheline and albite, which leads to the subsequent crystallization of K-feldspar. Figure 2e,f shows zonal segregations, the central parts of which consist of nepheline and albite, and the outer rim is composed of potassium feldspar. Nepheline and albite crystallized at a high NaCl/KCl ratio, while K-feldspar formed at a low NaCl/KCl ratio. Phosphorus, Ti, F, and Cl precipitate simultaneously with alkalis in the immediate vicinity of the contact and form minerals of the apatite group, titanite, and fluorine-rich Na- and Ca-Na-amphiboles. A low-salinity aqueous fluid, devoid of dissolved components as a result of the precipitation of albite, nepheline and other minerals, together with coexisting methane fluid, was entrapped in quartz under immiscible conditions at or below the CH₄–H₂O solvus. Figure 11 shows the general scheme of fluid evolution during fenitization based on our data.

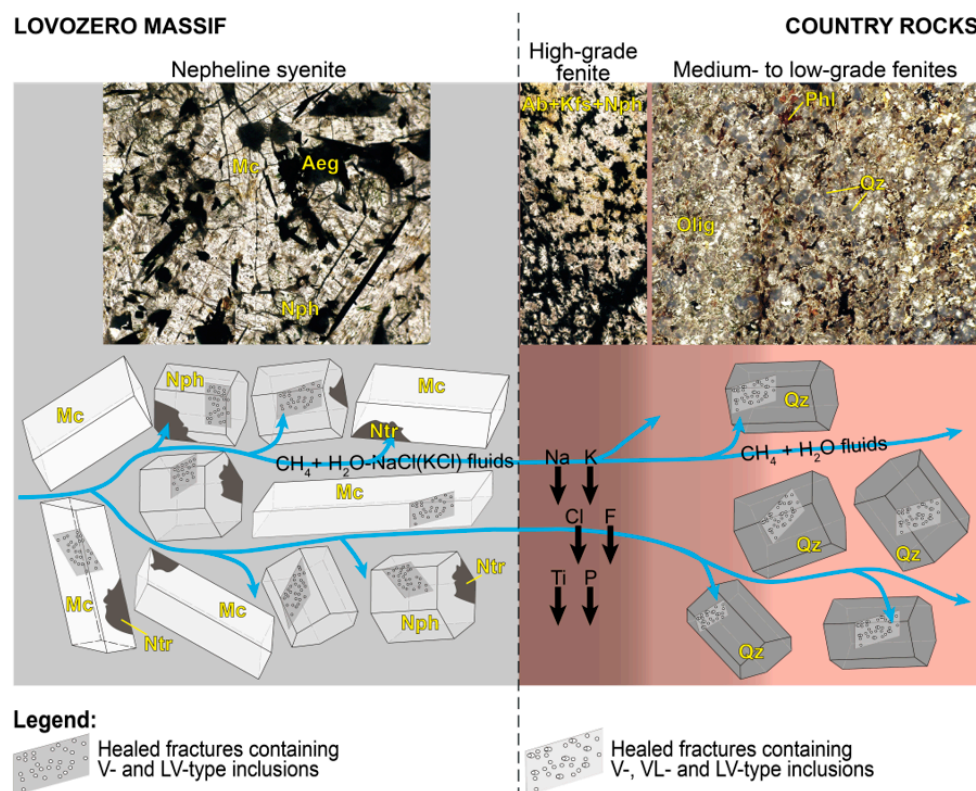


Figure 11. General scheme of fluid evolution during fenitization. At the post-magmatic stage, high saline aqueous {H₂O–NaCl(KCl)} and methane (CH₄) fluids coexisted in the endocontact foyaite. The aqueous fluid was mainly consumed in autometasomatic reactions (replacement of nepheline and K-feldspar by natrolite). Mixtures of two coexisting fluids were entrapped in nepheline and K-feldspar as V- and LV-type inclusions. During the infiltration of fluids into the amphibole-biotite gneiss, the salinity of the aqueous fluid decreased greatly due to the crystallization of fenite minerals. Mixtures of two coexisting (CH₄ and H₂O) fluids were entrapped in quartz as V-, VL- and LV-type inclusions. The blue arrow shows the direction of fluid’s movement. Black arrows indicate the precipitation of components. Please see Table 2 for the abbreviation of minerals.

Petrographic observations (Figure 2a,b) show that the fenitization at the contact of the massif with the country rocks was of a pervasive nature, i.e., fenitizing fluids flowed along the grain boundaries. However, as we established in this work, the aqueous and methane fluids flowing through the country rocks were immiscible. If fluid forms a continuous film through the grain boundaries, it can flow in response to gradients, but the presence of another immiscible phase effectively constricts the apertures available for it to pass through, reducing permeability [59]. Therefore, it can be assumed that such a narrow halo

of fenitization is due not only to the intense autometasomatism of alkaline rocks but also to the immiscibility of the fluids.

6. Conclusions

At the post-magmatic stage, the alkaline rocks of the Lovozero massif contained a heterogeneous mixture of two coexisting fluids, namely medium-salinity (8.6–15.1 eq. wt.% NaCl) aqueous {H₂O–NaCl(KCl)} and methane (CH₄) fluids. The coexistence of these two fluids indicates immiscibility conditions at or below CH₄–H₂O solvus. The aqueous fluid was mainly consumed in autometasomatic reactions, such as natrolitization of nepheline. The methane fluid and a small fraction of the aqueous fluid were entrapped in nepheline (and K-feldspar) as secondary mono-phase vapor (V = CH₄) and liquid-rich two-phase (LV; L = H₂O; V = CH₄) fluid inclusions located along the same healed microfractures.

At the contact of the massif, the mixture of high saline aqueous and methane fluids flowed from endocontact alkaline rocks into country amphibole-biotite gneiss. The aqueous fluid transported K₂O, Na₂O, P₂O₅, TiO₂, H₂O, F, Cl, and S to the exocontact zone. These components were precipitated in the immediate vicinity of the contact of the massif, and the salinity of the aqueous fluid significantly decreased. The methane and low-salinity aqueous fluids were entrapped in quartz as secondary fluid inclusions with variable phase ratios at or below CH₄–H₂O solvus.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/geosciences13100305/s1>, Table S1: Chemical compositions of rocks.

Author Contributions: Conceptualization, O.D.M. and Y.A.P.; methodology, O.D.M.; investigation, O.D.M. and Y.A.P.; resources, O.D.M.; data curation, J.A.M.; writing—original draft preparation, J.A.M.; writing—review and editing, Y.A.P. and O.D.M.; visualization, Y.A.P., O.D.M. and J.A.M. All authors have read and agreed to the published version of the manuscript.

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Appendix A

Table A1. Standards for WDS microprobe analyses.

Element	Standards
Na, Ti	lorenzenite
Al	pyrope
Si, Ca	wollastonite
P	fluorapatite
Fe, S	F ₁₀ S ₁₁
Cl	atacamite
K	wadeite
V	metallic V
Mn	MnCO ₃
Fe	hematite
Sr	celestine
Zr	ZrSiO ₄
Nb	metallic Nb
Ba	baryte
La, Ce	LaCeS ₂
Pr	LiPr(WO ₄) ₂

Table A1. Cont.

Element	Standards
Nd	LiNd(MoO ₄) ₂
Sm	LiSm(MoO ₄) ₂
Hf	metallic Hf
U	metallic U
Ta	metallic Ta
Th	thorite

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