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## NESKEVAARITE-Fe, – $\text{NaK}_3\text{Fe}(\text{Ti}, \text{Nb})_4(\text{Si}_4\text{O}_{12})_2(\text{O}, \text{OH})_4 \cdot 6\text{H}_2\text{O}$ , – A NEW LABUNTSOVITE GROUP MINERAL<sup>1</sup>

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Neskevaarite-Fe, a new labuntsovite-group mineral, was found in the Vuoriyarvi alkaline-ultramafic pluton, Northern Karelia, within a hydrothermally altered carbonatite body. The mineral occurs as rough brown translucent prismatic crystals up to 6 mm long. Associated minerals are dolomite, calcite, phlogopite, fluorapatite, pyrite, pyrrhotite, chalcopyrite, serpentine, and nenadkevichite. Another occurrence is a fieldspare-calcite vein located in the Kukisvumchorr Mt., Khibiny, Kola Peninsula, where the new mineral is closely associated with labuntsovite-Fe.

3 tables, 3 figures, 12 references .

The labuntsovite group takes a special position among a wide variety of aqueous alkaline Ti- and Nb-silicates («amphoterosilicates») due to a unique structural pattern inherent in these minerals. The basis of their structure is a framework consisting of chains of (Ti,Nb)O-octahedra (M) that are linked by four-membered rings of Si<sub>4</sub>O-tetrahedra (T). Large low-valent cations («extra-framework cations» hereafter) and H<sub>2</sub>O are situated in open cavities of this zeolite-like framework. In monoclinic members of the group, chains of (Ti,Nb)O-octahedra (M) can be linked also by additional D,O-octahedra, where D = Mg, Mn, Fe, and Zn (Chukanov *et al.*, 1999, 2002).

If present, the labuntsovite group minerals delimit the boundaries of very special conditions of mineral formation. Hypothetically, the labuntsovite parageneses were formed in alkaline hydrothermalites under a combination of high activities of water, K and/or Na, Ti and/or Nb, and Si at a relatively low P-T level.

While resembling each other in their structural pattern, the labuntsovite group minerals exhibit a considerable variability in their symmetry, topological features of frameworks, and, especially, chemical compositions. Recent

studies indicate that these minerals respond to minor changes in the geochemical environment during the course of crystallization, as well as in subsequent cation-exchange processes. Wide-ranged isomorphous substitutions are common for the extra-framework cations Na, K, Ca, Sr, and Ba (which is also typical of common aluminosilicate zeolites) along with octahedral framework cations. These controls are responsible for the variations within this group of minerals. It provides an effective tool for estimation of large-scale and local activities of mineral forming components. On the other hand, ion-exchange properties inherent in the labuntsovite-like minerals (Pekov *et al.*, 2002a) and their synthetic analogues (Dyer *et al.*, 1999) are of practical interest. The new mineral of the labuntsovite group described here has been found in a drillcore from the Neskevaara Hill, central part of the alkaline-ultrabasic massif Vuoriyarvi, Northern Karelia, Russia (holotype) and in the Khibiny peralkaline massif, Kola Peninsula, Russia. It was named **neskevaarite-Fe** after the discovery locality at the Neskevaara Hill. The suffix -Fe shows Fe prevailing in the D site of the structure, in accordance with accepted rules of

<sup>1</sup> Approved by the Commission on New Minerals and Mineral Names of the International Mineralogical Association, May 02, 2002

**Table 1. Chemical composition of neskevaaraite-Fe (columns 1 and 2) and labuntsovite-Fe (from the intergrowth with the former)**

| Component                      | 1   | 2     | 3     |
|--------------------------------|---|-------|-------|
|                                | wt.%  |       |       |
| Na <sub>2</sub> O              | 3.10 (2.21 – 3.38)  | 3.45  | 5.09  |
| K <sub>2</sub> O               | 8.83 (8.18 – 9.37)  | 9.11  | 7.74  |
| CaO                            | 0.00  | 0.03  | 0.00  |
| SrO                            | 0.00  | 0.07  | 0.00  |
| BaO                            | 3.37 (2.05 – 4.69)  | 5.07  | 8.28  |
| MgO                            | 0.75 (0.57 – 1.06)  | 0.05  | 0.98  |
| MnO                            | 0.50 (0.38 – 0.59)  | 1.03  | 0.11  |
| FeO                            | 1.82 (1.62 – 2.14)  | 1.98  | 1.75  |
| ZnO                            | 0.00  | 0.11  | 0.11  |
| SiO <sub>2</sub>               | 39.29 (38.25 – 40.12)   | 37.95 | 39.62 |
| TiO <sub>2</sub>               | 15.08 (13.30 – 15.94)   | 14.80 | 25.13 |
| ZrO <sub>2</sub>               | 0.00  | 0.08  | 0.08  |
| Nb <sub>2</sub> O <sub>5</sub> | 17.96 (17.21 – 19.04)   | 18.21 | 2.09  |
| H <sub>2</sub> O               | 9.26  | n.d.  | n.d.  |
| Total                          | 99.97   | 91.94 | 90.98 |
|                                | Coefficients in formulas,<br>as calculated per eight Si atoms |       |       |
| Na                             | 1.22  | 1.41  | 1.99  |
| K                              | 2.29  | 2.45  | 1.99  |
| Ca                             | –   | 0.01  | –     |
| Sr                             | –   | 0.01  | –     |
| Ba                             | 0.26  | 0.42  | 0.66  |
| Mg                             | 0.23  | 0.02  | 0.29  |
| Mn                             | 0.09  | 0.18  | 0.02  |
| Fe                             | 0.31  | 0.35  | 0.30  |
| Zn                             | –   | 0.02  | –     |
| Si                             | 8   | 8     | 8     |
| Ti                             | 2.31  | 2.35  | 3.82  |
| Zr                             | –   | 0.01  | –     |
| Nb                             | 1.65  | 1.74  | 0.19  |

**Note:** Neskevaara, Vuoriyarvi (holotype, an average of 7 analyses; the range values bracketed) 2–3 Kukisvumchorr, Khibiny (a neskevaaraite-Fe – labuntsovite-Fe intergrowing)

nomenclature for labuntsovite-group minerals (Chukanov *et al.*, 2002)

The Vuoriyarvi pluton is a typical representative of the central type intrusions composed of alkaline-ultramafic rocks and carbonatites (Kukharensko *et al.*, 1965). The labuntsovite-group minerals are rather common here. These are vuoriyarvite-K (Subbotin *et al.*, 1998), nenadkevichite, labuntsovite-Mg, labuntsovite-Fe, and korobitsynite. These minerals are especially abundant in the Neskevaara area where they occur in hydrothermally altered pyrochlore-bearing carbonatite and phoscorite. All labuntsovite-like minerals have been found there in drillcore samples collected from carbonatite veins at depths of 30 to 780 m from the ground surface. The veins are up to 1 km long and up to 100 m thick, the bulges being even thicker. The pyrochlore-group minerals and zirconolite are considered as sources of Nb and Ti for forming labuntsovite-like phases, which were

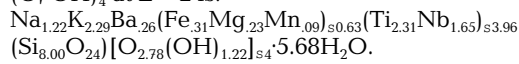
observed only within the zones of low-temperature hydrothermal alterations in carbonatite and phoscorite. These hydrothermally altered rocks are cavernous, and consist of dolomite (60–90%), calcite, siderite, magnesite, chlorite, serpentine, and carbonate-fluorapatite; minor components are barite, sulfides, Ba-, Sr-, and REE-carbonates, as well as quartz and feldspar. Neskevaaraite-Fe was found here in the only holotype drillcore sample as imperfect translucent prismatic brownish crystals up to 6 mm long in hydrothermally altered carbonatite in association with dolomite, calcite, phlogopite, fluorapatite, pyrite, pyrrhotite, chalcopyrite, serpentine, and nenadkevichite. Relicts of pyrochlore occur in the slightly altered host carbonate rock containing calcite, phlogopite, pyroxene, and pyrite as major components.

In Khibiny neskevaaraite-Fe was found in only one sample in the Kirovskii apatite mine at the southern part of the Kukisvumchorr Mt., Khibiny alkaline massif, Kola peninsula, Russia. A.S. Podlesny, a well-known collector, kindly presented this sample to the authors for studies. In this mine the new mineral occurs in cavities within a vein composed of approximately equal parts of medium-grained white calcite and yellowish K-feldspar. Neskevaaraite-Fe forms almost opaque yellowish-brown flattened prismatic crystals up to 1.8 cm long and 1 mm thick in calcite or in open cavities. It forms close intergrowths with labuntsovite-Fe. The latest members of this assemblage are small crystals of donneyite-Y and thin brownish-black solid bitumen coatings. Crystals of neskevaaraite-Fe from Khibiny are usually coarse, plate-shaped and elongated along [010]. However, some vugs carry well-developed crystals with clearly shaped vertices (Fig. 1). The major habit form is the prism formed by longitudinally striated faces {–201}. The additional forms are {100}, {–101}, and less frequently {001}, {021}. Parallel, probably syntaxial neskevaaraite-Fe – labuntsovite-Fe intergrowths were observed. Labuntsovite-Fe occurs as well-shaped transparent crystals (up to 5 mm long) with glossy faces of bright blood-red color; these are in sharp contrast to the murky yellow-brown striated and flattened crystals of neskevaaraite-Fe. All these intergrowths observed in several vugs are identical and consisting of only two crystals: a face {100} of labuntsovite-Fe crystal is contacted with probably a face {100} of coarse neskevaaraite-Fe crystal.

The new mineral has a white streak and vitreous luster; the Mohs' hardness is about 5. The mineral is brittle, has an uneven fracture and

exhibits no cleavage. The density as measured by the heavy liquid method is 2.88(3) g/cm<sup>3</sup>; and the value calculated from the X-ray data is 2.90 g/cm<sup>3</sup>. The mineral is optically biaxial, positive, a 1.677(1), b 1.684(2), g 1.790(5), 2V = 25(10)°, practically non-pleochroic; optical orientation: Y = b.

The cation composition was studied using electron microprobe analysis (Table 1). The water content was determined by the TGA in vacuum; maximum temperature was 950°C, and the heating rate 40°C/min. The empirical formula of the holotype based on  $[\text{Si}_4\text{O}_{12}]_2(\text{O}, \text{OH})_4$  at Z = 2 is:



The simplified formula of neskevaarite-Fe is  $\text{NaK}_3\text{Fe}(\text{Ti}, \text{Nb})_4(\text{Si}_4\text{O}_{12})_2(\text{O}, \text{OH})_4 \cdot 6\text{H}_2\text{O}$ .

Correctness of chemical and optical determinations carried out for neskevaarite-Fe was confirmed by the Gladstone-Dale criterium values (Mandarino, 1981):  $1 - K_p/K_c = 0.021$  for  $D_{\text{obs}}$ ;  $1 - K_p/K_c = 0.028$  for  $D_{\text{calc}}$ . In the Table 2 the X-ray powder diffraction data for neskevaarite-Fe are given (the RKG-86 device, the FeK $\alpha$ -radiation). The *hkl* indices were assigned taking into account  $I_{\text{calc}}$  obtained from the structural data.

The crystalline structure of neskevaarite-Fe was studied for a single crystal from Vuorijarvi using a 4-circle ENRAF NONIUS diffractometer. In the course of crystal structure refinement, microtwinning on (001) and (-401) was taken into consideration. The observed doubled *c* parameter of the orthorhombic pseudo-cell indicated the lemmleinite-type twinning with the following matrix of transition to a true monocline cell:  $[100/010/000.5] + \{100/-0.50 - 0.5\}$ . Estimated values of mass coefficients for the two twin components are 0.53 and 0.47.  $R_{\text{aniso}} = 0.066$ .

Neskevaarite-Fe is monoclinic, the space group *Cm*. The unit cell parameters are

Table 2. Neskevaarite-Fe from Vuorijarvi: X-ray powder diffraction data

| $I_{\text{obs}}$ | $D_{\text{obs}}$ E | $I_{\text{calc}}$ | $D_{\text{calc}}$ E | <i>hkl</i> |
|------------------|--------------------|-------------------|---------------------|------------|
| 100              | 6.93               | 32                | 6.95                | 020        |
|                  |                    | 100               | 6.93                | 001        |
| 20               | 6.45               | 28                | 6.39                | 200        |
|                  |                    | 36                | 6.39                | 20-1       |
| 80               | 4.93               | 32                | 4.91                | 021        |
|                  | 3.52               | 3                 | 3.60                | 40-1       |
| 10               |                    | 3                 | 3.48                | 040        |
| 10               | 3.42               | 7                 | 3.40                | 221        |
|                  |                    | 3                 | 3.39                | 22-2       |
|                  |                    | 17                | 3.20                | 400        |
| 100              | 3.21               | 64                | 3.20                | 42-1       |
|                  |                    | 22                | 3.19                | 40-2       |
| 90               | 3.11               | 41                | 3.11                | 041        |
|                  |                    | 51                | 3.10                | 022        |
|                  |                    | 13                | 2.95                | 112        |
| 30               | 2.91               | 19                | 2.90                | 420        |
|                  |                    | 14                | 2.90                | 42-2       |
|                  |                    | 6                 | 2.62                | 15-1       |
| 60               | 2.62               | 15                | 2.59                | 241        |
|                  |                    | 10                | 2.59                | 24-2       |
|                  |                    | 7                 | 2.59                | 202        |
|                  |                    | 11                | 2.50                | 44-1       |
| 50               | 2.49               | 10                | 2.50                | 401        |
|                  |                    | 19                | 2.50                | 40-3       |
| 5                | 2.14               | 5                 | 2.13                | 600        |
|                  |                    | 6                 | 2.13                | 60-3       |
| 5                | 2.03               | 5                 | 2.03                | 441        |
|                  |                    | 8                 | 2.03                | 44-3       |
| 10               | 1.929              | 7                 | 1.926               | 062        |
|                  |                    | 2                 | 1.925               | 043        |
|                  |                    | 3                 | 1.876               | 460        |
| 10               | 1.873              | 3                 | 1.875               | 46-2       |
|                  |                    | 2                 | 1.872               | 42-4       |
| 10               | 1.801              | 10                | 1.800               | 80-2       |
| 30               | 1.730              | 10                | 1.738               | 080        |
|                  |                    | 5                 | 1.734               | 004        |
|                  |                    | 13                | 1.697               | 44-4       |
| 40               | 1.687              | 2                 | 1.690               | 82-1       |
|                  |                    | 2                 | 1.690               | 82-3       |
|                  |                    | 3                 | 1.685               | 081        |
|                  |                    | 8                 | 1.558               | 84-1       |
| 20               | 1.558              | 3                 | 1.557               | 820        |
|                  |                    | 7                 | 1.557               | 84-3       |
|                  |                    | 2                 | 1.526               | 48-2       |
| 10               | 1.522              | 2                 | 1.524               | 423        |
|                  |                    | 4                 | 1.523               | 42-5       |
|                  |                    | 5                 | 1.426               | 481        |
| 40               | 1.422              | 6                 | 1.426               | 48-3       |
|                  |                    | 7                 | 1.425               | 443        |
|                  |                    | 4                 | 1.297               | 4.10.-1    |
| 30               | 1.297              | 3                 | 1.296               | 482        |
|                  |                    | 3                 | 1.294               | 404        |

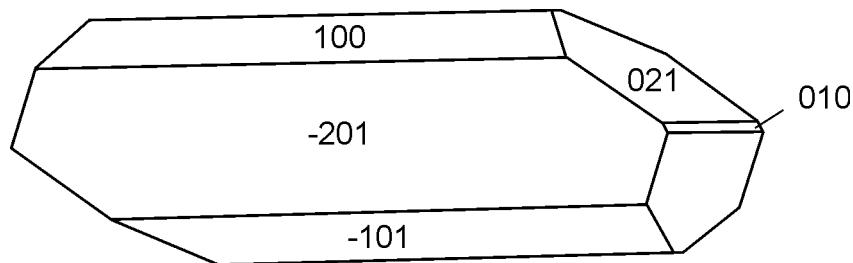


Fig. 1. Neskevaarite-Fe: a crystal from Khibiny

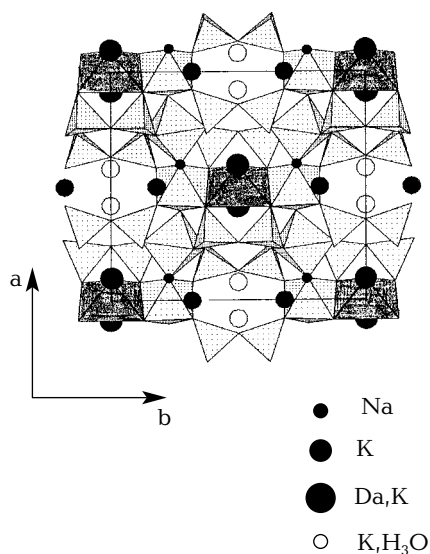


Fig. 2. *Neskevaaraitite-Fe*: the crystal structure; Fe octahedra are given in a dark tone, Ti and Nb – in a light tone.

$a = 14.450(6)$ ,  $b = 13.910(6)$ ,  $c = 7.836(4)$  Å;  $\beta = 117.42(1)^\circ$ ,  $V = 1398(2)$  Å<sup>3</sup>.

*Neskevaaraitite-Fe* is a member of the gutkovaite structural type which, in the labuntsovite group, is characterised by cation ordering in the split A site: A(I) and A(II). In labuntsovite and kuzmenkoite, instead, the A is unique and is occupied by Na and vacant in the two structural types, respectively. The split of the A site into A(I) and A(II) with different occupancy lowers the symmetry from  $C2/m$  [labuntsovite structural type, where A(I) and A(II) are related by an inversion centre] to  $Cm$  (gutkovaite structural type); that suggested to establish the gutkovaite subgroup within the labuntsovite group (Chukanov *et al.*, 2002).

The framework of the structure of all the labuntsovite minerals is identical to that occurring, for example, in labuntsovite-Mn (Chukanov *et al.*, 1999). It consists of corrugated columns of (Ti,Nb)-octahedra connected by (Si<sub>4</sub>O<sub>12</sub>) rings and additional D octahedra occupied by Mn<sup>2+</sup> in gutkovaite-Mn, Zn in alsakharovite-Zn, and Fe in neskevaaraitite-Fe. Alkali and alkali-earth cations and water molecules are situated in cavities of this framework. The main difference between the labuntsovite and gutkovaite structural types is represented by the A sites, as said above. The A(1) and A(2) sites located at 1.73 Å from each other and filled statistically with Na or K atoms, respectively. Potassium atoms occupy the positions, which in labuntsovite *sensu strictu* are filled

with H<sub>2</sub>O(1) molecules; this site, as well as the Sr site in alsakharovite-Zn, is dislocated towards Na. Reduced Na-H<sub>2</sub>O(2) distance (1.97 Å) is due to incomplete occupation of these sites both by cations and anions.

The split B-site in neskevaaraitite-Fe is occupied by K<sup>+</sup>-ions and minor amounts of H<sub>3</sub>O<sup>+</sup> distributed between sub-positions; such configuration of the B-position is inherent in alsakharovite-Zn. A weak band at 1717 cm<sup>-1</sup> indicates the presence of H<sub>3</sub>O<sup>+</sup> in the neskevaaraitite structure.

A crystallochemical formula of neskevaaraitite-Fe, as calculated from structural data at  $Z = 1$ , is:

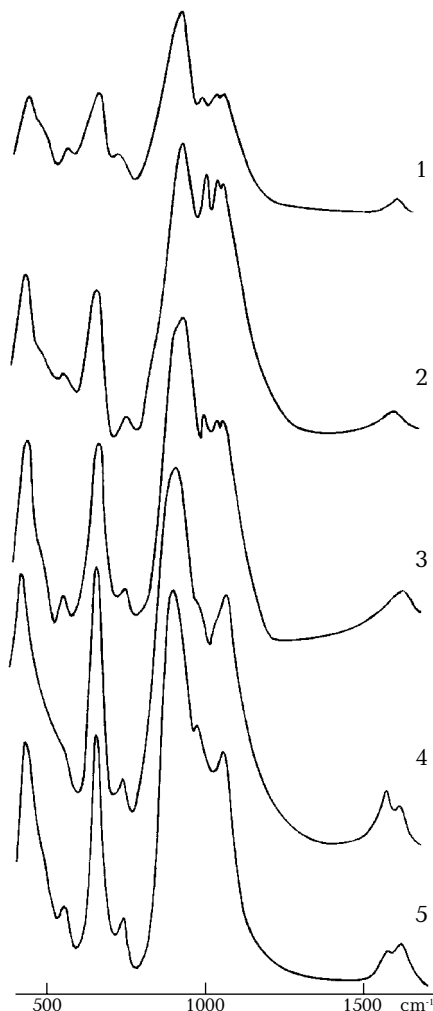
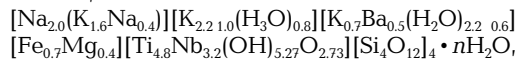


Fig. 3. IR-spectra of the gutkovaite and kuzmenkoite subgroup minerals: 1 – gutkovaite-Mn, 2 – alsakharovite-Zn, 3 – neskevaaraitite-Fe, 4 – kuzmenkoite-Mn, 5 – kuzmenkoite-Zn

**Table 3. The gutkovaite subgroup minerals: a comparison of crystallochemical characteristics**

| Mineral             | Gutkovaite-Mn  | Neskevaarite-Fe  | Alsakharovite-Zn  |
|---------------------|--|--|---|
| Simplified formula  | $\text{CaK}_2\text{Mn}(\text{Ti}, \text{Nb})_4(\text{Si}_4\text{O}_{12})_2(\text{O}, \text{OH})_4 \cdot 5\text{H}_2\text{O}$ | $\text{NaK}_3\text{Fe}(\text{Ti}, \text{Nb})_4(\text{Si}_4\text{O}_{12})_2(\text{O}, \text{OH})_4 \cdot 6\text{H}_2\text{O}$ | $\text{NaSrKZn}(\text{Ti}, \text{Nb})_4(\text{Si}_4\text{O}_{12})_2(\text{O}, \text{OH})_4 \cdot 7\text{H}_2\text{O}$ |
| Space group         | <i>Cm</i>  | <i>Cm</i>  | <i>Cm</i>   |
| <i>a</i> , Å        | 14.365   | 14.45  | 14.49   |
| <i>b</i> , Å        | 13.89  | 13.91  | 13.91   |
| <i>c</i> , Å        | 7.81   | 7.84   | 7.82  |
| $\beta$ , °         | 117.4  | 117.4  | 117.6   |
| Prevailing cations: |  |  |   |
| A(1)                | Ca   | Na   | Na  |
| A(2)                |  | K  | Sr  |
| B                   | K  | K  | K   |
| D                   | Mn   | Fe   | Zn  |
| M                   | Ti   | Ti   | Ti  |

where the compositions of the four key groups in the A, B, C, and D-positions are attributed to the first four pairs of brackets.

A partially ordered Ti and Nb distribution between different octahedral sites is a typical feature of neskevaarite-Fe: in one site Ti prevails, whereas another one is occupied by equal amounts of Ti and Nb.

It is noteworthy that the role of water in the gutkovaite sub-group minerals is different from that in other labuntsovite-group minerals. In labuntsovite, two independent sites of the water molecules occur, of which  $\text{H}_2\text{O}(1)$  is coordinates the A and B cation sites, whereas  $\text{H}_2\text{O}(2)$  coordinates only the A site. Both sites of  $\text{H}_2\text{O}$  can be displaced towards the A site, which is commonly occupied by Na. In tsepinite-Na both  $\text{H}_2\text{O}$  positions are displaced and occupied by  $\text{H}_3\text{O}^+$ . In kuzmenkoite  $\text{H}_2\text{O}(1)$  is preserved, and  $\text{H}_2\text{O}(2)$  is replaced by  $\text{H}_3\text{O}^+$ . On the contrary, in gutkovaite-like structures  $\text{H}_2\text{O}(2)$  is preserved, whereas  $\text{H}_2\text{O}(1)$  is displaced and may be occupied by cations (*e. g.*, Sr). In addition, in gutkovaite-type structures Ba selectively occupies only one vertices of the D-octahedron when the D site is vacant which is one of the reasons that the symmetry grade is lowered.

Rozenberg *et al.* (2002b) give the details of crystalline pattern of neskevaarite-Fe. Table 3 presents the characteristics of the gutkovaite group minerals.

The wave numbers of the IR-spectrum bands are ( $\text{cm}^{-1}$ ; sh - shoulder, s - strong band): 3530, 3340, 1653, 1083 s, 1059 s 1025 s, 951 s, 930 sh, 770, 686 s, 584, 530 sh, 458. By the IR-spectra, the gutkovaite sub-group members can be distinguished from other labuntsovite group minerals, including members of the kuzmenkoite sub-group (Fig. 3).

As a conclusion we emphasize that the gutkovaite and labuntsovite (*sensu strictu*) - subgroup members are essentially different. The fact that intergrowth of neskevaarite-Fe and labuntsovite-Fe was found to occur in Khibiny, suggests that these minerals as separate species consisting of the same chemical elements, but different in structural patterns. The individuality of neskevaarite-Fe is demonstrated also by the admixture chemistry. Neskevaarite-Fe is poor in Ba and Mg relative to associating labuntsovite-Fe, but it is rich in Mn and especially in Nb (see items 2 and 3 in Table 1). It is reasonable to assume that Nb stabilizes the neskevaarite-Fe structure as well as structures of other kuzmenkoite-subgroup minerals.

Type specimen of neskevaarite-Fe is deposited in the Fersman Mineralogical Museum of Russian Academy of Sciences, Moscow, Russia (reg. no. 2814/1).

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