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LITHIUM IN SEKANINAITE FROM THE TYPE LOCALITY, DOLNÍ BORY, CZECH REPUBLIC

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ABSTRACT

The (Si,Al)-ordered Fe end-member of the cordierite group, sekaninaite, from its type locality at Dolní Bory, Czech Republic, was analyzed, and found to contain 0.04 – 0.24 Li and 0.11 – 0.26 Na *apfu*; (Fe + Mn)/(Mg + Fe + Mn) (at.) ranges from 0.74 to 0.97. Lithium is incorporated by the substitution $^{ch}Na^{VI}Li(Mg,Fe)_{-1}$. This sekaninaite is the first member of the cordierite group reported to have substantial Li but negligible Be. All samples of Na,Be-, Na,Li,Be- and Na,Li-bearing cordierite–sekaninaite examined to date show a minor deficit in the tetrahedral site (0.237 in the literature, 0.036 in our data) and a slight excess of channel cations (Na,K,Ca) over the proportion of (Be,Li) (at.). The Li-bearing sekaninaite comes from rather simple granitic pegmatites, in which the only Li-bearing minerals are rare triphylite and cookeite. Thus cordierite-group minerals are crystal-chemical sinks for Li. This finding stresses the need to analyze for trace light elements, even in minerals from poorly fractionated and rare-element-depleted environments, provided the crystal chemistry is favorable.

Keywords: sekaninaite, cordierite group, lithium, beryllium, Czech Republic.

SOMMAIRE

Nous avons analysé la sekaninaïte, pôle ferrifère ordonné en Al,Si du groupe de la cordiérite, provenant de sa localité type, à Dolní Bory, en République Tchèque. Elle contient entre 0.04 et 0.24 atomes de Li et entre 0.11 et 0.26 atomes de Na par unité formulaire. Le rapport (Fe + Mn)/(Mg + Fe + Mn) (at.) va de 0.74 à 0.97. Le lithium est incorporé selon le schéma $^{canal}Na^{VI}Li(Mg,Fe)_{-1}$. Cette sekaninaïte fournit le premier exemple d'un membre du groupe de la cordiérite contenant une proportion appréciable de Li, mais en même temps, une proportion négligeable de Be. Tous les exemples de cordiérite – sekaninaïte contenant Na,Be, Na,Li,Be et Na,Li étudiés jusqu'ici font preuve d'un léger déficit d'occupation dans le site tétraédrique (0.237 dans la littérature, 0.036 pour nos données), et d'un léger excédent en teneurs de (Na,K,Ca) dans les canaux par rapport aux teneurs en (Be,Li), en proportions atomiques. La sekaninaïte à Li provient d'exemples de pegmatite granitique relativement simple, dans lesquels les seuls minéraux à contenir le Li essentiel sont triphylite et cookeïte, rares. C'est donc dire que les minéraux du groupe de la cordiérite seraient un hôte privilégié du Li. Cette situation souligne la nécessité de chercher à établir les teneurs en éléments légers à l'état de trace, même dans les minéraux provenant de milieux peu évolués et appauvris en éléments rares, pourvu que la situation cristallochimique en est favorable.

(Traduit par la Rédaction)

Mots-clés: sekaninaïte, groupe de la cordiérite, lithium, béryllium, République Tchèque.

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INTRODUCTION

Sekaninaite, ideally $Fe_2Al_4Si_5O_{18}$, is the Fe-dominant and Al–Si-ordered member of the cordierite group. It was synthesized by several authors ["ferrocordierite" of Schreyer (1965); *cf.* Boberski & Schreyer (1990)], first noticed as a natural mineral by Sekanina (1928), described as a new species by Staněk & Miškovský (1975; *cf.* Staněk & Miškovský 1964), and also studied by electron microprobe (Goldman *et al.* 1977, Selkregg & Bloss 1980, Schreyer *et al.* 1993) and by X-ray diffraction [structure refinement by Hochella *et al.* (1979), unit-cell dimensions and selected physical properties by Selkregg & Bloss (1980)]. In 1990, during the initial stages of examination of associated ferrogedrite, electron-microprobe analyses (EMPA) were performed at the University of Manitoba on sekaninaite from its type locality at Dolní Bory, western Moravia, Czech Republic. The analyses yielded totals slightly lower than expected from the previously known content of H_2O^+, and formulas deviating from the ideal stoichiometry (high levels of Na but deficit in the octahedrally coordinated sites). The stoichiometry ruled out potential substitution by Be, and Li was not seriously considered, as the parent granitic pegmatites are virtually devoid of lithium-bearing minerals. The low totals were first attributed to improper standards, but re-analysis of the examined specimens at the Ruhr-Universität confirmed the results obtained at Manitoba. Thus the presence of Li remained the only viable possibility, and was subsequently confirmed. We report here on our results of extensive chemical analyses of sekaninaite from its type locality, and on its geochemical significance.

SAMPLES EXAMINED

The high content of Na found in the first Li-bearing specimens examined suggested that the presence of octahedrally coordinated Li could be compensated by incorporation of Na into the channels of the structure. A total of 27 samples of sekaninaite from the collection of the Moravian Museum, and eight samples from the collection of the Department of Mineralogy, Petrography and Geochemistry, Masaryk University were analyzed by electron microprobe for Na as a monitor of Li, along with two samples from the R.B. Ferguson Museum of Mineralogy at the University of Manitoba, four samples from the research material of Dr. J. Staněk (Masaryk University) and three samples from the research collection of P.Č. Seven specimens covering the full range of Na_2O encountered, 0.13 – 1.57 wt.%, were selected for detailed analysis by EMPA, secondary-ion mass spectrometry (SIMS) and Mössbauer spectroscopy (Table 1). A specimen of sekaninaite collected at Dolní Bory in 1963 by W.S. (TS 25) was analyzed at the Ruhr-Universität, and also was subjected to Mössbauer analysis (Table 1).

TABLE 1. SAMPLES OF THE DOLNÍ BORY SEKANINAITE EXAMINED

#	Source	Na_2O,wt.%*
X3	Research collection of J. Staněk	1.30
X6	Research collection of J. Staněk	1.30
X33	Dept. Mineralogy, Petrography and Geochemistry, Masaryk University, No. 11.220	0.45
X38	Dept. Mineralogy and Petrology, Moravian Museum, No. 691	0.80
X39	Dept. Mineralogy and Petrology, Moravian Museum, No. 1185	0.62
X51	Dept. Mineralogy and Petrology, Moravian Museum No. a583	0.95-1.03
X64	Dept. Mineralogy and Petrology, Moravian Museum, No. a1441	1.08-1.28
TS25	Research collection of W. Schreyer	(0.90)

*Reconnaissance data determined by testing all of the 46 specimens available for study, except TS25.

ANALYTICAL METHODS AND MÖSSBAUER RESULTS

EMPA analyses of seven specimens at the University of Manitoba were done using a Cameca SX–50 instrument, with an operating voltage of 15 kV and a sample current of 20 nA, measured on a Faraday cup; counting time was 20 s for the $K\alpha$ lines of Na (albite standard), Fe (fayalite), Si (diopside), Mn (spessartine), Al (kyanite) and Mg (olivine), and 40 s for $K\alpha$ of K (orthoclase), Ca (diopside), Ti (titanite), Cr (chromite), Zn (gahnite), F (fluor-richterite), and $L\alpha$ of Cs (pollucite) and Rb (rubidian microcline). The data were reduced using the PAP procedure of Pouchou & Pichoir (1985).

SIMS analyses of the above specimens for Li, Be and B were performed at the CNR Centro di Studio per la Cristallochimica e Cristallografia in Pavia, under analytical conditions fully described by Ottolini *et al.* (1993). Briefly, positive ions of the isotopes ^{7}Li, ^{9}Be and ^{11}B (plus ^{30}Si, assumed as the matrix reference isotope), with emission kinetic energies ranging from ~75 to 125 eV, were monitored using a Cameca IMS–4f ion microprobe. Medium- to high-energy secondary ions were used to reduce matrix effects affecting especially Li–Si ionization and to improve measurement reproducibility with respect to low-energy ion analysis. The quantification of Li, Be and B ion signals was carried out by means of the empirical approach of working curves *via* calibration with standards. Since the relative yields for Li ions, with emission energies in the range 75–125 eV, showed a direct correlation with the silica content of the matrix (Ottolini *et al.* 1993), an empirical correction was made to account for matrix effects at this silica value: for the quantification of lithium, we used an ion yield that is typical of low-silica samples, which is lower by about 25% than that of high-silica samples. No special corrections were employed to quantify beryllium or boron contents. Under these circumstances, the accuracy for Li (as well as Be and B) is estimated to be better than 10% relative. Reproducibility for Li, Be and B measurements within a one-day analytical session was typically a few % relative; it was tested on homogeneous synthetic glass standards.

The concentrations of Li, Be and B reported here are the averages of five determinations on each sample. Lithium was found in the range of 0.11 – 0.55 wt.% oxide; Be and B were found to be negligible (tens of ppm oxides), insignificant for the formulae.

The Mössbauer spectra were collected at the Bayerisches Geoinstitut in Bayreuth, using powdered material in a conventional transmission Mössbauer spectrometer. Sample thicknesses varied from 2.5 to 5 mg Fe/cm^2, depending on the amount of material available. The room-temperature Mössbauer spectra of all samples are identical, consisting of one quadrupole doublet with hyperfine parameters $\delta = 1.212 \pm 0.005$ (relative to α–Fe) and $\Delta E_Q = 2.23 \pm 0.01$ mm/s. These parameters are consistent with Fe^{2+} in the octahedral site, and are in excellent agreement with results of previous studies (Duncan & Johnston 1974, Goldman *et al.* 1977). We did not find spectral evidence for Fe^{2+} in the channel sites, nor for Fe^{3+}. To determine the minimum detection-limit for Fe^{3+}, we fit all spectra to an additional doublet corresponding to Fe^{3+}, and used an F test to determine the level at which addition of the doublet is statistically significant (*e.g.*, Bevington 1969). The measured values for Fe^{3+}/ΣFe were all close to 0.5%, but in only two spectra (samples X–3 and X–6) was the presence of Fe^{3+} statistically significant at higher than the 50% level. This implies that the amount of Fe^{3+} in the remaining samples is less than 0.5%, and therefore below the detection limit. For samples X–3 and X–6, the amount of Fe^{3+} could possibly be as high as 0.5%, but is likely lower.

It is interesting to note that the color and pleochroism of Fe-bearing cordierite are interpreted to arise from charge transfer between octahedral Fe^{2+} and Fe^{3+} in channel(?), tetrahedral or octahedral sites (Goldman *et al.* 1977, Boberski 1987), which would imply that our samples contain Fe^{3+}. However, very little Fe^{3+} is required to produce sufficient absorption for a blue-to-violet color to be visible, since extinction coefficients of intervalence charge-transfer bands are typically 10 to 100 times greater than those for spin-allowed crystal-field transitions (Burns 1993). A value of Fe^{3+}/ΣFe certainly less than 0.5% would be sufficient (Goldman *et al.* 1977). The same Mössbauer analysis was performed to determine the detection limit for channel Fe^{2+}; it was also found to be 0.5%.

The sample TS 25 examined at the Ruhr-Universität was analyzed on the Cameca CAMEBAX instrument, under conditions analogous to those stated above. Wet-chemical analysis was performed for Li and Be on material cleaned under a binocular microscope. The concentration of both elements was determined by inductively coupled plasma – emission spectroscopy. This sample was also subjected to the Mössbauer spectroscopic examination discussed above.

CHEMICAL COMPOSITION OF SEKANINAITE

Table 2 shows the combined results of EMPA, SIMS, Mössbauer and, in the case of sample TS 25, wet-chemical analysis. The Fe$_2$O$_3$ contents could not be quantified. To document the presence of Fe^{3+} indicated by the medium to deep blue color of sekaninaite, its content was fixed for all samples at the detection limit of the Mössbauer technique, corresponding to the maximum possible (although in most cases unlikely) Fe^{3+} contents of samples X–3 and X–6 (as discussed above). Any error involved in this procedure is trivial, because assigning 0.5% or less of ΣFe to the trivalent state instead of none has negligible effect on the atomic contents of other elements.

TABLE 2. CHEMICAL COMPOSITION OF SEKANINAITE FROM DOLNÍ BORY

Sample #	X33	X39	X38	TS25	X51	X3	X64	X6
SiO$_2$,wt.%	45.47	46.24	44.62	44.42	45.03	45.56	44.95	45.61
TiO$_2$	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02
Al$_2$O$_3$	30.95	31.41	30.89	31.38	30.36	30.80	30.23	30.99
B$_2$O$_3$	0.002	0.002	0.004	-	0.003	0.004	0.003	0.005
Fe$_2$O$_3$*	0.09	0.09	0.10	0.10	0.10	0.10	0.09	0.10
Cr$_2$O$_3$	0.01	0.01	0.00	-	0.01	0.00	0.01	0.01
FeO	15.59	15.73	18.06	18.00	17.07	17.39	16.58	17.27
MnO	0.63	0.70	0.96	1.02	1.38	1.22	1.87	1.40
ZnO	0.01	0.00	0.00	-	0.00	0.03	0.01	0.01
MgO	3.06	2.64	1.05	0.85	0.38	0.42	0.33	0.42
CaO	0.04	0.05	0.06	0.04	0.02	0.03	0.01	0.03
BeO	0.008	0.006	0.002	0.002	0.001	0.001	0.003	0.001
Li$_2$O	0.107	0.169	0.169	0.35	0.415	0.474	0.489	0.551
Na$_2$O	0.54	0.69	0.74	0.90	1.11	1.23	1.16	1.19
K$_2$O	0.06	0.00	0.01	0.02	0.01	0.02	0.01	0.01
Cs$_2$O	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00
H$_2$O+	-	-	-	2.02	-	-	-	-
TOTAL	96.567	97.737	96.655	99.10	95.889	97.279	95.755	97.617
Si	4.968	4.984	4.940	4.900	5.010	4.998	5.009	4.984
Al	3.986	3.994	4.030	4.077	3.981	3.982	3.970	3.991
F^{3+}	0.007	0.008	0.008	0.008	0.008	0.008	0.008	0.008
Be	0.003	0.002	0.001	0.001	0.000	0.000	0.001	0.000
$^{IV}\Sigma$	8.964	8.988	8.979	8.986	8.999	8.988	8.988	8.983
Fe^{2+}	1.425	1.419	1.672	1.659	1.588	1.595	1.545	1.578
Mg	0.498	0.424	0.173	0.140	0.063	0.069	0.055	0.068
Mn	0.058	0.065	0.090	0.095	0.130	0.113	0.176	0.130
Zn	0.001	0.000	0.000	-	0.000	0.002	0.001	0.001
Ti	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.002
Cr	0.001	0.001	0.000	-	0.001	0.000	0.001	0.001
Li	0.045	0.069	0.075	0.154	0.183	0.209	0.220	0.239
$^{VI}\Sigma$	2.028	1.978	2.010	2.048	1.965	1.988	1.998	2.019
Na	0.114	0.146	0.159	0.193	0.239	0.262	0.251	0.252
K	0.008	0.000	0.001	0.003	0.001	0.003	0.001	0.001
Ca	0.005	0.006	0.007	0.005	0.002	0.007	0.001	0.004
$^{ch}\Sigma$	0.127	0.152	0.167	0.201	0.242	0.272	0.253	0.257
H$_2$O	-	-	-	0.743	-	-	-	-

Calculated on the basis of 18 atoms of oxygen per anhydrous formula unit.
* See text for explanation. – Concentration not determined.

The atomic contents were assigned to the tetrahedral, octahedral, and channel sites in the conventional manner (Table 2). The only degree of uncertainty concerns the role of Fe^{3+}, which is listed as tetrahedral in accordance with its most common role, well documented by Černý & Povondra (1966), among others, and favored by Schreyer *et al.* (1990). Alternatively, Fe^{3+} could reside in the octahedra or, possibly, in the channels (Goldman *et al.* 1977, Boberski 1987).

DISCUSSION

The formula of sekaninaite

Sekaninaite from its type locality basically conforms to the bulk composition established by Staněk & Miškovský (1975), but with significant addition of Li, the content of Fe^{3+} much lower than their values, and slight deviations from the ideal contents of Si and Al. The composition is, however, variable among different samples. The atomic ratio (Fe + Mn)/(Fe + Mn + Mg) varies from 0.74 to 0.97 in the samples examined, bracketing the values found in the holotype samples of Staněk & Miškovský (1975): 0.819 and 0.826. Also, ranges of Li from 0.045 to 0.239 and Na from 0.114 to 0.262 atoms per unit cell (*apfu*) were found. Sekaninaite is the first member of the cordierite group known to incorporate substantial Li but negligible Be.

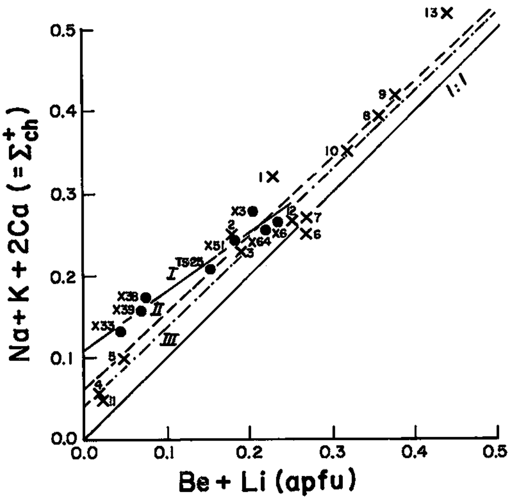

FIG. 1. Sum of the charges on the channel-hosted cations *versus* total of (Be + Li). Samples of sekaninaite from Dolní Bory are marked by solid circles. 1 to 3: Alpe Sponda, 4 and 5: Miregen, 6 and 7: Haddam (Armbruster & Irouschek 1983); 8 to 10: El Peñón (Gordillo *et al.* 1985); 11 and 12: Soto (Schreyer *et al.* 1979); 13: Věžná (Černý & Povondra 1966). Regression lines: I: sekaninaite Dolní Bory, II: all data, III: data from the literature (1 to 13 above). See the text for regression equations.

Lithium is incorporated into the octahedra by a coupled substitution $^{ch}Na^{VI}Li\,(Mg,Fe)_{-1}$, suspected in natural cordierite by Armbruster & Irouschek (1983), confirmed in natural sekaninaite by Gordillo *et al.* (1985), and experimentally verified in end-member cordierite by Kirchner *et al.* (1984). The substitution $LiSi([Mg,Fe]Al)_{-1}$, proposed but unproven by Ginsburg & Stavrov (1961), possibly shown by results of an ancient analysis by von Kokscharow (1858), and potentially exhibited by experimental products of Karkhanavala & Hummel (1953), is not involved. However, the content of Na is in many cases significantly higher than that dictated by the above mechanism, as also found in Be,Li-bearing samples by Schreyer *et al.* (1979), Armbruster & Irouschek (1983), Gordillo *et al.* (1985) and Grew *et al.* (1990) (Fig. 1). This relationship may also hold for sekaninaite from Kemiö (Povondra *et al.* 1984) and cordierite from Haddam (Povondra & Čech 1978), but the lithium content of these samples was not determined. Linear regressions (Fig. 1) calculated for the samples of sekaninaite examined from Dolní Bory (I), for the data by Gordillo *et al.* (1985; III), and for the two sets combined (II) are:

$$\mathrm{Na} = 0.6978\,(\mathrm{Be} + \mathrm{Li}) + 0.1091 \quad (r = 0.967) \qquad \text{(I)}$$

$$\mathrm{Na} = 0.9248\,(\mathrm{Be} + \mathrm{Li}) + 0.0622 \quad (r = 0.9660) \qquad \text{(II)}$$

and

$$\mathrm{Na} = 0.948\,(\mathrm{Be} + \mathrm{Li}) + 0.0422 \quad (r = 0.940) \qquad \text{(III)}$$

As shown in Figure 2, the ratio Si/(Al + Be) is somewhat variable, albeit largely within the limits of experimental error. The substitution $NaAlSi_{-1}$ may account for the Si-deficient samples TS 25 and X–3; for the other samples, which seem to be Al-deficient, the substitution $NaMgAl_{-1}$ can be invoked (Fig. 2). Both mechanisms were proven experimentally and in natural specimens (Schreyer 1964, Wolfsdorff & Schreyer 1992). Alternatively, the excess Na could be charge-balanced by introduction of $(OH)^{-}$ groups attached directly to Na^{1+} in the channels. So far, hydroxyl has not been documented in minerals of the cordierite group (*e.g.*, Schreyer 1985; P. Mirwald, pers. comm. 1995), although Aurisicchio *et al.* (1994) provided convincing evidence for Na(OH) couples in the channels of beryl.

It must be stressed, however, that the deviations of the sekaninaite formula from ideality are very small, and do not exceed analytical error. Slight inaccuracies in calibration for some elements may account for some of the systematic shifts, as shown by "error" bars in Figure 3. This graph illustrates deficit in occupancy of

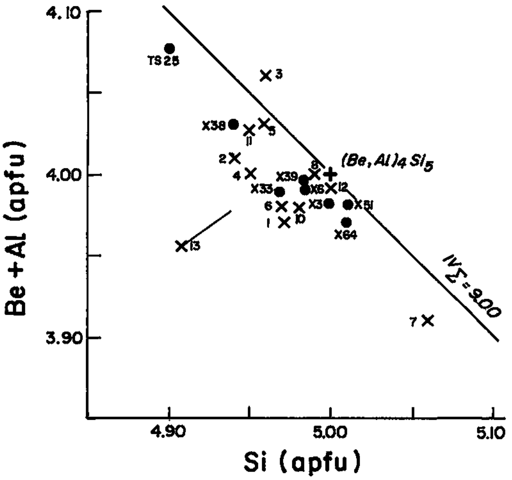

FIG. 2. Sum of (Be + Li) *versus* Si. Symbols and sample numbers are as in Figure 1. End of the vector extending from sample 13 marks approximate plot of this cordierite sample if its tetrahedrally coordinated Fe^{3+} is proportionally split between (Be + Al) and Si. Note that Si is slightly less than 5.00 in most samples, and the total population of tetrahedrally coordinated cations is slightly less than 9.00 in all samples but one.

the tetrahedral sites, up to 0.036 atoms out of the total of 9.000. This deficit would be lower in samples for which Fe^{3+} was not determined. Assuming all the Fe as divalent would increase the total occupancy of octahedra above 2.00. It must be noted that a similar deficit in the *T* site is shown by the Argentinian samples of Schreyer *et al.* (1979), for which Fe^{3+} was determined.

Boron and beryllium are present in insignificant amounts only. Appreciable contents of Be^{2+}, substituting for Al^{3+} and compensated by channel Na^+, were found in several occurrences of cordierite (Černý & Povondra 1966, 1967, Piyar *et al.* 1968, Povondra & Čech 1978, Selkregg & Bloss 1980, Povondra *et al.* 1984, Orlandi & Pezzotta 1995, Schreyer *et al.* 1979). However, B is found in negligible amounts only in minerals of the cordierite group, although it was rarely sought (*cf.* Grew *et al.* 1990, 1991, 1995).

Geochemical significance of light elements in rock-forming minerals

The results of our re-examination of sekaninaite emphasize again the fundamental need for analyses of minerals for light elements, even in environments that are not particularly suggestive of the potential presence of such elements in significant quantities. Minerals never suspected to contain, and never analyzed for, light elements are recognized today as their carriers in increasing numbers. This is particularly significant in environments in which light-element concentrations are unexpected, such as ordinary granites (Schreyer *et al.* 1979, Hawthorne *et al.* 1993), metamorphic rocks in general (as reviewed by Hawthorne 1995) and those equilibrated in the granulite facies in particular (*e.g.*, Grew *et al.* 1990, 1991, 1995).

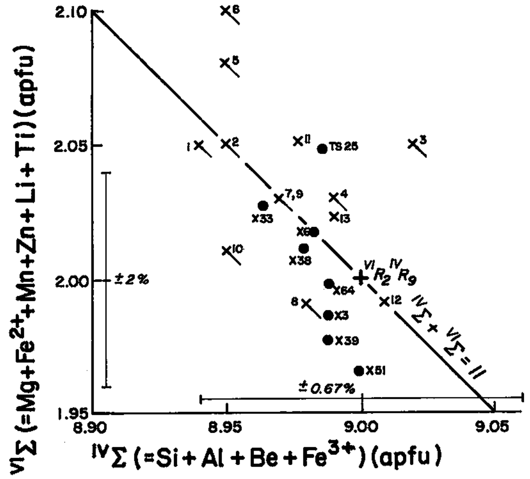

FIG. 3. Sum of octahedrally coordinated cations *versus* sum of tetrahedrally coordinated cations. Symbols and sample numbers are as in Figure 1. Note that the sum of tetrahedrally coordinated cations is slightly less than 9.00 in all samples but one, whereas the sums of octahedrally coordinated cations are slightly higher than 2.00 in most samples. Samples for which ΣFe was determined as FeO only are marked by vectors pointing in the direction of the shift that would be generated by converting some Fe to trivalent state and tetrahedral coordination: note that all but one of these samples have octahedral-site occupancy greater than 2.00, and all but one have tetrahedral-site occupancy less than 9.00. However, note the "error" bars added to emphasize the very small deviation from ideality in most samples.

The present case serves as yet another example of the above feature. The Dolní Bory – Hatě pegmatites show a poorly evolved geochemical signature: the rare-earth elements, Ti, B, W, Nb>Ta, and P are dominant in the accessory minerals (Staněk 1954, 1991). The only mineralogical expression of Li was identified there in extremely rare primary triphylite, and in equally scarce secondary cookeite (J. Staněk and P. Povondra, pers. comm., 1993). However, relatively large quantities of Li must have been incorporated into sekaninaite, which was rather abundant in some of the pegmatite veins, in crystals up to 70 cm long (Staněk 1954).

Under conditions of high activity of Na, minerals of the cordierite group evidently are good sinks for Li, even if this element is present in negligible concentration only in the parent medium. Because of the close structural similarity to beryl, Cs also may be expected to enter the channels of the cordierite framework. Cordierite is the only magmatic mineral in granitic rocks in which Cs behaves compatibly (London 1995). Concentrations of Li and Cs should be examined in cordierite from highly fractionated pegmatites and from Li,Rb,Cs-enriched metasomatic assemblages dispersed in their host rocks.

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