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CHLORINE IN VESUVIANITES

Abstract. Vesuvianites with high contents of chlorine near 1 *apfu* occur in the rodingite-like rocks of the Wiluy River, Sakha-Yakutia, Russia — the type locality for grossular and wiluite. Chlorine is incorporated into the vesuvianite structure at the *O*(10) site substituting O, OH or F. The classification of vesuvianite-group minerals is discussed on the basis of available structural data from Cl- and F-bearing vesuvianites. A ternary O-F(+Cl)-OH diagram (hypothetical "oxyvesuvianite" — fluorvesuvianite — hypothetical "hydroxylvesuvianite") is proposed for distinguishing fluorvesuvianite. A rule of 50% Cl and a boundary content of 0.5 *apfu* of Cl are proposed for distinguishing "chlorvesuvianite".

Key-words: vesuvianite-group minerals, fluorvesuvianite, chlorine-bearing vesuvianite, structure, microprobe, mineral nomenclature

INTRODUCTION

Minerals of the vesuvianite group are rock-forming minerals with complicated structures and variable compositions. A simplified crystal chemical formula for vesuvianite can be written as $X_{18}X'Y'Y_{12}T_{0-5}(ZO_4)_{10}(Z_2O_7)_4O_{1-2}W_9$ or $Ca_{19}(Al,Mg,Fe,Mn,Ti...)_{13}(B,Al)_{0-5}(SiO_4)_{10}(Si_2O_7)_4O_{1-2}\,(OH,O,F,Cl)_9$ (Arem, Burnham 1969; Arem 1973; Ricklidge et al. 1975; Groat et al. 1992a, b, 1994, 1996, 1998; Ohkawa 1994; Lager et al. 1999; Armbruster, Gnos 2000a, b, c; Galuskin et al. 2003a). Limited isomorphism, on the hydrogarnet scheme $Si^{4+} \rightarrow 4H^{+}$, is observed in isolated Si-tetrahedra in Si-deficient, OH-substituted, boron-bearing vesuvianite from the Wiluy River, Sakha-Yakutia, Russia (Galuskin et al. 2003b). The *X′* and *Y′* sites have half occupation in the vesuvianite structure. Two schemes of cation-vacancy ordering in the *Y′* and *X′* channel sites along the fourfold axes (string ordering) are possible on the unit cell level, which leads to two space groups: *P4/n* and *P4nc* (Allen, Burnham 1992; Ohkawa 1994; Armbruster, Gnos 2000b, c). Two structural forms of tetragonal vesuvianite are

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known — high symmetry (high-temperature) and low symmetry (low-temperature) forms which are distinguished by the sizes of ordered domains (Allen, Burnham 1992; Żabiński, Paluszkiewicz 1994). The vanishingly small size of long-range disordered domains in the "high" vesuvianite defines the space group as *P4/nnc*. Long-range ordering of string-ordered domains in the "low" vesuvianite leads to a reduction of symmetry from *P4/nnc* to *P4/n* or *P4/nc* (Giuseppetti, Mazzi 1983; Fitzgerald et al. 1986; Allen, Burnham 1992; Ohkawa 1994; Pavese et al. 1998; Armbruster, Gnos 2000a, b, c).

The vesuvianite group includes four mineral species: vesuvianite (Werner 1795), wiluite (B > 2.5 *apfu* in the *T* site; Lyakhovich 1954; Groat et al. 1998), manganvesuvianite (Mn^{3+} > 0.5 *apfu* in the *Y′* site; Armbruster et al. 2002) and fluorvesuvianite (F > 4.5 *apfu* in the *W* site; Britvin et al. 2003). Chlorine contents do not usually exceed 0.05 *apfu* in low-temperature vesuvianite from rodingites whereas these contents vary from 0.1 to 0.5 *apfu* in high-temperature fluorine-bearing vesuvianite from skarns (Fitzgerald et al. 1992; Groat et al. 1992; Galuskin et al. 2003a). Chlorine contents reach 0.7–0.8 *apfu* in fluorine enriched vesuvianites in skarn from Ontario in Canada (Groat et al. 1992, 1995) and from Oita in Japan (Ohkawa 1994).

Vesuvianite with the highest recorded contents of Cl (~ 1 *apfu*) was detected in the achtarandite rocks from the Wiluy River, Sakha-Yakutia, Russia (Galuskin 2005). In this paper, we present data on the morphology and composition of chlorine-bearing vesuvianites from the Wiluy occurrence. We discuss the role of chlorine in the vesuvianite structure and the problem of vesuvianite-group classification and nomenclature.

METHODS OF INVESTIGATION

The morphology and composition of the vesuvianite-group minerals was investigated using a FEI/Philips XL30 ESEM scanning electron microscope and a Cameca SX100 microprobe. Natural and synthetic standards were used. The main elements were measured at beam conditions of 15 kV and 20 nA for 20 s at each point. The measurement of boron content was performed at 5 kV and 100 nA for 50–100 s at each point using a danburite standard, following the method described in detail by Galuskin et al. (2003a, b) and Galuskin (2005).

MORPHOLOGY AND COMPOSITION OF CHLORINE-BEARING VESUVIANITES

Systematic investigations of the vesuvianite-group minerals from the well-known Wiluy occurrence, the type locality for grossular, wiluite and scandian garnet (Lyakhovich 1954; Galuskina et al. 1998, 2005; Groat et al. 1998), led to the recognition of vesuvianites with the highest known content of Cl ~ 1 *apfu* (Galuskin 2005). Increased contents of Cl were detected in the late generations of the vesuvianite-group minerals, which formation was connected with low-temperature alterations (rodingitization and serpentinization) of early high-temperature melilite Mg-skarns. These skarns had formed in a giant xenolith of Ordovician calcareous-clay rocks within Triassic gabbro-dolerite of the Siberian Trap Formation (Lyakhovich 1954; Galuskina et al. 1998, 2001; Galuskin et al. 2003b; Galuskin 2005). Cl-bearing vesuvianites in the rodingite-like rocks are associated with garnet (hydrogrossular-andradite series), clinochlore, diopside, calcite and serpentine.

Chlorine concentrates in the <001> and <101> growth sectors in crystals of Si-deficient vesuvianite typically occurring as split forms and spherulites (Figs 1, 2, Table 1; Galuskin et al. 2003b, c; Galuskin 2005). The thin zones of Si-deficient vesuvianite with increased Cl contents are found in zoned wiluite-vesuvianite crystals and their spherulites (Galuskin 2005; Fig. 2, Table 1). Skarn wiluite with high B contents is characterized by low Cl contents (Fig. 2).

The highest Cl contents were detected in the thin zones of small vesuvianite crystals (Figs 2, 3; Table 1) where Cl also concentrates in the <001> and <101> growth sectors (Fig. 3). Interestingly, Cl concentrates both in zones with anomalously high Ti contents (< 2.7 *apfu*) and high Al contents (Fig. 3). Enrichment in Ti characterizes synchronous zones in vesuvianite and garnet (Fig. 3).

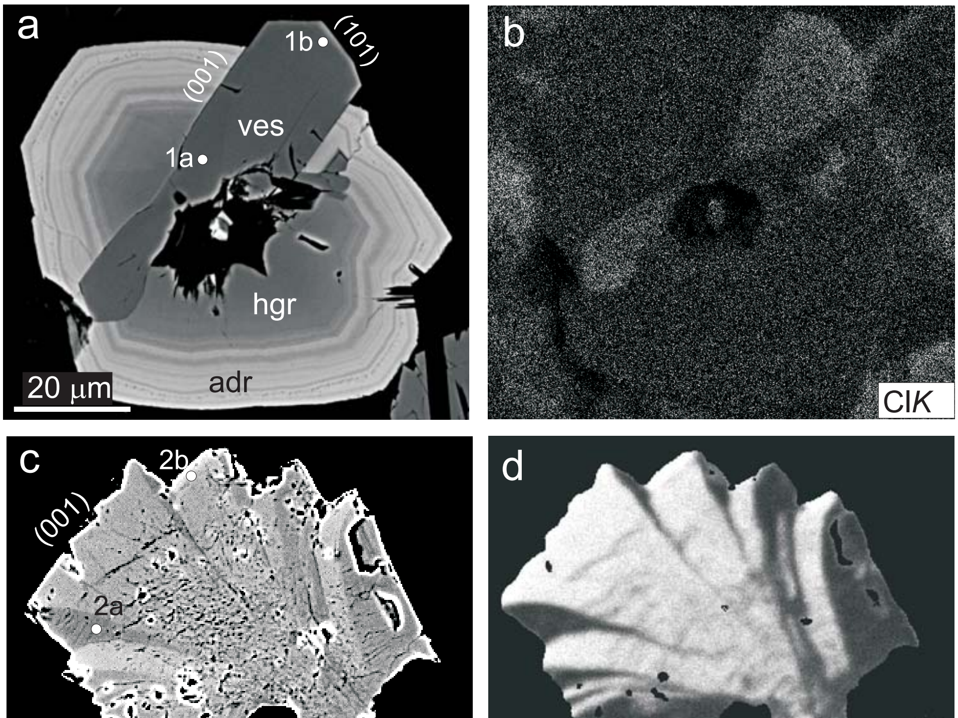

Fig. 1. Si-deficient vesuvianites from Wiluy: a, b — BSE image of {101} + {100} crystal and zoned grossular crystal (a) and sectoral distribution of Cl in the crystal (b); c, d — split crystal: BSE image (c) and distribution of Cl (d); adr — andradite, hgr — hydrogrossular, ves — vesuvianite

TABLE 1

Composition of Cl-bearing vesuvianites from the Wiluy River, Sakha-Yakutia, Russia

Compo-nent [wt.%] / Sector	1a	1b	2a	2b	3	4	5a	5b	6
	<100>	<101>	<100>	<001>	<001>	<001>	<100>	<001>	<001>
SiO_2	32.83	34.35	35.28	36.60	34.37	36.74	35.85	35.44	35.53
TiO_2	0.49	0.80	0.09	1.17	0.20	n.d.	3.93	6.86	5.26
B_2O_3	1.95	1.73	1.87	0.97	1.56	0.82	0.66	0.59	0.81
Al_2O_3	14.56	13.43	15.32	15.30	16.11	16.60	11.85	9.42	10.39
Cr_2O_3	n.d.	0.03	n.d.	n.d.	0.03	n.d.	0.19	0.29	0.06
Fe_2O_3	4.15	4.79	0.61	0.98	2.99	0.89	5.10	4.31	4.47
MnO	0.68	0.41	0.07	0.02	0.12	0.04	n.d.	n.d.	0.04
MgO	3.91	4.21	5.51	5.01	3.87	4.82	3.72	4.10	4.19
CaO	36.56	36.14	36.72	36.17	36.71	37.21	35.60	35.06	35.15
H_2O	3.82	2.80	3.18	1.92	2.83	2.86	1.91	1.44	1.46
F	0.37	0.23	n.d.	n.d.	0.25	0.26	n.d.	0.07	0.24
Cl	0.01	0.43	0.03	0.94	0.65	0.79	0.19	1.20	0.99
–O=F+Cl	0.16	0.19	0.01	0.21	0.25	0.29	0.04	0.30	0.32
Total	99.18	99.15	98.67	98.86	99.88	101.04	99.03	98.47	98.26
Calculated on 78(O+Cl+F) and normalized on 19Ca									
Ca/X	19	19	19	19	19	19	19	19	19
Ti^{4+}	0.18	0.29	0.03	0.43	0.07		1.47	2.61	2.00
Al^{3+}	8.30	7.68	8.72	8.54	8.99	9.24	6.78	5.55	6.12
Cr		0.01			0.01		0.08	0.12	0.02
Mg	2.83	3.08	3.97	3.66	2.79	3.43	2.76	3.09	3.15
Mn^{2+}	0.28	0.17	0.03	0.01	0.05	0.02			0.01
Fe^{3+}	1.52	1.77	0.22	0.36	1.09	0.32	1.91	1.64	1.70
Y	13	13	12.97	13	13	13	13	13	13
Si/*Z*	15.93	16.86	17.03	17.95	16.78	17.51	17.86	17.93	17.92
B	1.64	1.47	1.56	0.82	1.30	0.67	0.57	0.51	0.71
Al	0.13	0.09		0.30	0.18	0.09	0.18	0.07	0.06
T	1.77	1.56	1.56	1.12	1.48	0.76	0.75	0.58	0.77
S^{6+}					0.03		0.03		
OH	12.36	9.17	10.24	6.28	9.12	9.09	6.35	4.86	4.91
F	0.56	0.36			0.38	0.39		0.12	0.38
Cl	0.01	0.35	0.02	0.78	0.53	0.64	0.16	1.03	0.84

1–4 — Si-deficient vesuvianite: 1 — {100}+{101} crystal (Fig. 1a, b); 2 — split {100}+{001} crystal (Fig. 1c, d); 3 — zone (~10 μm) in skeletal growing of vesuvianite on wiluite; 4 — zone (~5 μm) in wiluite-vesuvianite spherulites; 5 (Fig. 3) and 6 — thin zones in small vesuvianite crystals.

All Fe as Fe_2O_3, H_2O calculated on charge balance; n.d. — not detected.

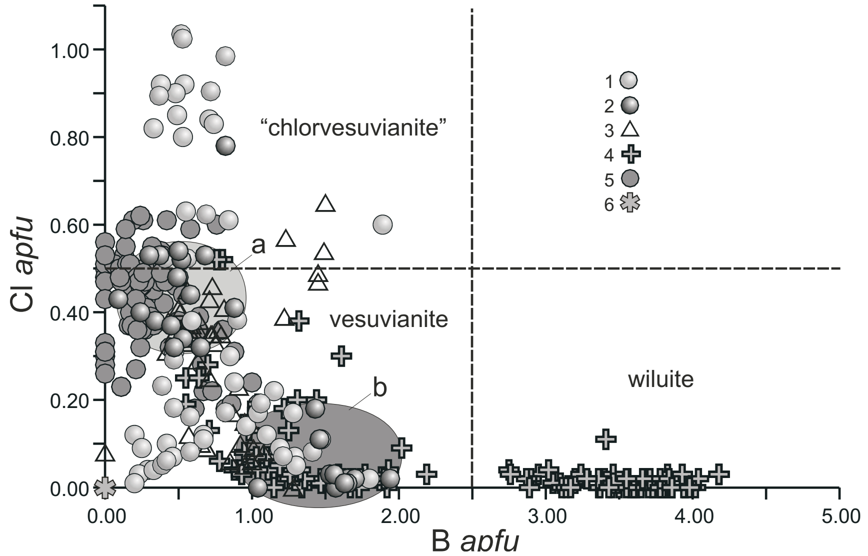

Fig. 2. Vesuvianite analyses plotted on a B *vs* Cl diagram:
1 — small vesuvianite crystals from rodingite-like rocks; 2 — Si-deficient vesuvianite crystal shown in Fig. 1 (a — fields of analyses of <001> sectors enriched in Cl, b — <100> sectors with Cl contents < 0.1 *apfu*); 3 — spherulites of Si-deficient vesuvianite from achtarandite pseudomorphs; 4 — wiluite crystals with late vesuvianite zones; 5 — whiskers of fluorine-chlorine-boron-bearing vesuvianite from Polar Yakutia (Galuskin et al. 2003a); 6 — fluorvesuvianite (Britvin et al. 2003)

Analyses of the vesuvianite-group minerals from Wiluy plot along the O-OH line on the ternary classification diagram O-F(+Cl)-OH [hypothetical "oxyvesuvianite" — fluorvesuvianite — hypothetical "hydroxylvesuvianite", (Fig. 4)] made on the basis of *W* site. In comparison, analyses of fluorine-boron-chlorine-bearing vesuvianite from altered skarn of Polar Yakutia (Galuskin et al. 2003a) plot in the central part of the diagram; some plot in the fluorvesuvianite field. Clorine contents vary from 0.2 to 0.6 *apfu* in vesuvianites from Polar Yakutia (Fig. 2; Galuskin et al. 2003a).

An inhomogeneous (zonal-sectoral) Cl distribution and a prevalence of Cl over F is a common property of Cl-bearing vesuvianites from the rodingite-like rocks of the Wiluy occurrence (Figs 1–3, Table 1). It determines their position in the "hydroxylvesuvianite" field on the classification diagram (Fig. 4). Fluorine prevails significantly over chlorine in all known vesuvianites with high Cl contents (Groat et al. 1992b, 1994; Ohkawa 1994; Galuskin et al. 2003a). The Cl content in a typical sample of fluorvesuvianite from Lupikko, Pitkäranta, Karelia, Russia is lower than the detection limit of microprobe measurements (Britvin et al. 2003).

DISCUSSION

Before discussing the question of Cl in vesuvianite, a digression on the role of fluorine in vesuvianite is appropriate. This topic, though discussed many times

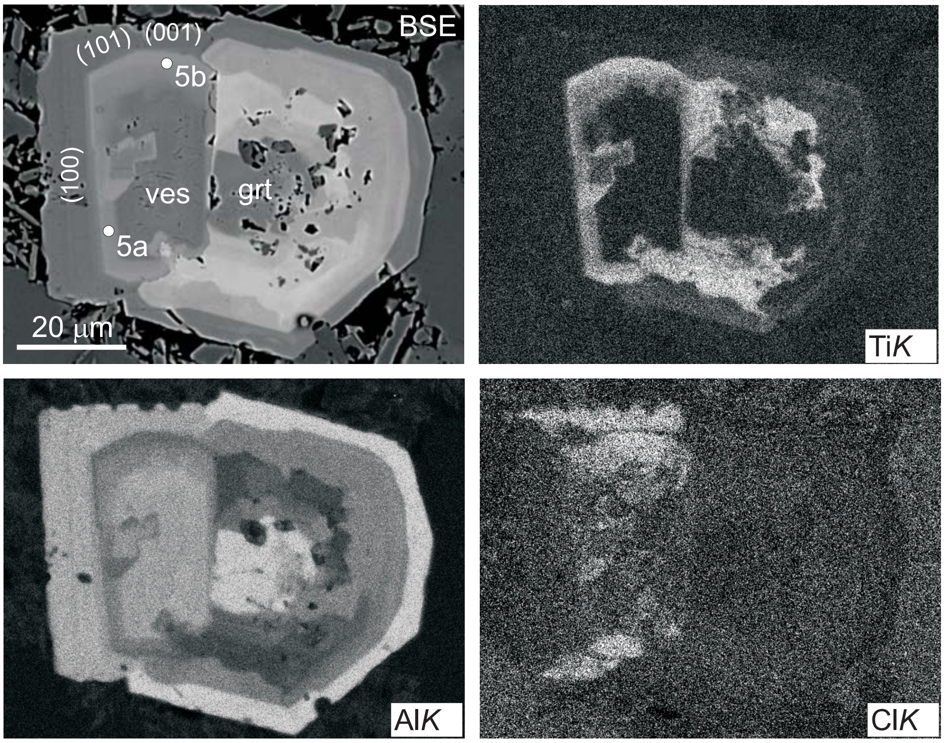

Fig. 3. BSE image and X-ray element maps of a zoned-sectoral vesuvianite crystal (ves) and zoned garnet (grt) intergrowth

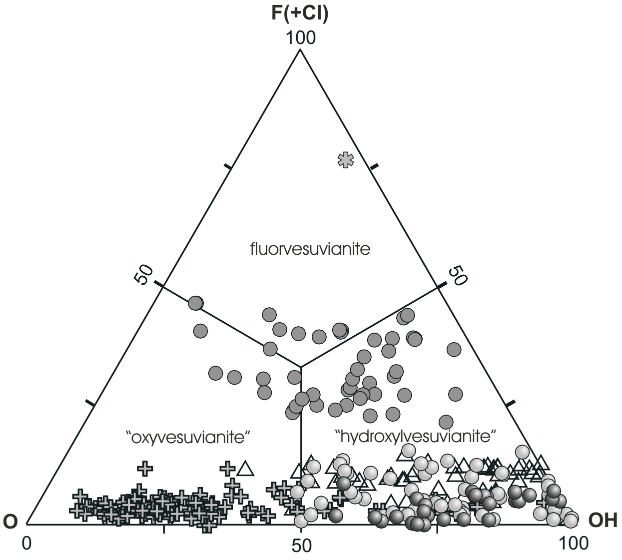

Fig. 4. Vesuvianite compositions plotted on the ternary diagram O ["oxyvesuvianite"] — F(+Cl) [fluorvesuvianite] — OH ["hydroxylvesuvianite"]. For Si-deficient vesuvianite, OH groups participating in the formation of hydrogarnet defects are omitted. For explanation of symbols, see Fig. 2

(Groat et al. 1992a, b; Ohkawa 1994; Galuskin et al. 2003a), merits revisiting after the finding of fluorvesuvianite by Britvin et al. (2003). Fluorine can replace OH groups or O at the 8 *O*(11) sites and at the 2 *O*(10) sites. The two *O*(11) sites and the two *O*(7) sites coordinate the tetrahedral vacancy which can be occupied by B or Al(Fe^{3+}?) [*T*(1) site]. In addition, *O*(11) can participate as a donor and as an acceptor in hydrogen bonding [*H*(1) positions at vacant *T*(1) site] (Groat et al. 1995, 1996; Lager et al. 1999; Armbruster, Gnos 2000a). Though the two *O*(10) sites, separated by about 2.7 Å (Fig. 5a), can be simultaneously occupied by two fluorine atoms, bond-valence analysis and the character of inter-atomic distances for channel sites indicate that, statistically, fluorine can occupy only half of these sites (Groat et al. 1992b; Ohkawa 1994). Hydrogen, asymmetrically sited between two *O*(10) sites, may form only one OH group (Fig. 5a). Thus, F and OH can occupy only one *O*(10) site at most. For this reason, in their definition of fluorvesuvianite, Britvin et al. (2003) proposed 4.5 *apfu* of F as the mineral species boundary. Such a boundary is appropriate for fluorvesuvianite in a binary solid-solution series only, e.g, "hydroxylvesuvianite"-fluorvesuvianite. The proportion of analyses with F contents much less than 4.5 *apfu* place the vesuvianites from Polar Yakutia, with significant contents of "oxyvesuvianite", in the fluorvesuvianite field on the ternary diagram O-F(+Cl)-OH (Fig. 4). Clearly, in this case, the definition of the mineral species should involve the ternary solid-solution series (rule of 33.3%, Nickel 1992).

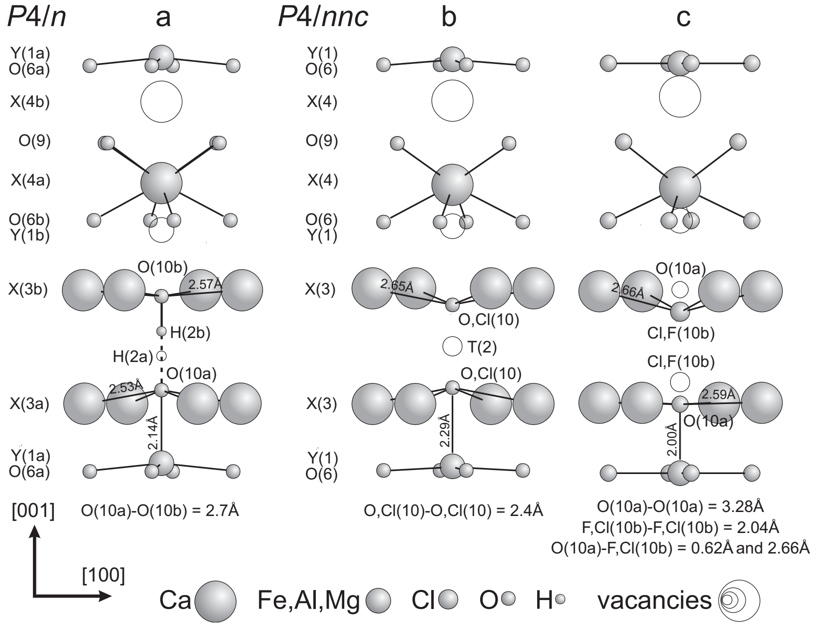

Fig. 5. Positions of the *O*(10) site in vesuvianites with different Cl contents; rods formed by channel positions located along the 4-fold axes are shown:
a — low-temperature *P*4/*n* vesuvianite from Kazakhstan, Cl < 0.01 wt.% (Galuskin et al. in preparation) ;
b — Si-deficient *P*4/*nnc* vesuvianite from Wiluy, Cl ~ 0.2 *apfu* (Galuskin et al. in preparation);
c — fluorine chlor-bearing *P*4/*nnc* vesuvianite from Polar Yakutia, Cl ~ 0.45 *apfu*
(atomic coordinates from Galuskin et al. 2003b)

Chlorine, with an ionic radius significantly larger than that of both F and O, can exceptionally occupy the *O*(10) channel site (Ohkawa 1994; Galuskin et al. 2003a). At Cl contents <0.4–0.5 *apfu*, a high degree of disordering and washing of the *O*(10) site leads to anomalously short (~ 2.4 Å), structurally-refined *O*(10)-*O*(10) inter-atomic distances (Fig. 5b). This effect is accompanied by increases in *O*(10)-*X*(3) inter-atomic distances up to 2.65 Å compared to 2.53–2.57 Å for chlorine-free vesuvianites (Fig. 5a, b). Okhawa (1994) and Galuskin et al. (2003a) showed that the *O*(10) site may be shared by two *O*(10a) and *Cl*(+F)(10b) sites in vesuvianite with Cl contents > 0.4–0.5 *apfu*. A fragment of the structure for chlorine vesuvianite from Polar Yakutia is shown in Figure 5c (Galuskin et al. 2003a). Analysis of the inter-atomic distances indicates that only two combinations of *O*(10a)-*O*(10a) with a distance of 3.28 Å and *O*(10a)-*Cl*(10b) with 2.66 Å are possible. The distance between two *Cl*(10b) sites is 2.04 Å except when both are simultaneously filled (Fig. 5c, Galuskin et al. 2003a).

The maximum theoretical Cl content in vesuvianite is 1 *apfu*. This is 1/9 part of the *W* site used for the definition of fluorvesuvianite and 1/2 of the *O*(10) site. At the *O*(10) site, O, OH, F, and/or Cl may be found; "chlorvesuvianite" is distinguished when Cl prevails (Nickel, Grice 1998). As an accurate evaluation of the distribution of F and OH groups between the *O*(11) and *O*(10) sites is not possible on the basis of microprobe data, the rule of 50% should be used for Cl (Nickel, Grice 1998).

Formally handled, the total anion content at the *O*(10) sites equals 2. "Chlorvesuvianite" can be defined as a mineral species only when the Cl content > 1 *apfu* — more than is theoretically possible. Conversely, the end-member formula of "chlorvesuvianite" which takes account of the sharing of *O*(10) by two different sites will be $X_{18}X'Y'Y_{12}T_{0-5}(ZO_4)_{10}\ (Z_2O_7)_4OClW_8$. In the context of this formula, it may be claimed that 0.5 *apfu* Cl should be a boundary value for the definition of "chlorvesuvianite".

"Chlorvesuvianite" needs further investigation. "Chlorvesuvianite" with 0.87 Cl *apfu*, for which the structure was refined, is that from the Obira mine, Oita, Japan (Ohkawa 1994). Chlorine can probably occupy other sites in the vesuvianite structure. For example, in analysis 5b (Table 1), the Cl content corresponds to 1.03 *apfu* — exceeding the theoretically possible content in the vesuvianite-group minerals. Part of Si-tetrahedra are likely to be substituted by F in fluorvesuvianite according to the scheme $(SiO_4)^{4-} \rightarrow 4F^-$ (Britvin et al. 2003). In wadalite, an unusual zeolite and garnet-like phase, Cl occupies a site which in grossular and vesuvianite is occupied by isolated SiO_4 tetrahedra (Tsukimura et al. 1993; Galuskin 2005).

CONCLUSIONS

The Cl content of vesuvianite can reach a theoretically possible *apfu* ~1 in the <001> growth sector. Vesuvianites with maximum Cl contents also contain minor F and, commonly, high Ti contents. In contrast to fluorvesuvianite, which should be defined using the rule of 33.3% for the *W* site, "chlorvesuvianite" should be distinguished using the rule 50% for half of the *O*(10) site, i.e., when Cl > 0.5 *apfu*. Cl enrichment of the <001>

and <101> sectors during the growth of vesuvianite is connected with structural channels parallel to the [001].

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Streszczenie

Uproszczony wzór krystalochemiczny wezuwianu, skałotwórczego minerału o złożonym składzie i skomplikowanej strukturze, można przedstawić w następujący sposób: $X_{18}X'Y'Y_{12}T_{0–5}$ $(ZO_4)_{10}(Z_2O_7)_4O_{1–2}W_9$ lub $Ca_{19}(Al,Mg,Fe,Mn,Ti...)_{13}(B,Al)_{0–5}(SiO_4)_{10}$ $(Si_2O_7)_4O_{1–2}(OH,O,F,Cl)_9$.

Szczegółowe badania składu i morfologii minerałów grupy wezuwianu z rodingitopodobnych skał i serpentynitów stanowiska wilujskiego, Sacha-Yakucja, Rosja, pozwoliły na stwierdzenie wezuwianów o najwyższych znanych zawartościach Cl około 1 *apfu*. Chlor wzbogaca sektory wzrostu <001> i <101> kryształów Si-deficytowego wezuwianu i wezuwianu „normalnego", niekiedy bogatego w tytan, oraz cienkie późne strefy wezuwianu na wiluicie. Geneza wezuwianów zawierających Cl jest związana z etapem serpentynizacji i rodingityzacji wczesnych magnezowych skarnów melilitowych.

Analiza możliwych podstawień izomorficznych w pozycjach strukturalnych *O*(10) i *O*(11) pokazuje, że w celu wyróżnienia fluorwezuwianu powinna być stosowana zasada 33,3% oraz diagram trójkątny O-F(+Cl)-OH (hipotetyczny człon „oxywezuwian" — fluorwezuwian — hipotetyczny człon „hydroksylwezuwian"), natomiast przy wyróżnieniu „chlorwezuwianu" powinno się uwzględniać tylko pozycję *O*(10). Biorąc pod uwagę, iż maksymalna, teoretyczna zawartość Cl w wezuwianach wynosi 1 *apfu*, autorzy proponują przyjąć wartość 0,5 *apfu*, tzn. 50% połowy pozycji *O*(10), za wartość graniczną pomiędzy hipotetycznym „chlorwezuwianem" a innymi minerałami z grupy wezuwianu.