Compositional trends in manganocolumbite from the Puklice I pegmatite, western Moravia, Czech Republic

Vývoj chemického složení manganocolumbitu v pegmatitu Puklice I, západní Morava, Česká republika (Czech summary)

(3 text-figs.)

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Manganocolumbite occurs in the albite-muscovite-lepidolite unit of the zoned lepidolite pegmatite Puklice I near Jihlava. Its zoned blade-like crystals are embedded in various minerals. The cores show slight variation in Ta/(Ta+Nb) but extensive changes in Mn/(Mn+Fe), whereas the rims display variation in Mn/(Mn+Fe), and extensive Ta/(Ta+Nb) fractionation. Increased amounts of WO_3 (up to 8.96 wt. %; 0.555 W apfu) and TiO_2 (up to 1.64 wt. %; 0.299 Ti apfu) are typical in crystal cores. The substitution $W^{6+}Ti^{4+}Nb^{5+}_{-2}$ is very likely at W and Ti concentration of < 0.2 apfu. A higher degree of fractionation in terms of Mn-Fe and Ta-Nb was observed in manganocolumbite from the lepidolite subunit relative to the muscovite subunit. The fractionation trends of manganocolumbite and overall chemical variation within a single textural-paragenetic unit are comparable with those in other lepidolite pegmatites. No differences in the chemical composition were observed in relation to the host mineral of manganocolumbite.

Key words: manganocolumbite, chemical composition, granitic pegmatite, Puklice, Moravia

Introduction

Members of the columbite-tantalite groups represent the most abundant Nb,Ta-oxide minerals occurring in granitic pegmatites (Černý - Ercit 1985 1989). Their fractionation trends in terms of the Ta/(Ta+Nb) and Mn/(Mn+Fe) ratios differ in various pegmatite types and subtypes and reflect an overall fractionation trend within the parent pegmatite body. They are influenced by several factors, but activity of fluorine and/or F-based complexes in the parent medium seems to be the most important one; in lepidolite pegmatites, high activity of $(R^{1+}F)$ enhances significant Mn-enrichment preceding the bulk of the Ta/(Ta+Nb) fractionation, whereas spodumene or petalite pegmatites characterized by low F-activity display rather simultaneous Fe-Mn and Nb-Ta fractionation, or a significant Ta/(Ta+Nb) fractionation accompanied by only a slight or negligible Mn-enrichment (Černý - Ercit 1985, Wise 1987, Černý 1992, Spilde - Shearer 1992).

In the Puklice I pegmatite, manganocolumbite occurs within a single mica-rich textural-paragenetic unit which shows the mineral composition evolving from a muscovite- to a lepidolite- bearing albitic subunit. Manganocolumbite is hosted by various minerals including quartz, albite, verdelite, rubellite, beryl, lepidolite and pollucite. Consequently, compositional trends within individual crystals could reflect specific conditions during the crystallization within different solidification segments of the albite-muscovite-lepidolite unit.

Geology and occurrence

The Puklice I pegmatite is a representative of the Jihlava pegmatite district, defined by Novák et al. (1992), and briefly characterized by Staněk (1962), Němec (1984, 1989a, 1993) and Černý - Němec (1995). About 35 Li-enriched pegmatite dikes are known in this region (Němec 1993), most of them belonging to the lepidolite subtype (for classification see Černý 1991). Elbaite is a typical subordinate to accessory mineral in all dikes; however, amblygonite-montebrasite, rare petalite (+ secondary spodumene) occur at several localities (Němec 1993).

The Puklice I pegmatite, briefly described by Jaroš (1928), is situated about 2 km E of the village Puklice, and about 7 km SE of the town Jihlava. This area is rich in pegmatites; Němec (1993) recorded at least nine Li-enriched pegmatite dikes within the Puklice region alone (about 6 km^2). The pegmatite examined forms a symmetrically zoned dike up to 3 m thick, with W-trending strike. Contacts with surrounding rocks were not detected due to extensive weathering; the dike probably penetrated the gneiss which surrounds the Jihlava syenite massif.

The following textural-paragenetic units evolved from the contact inwards: (i) granitic unit (Qtz + Pl + Kfs + Bt), (ii) coarse-grained unit (Qtz + Ab + Kfs + Ms + Scl), (iii) graphic unit (Kfs + Qtz) and (iv) blocky K-feldspar unit. Nests of (v) albite-muscovite-lepidolite unit, locally with relics of blocky K-feldspar, occur in central parts of the dike. They display several subunits that differ in their mineral composition and texture. The most abundant coarse-grained albite-muscovite subunit consists of quartz, albite, muscovite, tourmaline (black schorl to green elbaite), garnet ($Sps_{69.3-68.7}$, $Alm_{31.0-30.4}$) and accessory cassiterite, zircon, zinnwaldite, manganocolumbite, apatite, magnetite and löllingite (Staněk 1962, Němec 1983). In the lepidolite subunit, muscovite is substituted by lepidolite, and tourmaline is represented by green and pink elbaite; they are associated with colourless beryl, garnet, rare amblygonite-montebrasite, lacroixite and pollucite-analcime (Teertstra et al. 1995). Other accessory minerals include zircon, cassiterite, manganocolumbite, topaz, and fluorite. A mostly medium-grained subunit (Ab + Lpd ± green to pink elbaite) showing a distinct flow-like preferred orientation is found locally in the centre of the dike; however, textural relationships are ambiguous. Niobium-tantalum oxide minerals were not found in this subunit.

Experimental methods and samples

Electron microprobe analyses of manganocolumbite were carried out on a Cameca Camebax SX-50 instrument in the wavelength-dispersion mode: a beam diameter of 1-2 μm, an accelerating potential of 15 kV, a sample current of 20 (40) nA measured on Faraday cup and a counting time of 20 (50) s were used for Fe, Mn, Sn, Ti, Nb, Ta and for Mg, Ca, Sb, As, Bi, Sc, Zr, U and W, respectively. The following standards were used: manganotantalite (TaMα), $FeNb_2O_6$ (FeKα), $MnNb_2O_6$ (NbLα, MnKα), SnO_2 (SnLα), rutile (TiKα), ZrO_2 (ZrLα), $NaScSi_2O_6$ (ScKα), $MgNb_2O_6$ (MgKα), $BiTaO_4$ (BiMα), mimetite (AsLα), $CaNb_2O_6$ (CaKα), stibiotantalite (SbLα), UO_2 (UMα) and tungsten metal (WMα). Data were reduced using the PAP routine of Pouchou - Pichoir (1985). The normalization on 12 cations and 24 anions per formula unit was used for calculation of Fe^{3+}.

Unit-cell dimensions of manganocolumbite were refined using the CELREF least-squares program (Appleman - Evans 1973) for data obtained on the Philips PW1710 automated powder diffractometer with graphite-monochromatized CuKα radiation at 40 kV and 40 mA. Annealed CaF_2, with a = 5.46379(4) nm was used as an internal standard.

Fragments of manganocolumbite crystals from 12 rock samples were examined, including all paragenetic and textural varieties of the albite-muscovite-lepidolite unit except the central subunit with distinct preferred orientation. Manganocolumbite forms black blade-like euhedral crystals up to 2 mm in size, locally showing a distinct rupture or elastic deformation. Almost all grains examined display simple zoning in the BSE image; a relatively homogeneous core is sharply separated from rims with slight to moderate progressive zoning (see Lahti 1987 for other examples). Oscillatory zoning was observed only sporadically within the rims; so was patchy zoning and younger veining. Manganocolumbite is hosted by various minerals; the complete mineral assemblages are summarized in Table 1. No other Nb, Ta-oxide minerals have been detected within the dike, such as microlite-pyrochlore, fersmite, ixiolite or tantalian rutile described recently from other dikes in the Jihlava pegmatite district (Černý - Němec 1995).

Table 1. Mineral assemblages of examined manganocolumbite samples

Sample	Mineral assemblage substantial	accessory	Host mineral(s)
	Muscovite subunit		
P 12a	Qtz, Ab, Ms, Kfs		Qtz
P 12b	Qtz, Ab, Ms, Kfs	Mag	Qtz
P 3	Qtz, Ab, Ms, Vrd, Srl	Zrn	Qtz, Ab
P 8	Qtz, Ab, Ms, Vrd	Cst	Vrd
P 13	Qtz, Ab, Ms		Ab
	Lepidolite subunit		
P 2	Qtz, Lpd	Zrn	Qtz
P 18	Qtz, Lpd, Ab		Qtz
P 5	Ab, Qtz, Lpd	Toz, Zrn, Cst	Ab, Qtz
P 14a	Qtz, Lpd		Qtz
P 19	Pol, Qtz, Lpd		Pol
P 21	Qtz, Lpd, Ab	Rbl, Brl	Qtz, Lpd
P 6	Qtz, Pol, Lpd	Rbl	Lpd, Rbl

All abbreviations according to Kretz (1983); Vrd - verdelite, Rbl - rubellite, Pol - pollucite

Chemical composition

All analyzed samples correspond to manganocolumbite; however, extensive compositional variation in terms of both Mn/(Mn+Fe) and Ta/(Ta+Nb) ratios are observed (Fig. 1a,b). The crystal cores are characterized by slight variation in Ta/(Ta+Nb) = 0.17-0.08, but extensive variation in Mn/(Mn+Fe) = 0.745-0.992. On the contrary, the rims show slight variation in Mn/(Mn+Fe) = 0.93-1.00 (exept the sample P 12b), but extensive fractionation in terms Ta/(Ta+Nb): 0.15-0.50 (Fig 1b). Younger veining observed in the samples P 12b and P 6 is characterized by distinct increase of the Ta/(Ta+Nb) ratios.

Besides the main components illustrated in the columbite-tantalite quadrilateral (Fig. 1a,b), most of the other constituents show very low concentrations (maxima at 0.011 Ca, 0.004 Mg, 0.011 Y, 0.004 Sb, 0.007 As, 0.008 Bi, 0.012 U, 0.025 Zr and 0.040 Sn apfu); Sc is below the detection limit in all samples. Tungsten and titanium represent the only minor elements showing increased contents, up to 8.96 wt. % WO_3 (0.555 W apfu) and 1.64 wt. % TiO_2 (0.299 Ti apfu). They are concentrated particularly within the crystal cores (Fig. 2), and samples from the muscovite subunit seem to be very slightly enriched in these elements relative to those from the lepidolite subunit. However, the most W-enriched manganocolumbite is enclosed in altered pollucite (sample P 19). No changes in the minor elements were observed within the younger veining.

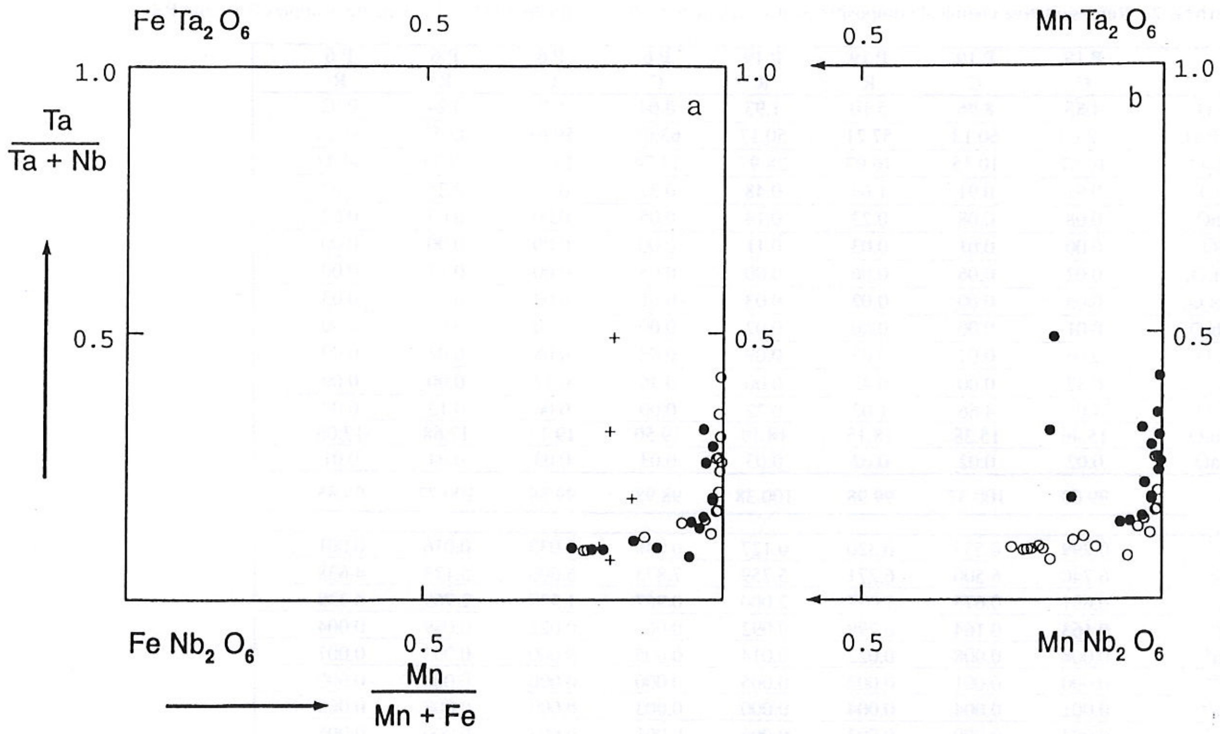

Fig. 1. Compositions of manganocolumbite from the Puklice I pegmatite in the columbite-tantalite quadrilateral
Part a) *dots* - muscovite subunit, *circles* - lepidolite subunit, *crosses* - sample P 12b; part b) *circles* - cores of grains, *dots* - rims.

Representative compositions of manganocolumbite are given in Tables 2 and 3.

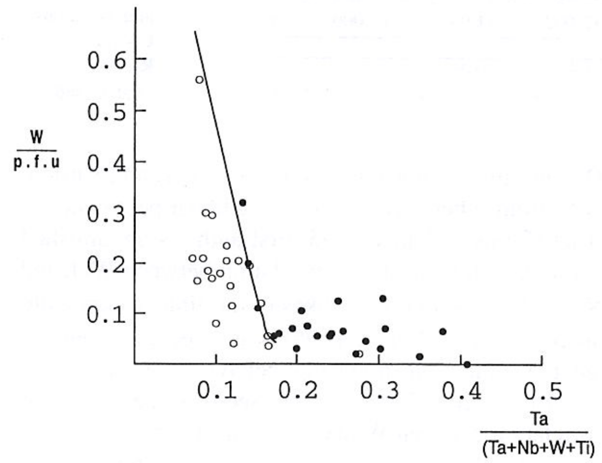

Fig. 2. Plot of W versus Ta/(Ta+Nb+W+Ti) in manganocolumbite
circles - cores of grains, *dots* - rims

X-ray study

The unit cell-dimensions obtained from two samples; a = 14.369(8), b = 5.752(3), c = 5.095(2) nm, and 14.380(7), 5.741(5), 5.103(3), respectively, indicate an intermediate degree of disorder, comparable to that of other columbite-tantalite samples from lepidolite pegmatites of the Moldanubicum (M. Novák - unpublished data).

Discussion

The main compositional trend of columbite from the Puklice I pegmatite perfectly corresponds to those from other lepidolite pegmatites in the Jihlava pegmatite district which do not exhibit contamination from surrounding rocks (Černý - Němec 1995, author's unpublished data) (Fig. 3b). Extreme Mn-enrichment precedes Nb-Ta fractionation; the latter does not reach the field of manganotantalite, which is attained at several other localities of the Jihlava pegmatite district (Fig. 3b). The fractionation trends of columbite from the lepidolite-subtype pegmatites with subordinate elbaite (Himalaya dike system, Mesa Grande, California, Foord 1976 and Sahatany Valley, Madagascar, Ranorosoa 1986) also appear to be almost identical with the Puklice I fractionation trend (Fig. 3a); however, there are more or less significant differences in other lepidolite-subtype pegmatites such as the Silverleaf and Annie Claims, Greer Lake, Manitoba (Černý et al. 1986), Brown Derby, Gunnison County, Colorado (Černý - Ercit 1985) and Bob Ingersol I, Black Hills, South Dakota (Spilde - Shearer 1992) (Fig. 3a). The trend of manganocolumbite from the examined Puklice I lepidolite pegmatite with subordinate elbaite also is almost identical to those established for the elbaite-subtype pegmatites in the Moldanubicum, such as Komárovice, Ctidružice and Vlastějovice (see Fig. 3b, compare Novák - Povondra 1995).

Table 2. Representative chemical compositions of manganocolumbites from the Puklice I pegmatite, samples P 19 and P 6

	P 19 C	P 19 C	P 19 R	P 19 R	P 6 C	P 6 C	P 6 R	P 6 R
WO_3	4.85	8.96	5.10	1.93	0.64	0.50	0.24	0.02
Nb_2O_5	62.60	60.13	57.21	50.17	63.05	59.61	43.52	37.13
Ta_2O_5	10.67	10.35	16.03	28.97	14.74	19.92	38.74	44.43
TiO_2	0.91	0.91	1.64	0.48	0.37	0.12	0.25	0.02
SnO_2	0.08	0.08	0.23	0.14	0.05	0.00	0.03	0.02
UO_2	0.00	0.01	0.03	0.11	0.00	0.00	0.00	0.00
Bi_2O_3	0.02	0.06	0.06	0.00	0.05	0.00	0.12	0.00
As_2O_3	0.03	0.00	0.02	0.03	0.01	0.02	0.00	0.03
Sb_2O_3	0.01	0.00	0.00	0.02	0.00	0.00	0.00	0.00
Y_2O_3	0.06	0.01	0.05	0.09	0.08	0.08	0.02	0.00
$Fe_2O_3^*$	0.87	0.00	0.42	0.00	0.46	0.17	0.00	0.09
FeO	4.05	4.66	1.02	0.22	0.00	0.00	0.13	0.00
MnO	15.46	15.38	18.15	18.19	19.50	19.13	17.68	17.06
CaO	0.02	0.02	0.02	0.03	0.03	0.03	0.04	0.01
tot	99.63	100.57	99.98	100.38	98.98	99.58	100.77	98.85
W^{6+}	0.299	0.555	0.320	0.127	0.040	0.032	0.016	0.001
Nb^{5+}	6.740	6.500	6.271	5.759	7.873	6.600	5.172	4.638
Ta^{5+}	0.691	0.673	1.057	2.000	0.967	1.327	2.769	3.339
Ti^{4+}	0.163	0.164	0.299	0.092	0.067	0.022	0.049	0.004
Sn^{4+}	0.008	0.008	0.022	0.014	0.005	0.000	0.003	0.007
U^{4+}	0.000	0.001	0.002	0.006	0.000	0.000	0.000	0.000
Bi^{3+}	0.001	0.004	0.004	0.000	0.003	0.000	0.008	0.000
As^{3+}	0.004	0.000	0.003	0.005	0.001	0.003	0.000	0.005
Sb^{3+}	0.001	0.000	0.000	0.002	0.000	0.000	0.000	0.000
Y^{3+}	0.008	0.001	0.006	0.012	0.010	0.010	0.003	0.000
Fe^{3+}	0.156	0.000	0.077	0.000	0.083	0.031	0.000	0.018
Fe^{2+}	0.806	0.932	0.207	0.047	0.000	0.000	0.029	0.000
Mn^{2+}	3.118	3.115	3.727	3.912	3.983	3.969	3.937	3.993
Ca^{2+}	0.005	0.005	0.005	0.008	0.008	0.008	0.011	0.003
sum	12.000	11.956	12.000	11.985	12.040	12.002	11.997	12.009
Mn/(Mn+Fe)	0.76	0.77	0.93	0.99	0.98	0.99	0.99	1.00

Normalized on 12 cations and 24 anions; C - core, R - rim, * calculated

All data plotted in the columbite-tantalite quadrilateral (Fig. 1a,b) come from columbite from a single textural-paragenetic unit (zone), although there is a distinct internal fractionation evolution from the muscovite to lepidolite subunit. In general, the compositional range within this unit is comparable with those observed within individual zones in other complex pegmatites such as Peerless, Bob Ingersol I and Tin Mountain, Black Hills, South Dakota (Černý et al. 1985, Spilde - Shearer 1992). However, these large and highly differentiated pegmatites with several distinct zones display a significantly broader overall compositional range in the columbite-tantalite group.

Increased W-contents (up to 8.96 wt. % WO_3, 0.555 W apfu) in most of the manganocolumbite from Puklice I seem to be exceptionally high. Tungsten has not been routinly analysed for untill recently, but, if determined, the WO_3 content in columbite from complex pegmatites is mostly about 1 wt. % or less (v. Knorring - Fadipe 1981, Lahti 1987, Spilde - Shearer 1992). Nevertheless, W-enriched columbite has been reported from Mozambique (v. Knorring - Fadipe 1981), the SW Pamir Mts. (Konovalenko et al. 1982), Meldon aplite, Devonshire (v.Knorring - Condliffe 1984) and particularly from other localities of the Jihlava pegmatite district (Černý - Němec 1995, first author's unpublished data). A general positive correlation between WO_3 and Nb_2O_5 described in other dikes of the Jihlava pegmatite district (Černý - Němec 1995) is also recorded, but detailed examination shows that behaviour of tungsten is rather more complicated. There seems to be a positive correlation between W and Σ Ti, Sn, U; the atomic ratio W/(Ti + Sn + U) is close to 1 for most compositions, where W and/or Ti < 0.2 apfu This fact could indicate the substitution $(W^{6+}Ti^{4+})_1 Nb_{2-}$ in the columbite structure, but restricted to the values lower than 0.2 apfu. The same substitution mechanism was observed in columbite from leucocratic granite at Cínovec (Johan - Johan 1994) and in columbite from pegmatite at Chlumek near Velké Meziříčí (Novák - Čech 1995).

Small but distinct differences in the compositional evolution of columbites from the muscovite and lepidolite subunit are marked by lower Ta/(Ta+Nb) fractionation within the cores during the Mn-enrichment and by lower Mn/(Mn+Fe) ratios in the course

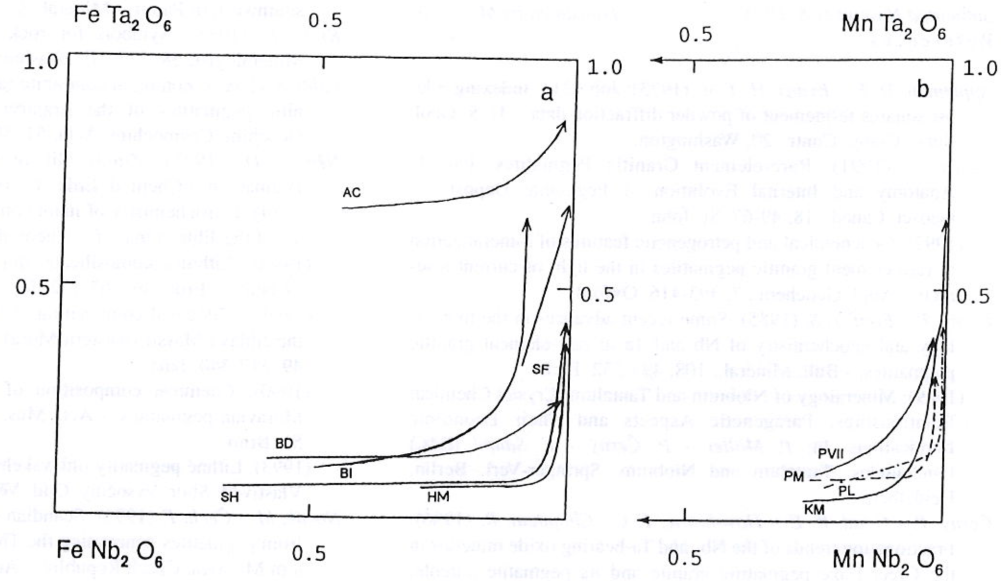

Fig. 3. Compositional trends of columbite-tantalite in the columbite-tantalite quadrilateral
Part a) lepidolite pegmatites (Foord 1976, Černý - Ercit 1985, Černý et al. 1986, Ranorosoa 1986, and Spilde - Shearer 1992). Abbreviations: PM - Puklice I, muscovite subunit; PL - Puklice I, lepidolite subunit; PIV - Puklice IV; PVII - Puklice VII; KM - Komárovice; AC - Annie Claim, Greer Lake, Manitoba; SF - Silverleaf, Greer Lake, Manitoba; BD - Brown Derby, Colorado; BI - Bob Ingersol, Black Hills, South Dakota; SH - Sahatany Valley, Madagascar; HM - Himalaya dike system, California; part b) pegmatites of the Jihlava pegmatite district (this work, Černý - Němec 1995, unpublished data of M. Novák)

Table 3. Chemical composition of manganocolumbite in the sample P 12b

	C	C	R	R	R
WO_3	4.79	3.48	1.56	1.01	0.82
Nb_2O_5	62.39	66.47	53.33	44.09	29.48
Ta_2O_5	11.57	9.00	24.21	33.84	47.73
TiO_2	1.16	1.20	0.66	0.62	0.85
SnO_2	0.11	0.10	0.05	0.14	0.22
UO_2	0.00	0.00	0.00	0.00	0.02
ZrO_2	0.00	0.00	0.00	0.00	0.25
Bi_2O_3	0.00	0.00	0.00	0.02	0.00
As_2O_3	0.02	0.00	0.01	0.02	0.04
Y_2O_3	0.02	0.09	0.04	0.00	0.02
$Fe_2O_3^*$	0.00	0.07	1.06	1.16	2.13
FeO	4.23	3.86	2.05	2.49	1.25
MnO	15.66	16.36	16.08	14.50	13.55
CaO	0.04	0.01	0.00	0.02	0.02
tot	99.99	100.65	99.06	97.91	96.38
W^{6+}	0.296	0.210	0.102	0.070	0.061
Nb^{5+}	6.715	6.996	6.055	5.293	3.848
Ta^{5+}	0.749	0.570	1.654	2.444	3.748
Ti^{4+}	0.208	0.210	0.125	0.124	0.185
Sn^{4+}	0.010	0.009	0.005	0.015	0.025
U^{4+}	0.000	0.000	0.000	0.000	0.001
Zr^{4+}	0.000	0.000	0.000	0.000	0.035
Bi^{3+}	0.000	0.000	0.000	0.001	0.000
As^{3+}	0.003	0.000	0.002	0.003	0.007
Y^{3+}	0.003	0.011	0.005	0.000	0.003
Fe^{3+}	0.000	0.013	0.201	0.231	0.462
Fe^{2+}	0.842	0.752	0.431	0.553	0.303
Mn^{2+}	3.158	3.226	3.421	3.261	3.314
Ca^{2+}	0.010	0.002	0.000	0.006	0.006
sum	11.993	12.000	12.000	12.000	12.000

Normalized on 12 cations and 24 anions; C - core, R - rim
* calculated

of Nb-Ta fractionation of crystal rims (Fig. 1a); nevertheless, the differences are relatively negligible. The sample P 12b displays significant deviation towards Fe-rich compositions (Fig. 1a) with increasing $Fe^{3+}_{calc.}$ and unexplained decreasing of oxide totals from core to rim, respectively (Fig. 1a,b; Table 3); the content of Ti, a typical element indicative of contamination (Černý - Němec 1995), is also increased. This deviation could reflect an influence of Fe-rich fluids with high $f(O_2)$ (Fig. 1a,b, Table 3) from surrounding rocks during the late Ta-enrichment process (veining). However, no other features of contamination have been observed within the Puklice I pegmatite. Apart from the sample P 12b associated with magnetite, there are no differences found in the chemical composition of manganocolumbite which may be relative to a host mineral.

Acknowledgements. Most of the work was done during the first author's tenure of Postdoctoral Fellowship awarded by the Faculty of Sciences, University of Manitoba. The laboratory work was supported by NSERC Research and Major Installation Grants to P. Černý and by NSERC Major Equipment Grants to F. C. Hawthorne, both at University of Manitoba. The authors are indebted to Dr. P. Černý for critical reading of manuscript, and they thank R. Chapman and N.Ball for their assistance in microprobe analysis and X-ray powder diffraction study.

Submitted November 8, 1995 *Translated by M. Novák*

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Vývoj chemického složení manganocolumbitu v pegmatitu Puklice I, západní Morava, Česká republika

Manganocolumbit ze zonálního lepidolitového pegmatitu Puklice I u Jihlavy se vyskytuje v albit-muskovit-lepidolitové jednotce. Jeho zonální tence tabulkovité krystaly jsou zarostlé v různých minerálech. Jádra krystalů vykazují jen nepatrné kolísání poměru Ta/(Ta+Nb), ale výrazný nárůst Mn/(Mn+Fe), okraje pak velmi výrazný nárůst frakcionace Ta/(Ta+Nb), ale téměř stabilní poměr Mn/(Mn+Fe). Zvýšená množství WO_3 do 8,96 váh. % (0,555 W apfu) a TiO_2 do 1,64 váh. % (0,299 Ti apfu), jsou typická pro jádra krystalů i pro hodnoty W a Ti < 0,2 apfu je velmi pravděpodobná substituce $W^{6+}Ti^{4+}Nb^{5+}_{-2}$. Manganocolumbit z lepidolitové subjednotky vykazuje poněkud vyšší stupeň frakcionace Mn/Fe a Ta/Nb ve srovnání s manganocolumbitem muskovitové subjednotky. Trendy frakcionace manganocolumbitu i celkové kolísání jeho chemického složení v jediné texturně paragenetické jednotce jsou srovnatelné s jinými lepidolitovými pegmatity. Nebyl zjištěn žádný rozdíl v chemickém složení manganocolumbitu v závislosti na minerálu, v němž je zarostlý.