The Canadian Mineralogist
Vol. 36, pp. 609-635 (1998)

OXIDE MINERALS OF THE SEPARATION RAPIDS RARE-ELEMENT GRANITIC PEGMATITE GROUP, NORTHWESTERN ONTARIO

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ABSTRACT

The newly discovered Separation Rapids pegmatite group, situated in mafic metavolcanic host-rocks that represent the eastern extremity of the Bird River metavolcanic – metasedimentary belt, contains Ontario's first occurrences of wodginite-group minerals (mainly wodginite and ferrowodginite), the pyrochlore-group minerals stibiomicrolite, stibiobetafite and yttropyrochlore, ferrotapiolite, and probably the first occurrence in North America of nigerite from a granitic pegmatite. This example of the rare-element class of granitic pegmatites hosts both beryl- and petalite-subtype pegmatites. Columbite–tantalite and cassiterite are the predominant oxide species. On the basis of columbite–tantalite compositions, the pegmatites have been divided into an Fe-suite and a Mn-suite. Both beryl and petalite pegmatites occur in each suite. On the basis of ferrocolumbite compositions, the associated Separation Rapids pluton is considered to be the parent of at least the Fe-suite of pegmatites. The Fe suite includes beryl pegmatites within and adjacent to the pluton, in which ferrocolumbite coexists with ferrowodginite, and, with increasing evolution, petalite-bearing pegmatites that contain ferrotantalite and wodginite. In individual pegmatites, columbite–tantalite variation is mainly in Ta/(Ta + Nb). Minor microlite, antimonian microlite and stibiomicrolite are found replacing earlier phases. Cassiterite is the final Nb–Ta-bearing oxide to crystallize. Pegmatites belonging to the Mn-suite follow a similar pattern of crystallization, with early manganocolumbite followed by manganotantalite, the latter coexisting with wodginite. Manganocolumbite within individual samples varies appreciably in Mn/(Mn + Fe), whereas the variation in manganotantalite is mainly in Ta/(Ta + Nb). In pods rich in "cleavelandite" and Li-mica within one of the beryl pegmatites, extreme Mn-enrichment has produced near-end-member manganotantalite and W-bearing wodginite. Microlite is an important late phase, which is either primary or forms as a replacement, mainly of wodginite. The presence of microlite, lithian mica and topaz in Mn-suite pegmatites (and aplites) indicates that they were derived from a more F-rich melt than that which produced the Fe-suite of pegmatites. Albitization also is more apparent in the Mn-suite of pegmatites. The wall zone of Marko's pegmatite, the largest body in the eastern subgroup and part of the Mn-suite, is unique in hosting titanowodginite, "ferrotitanowodginite", stibiobetafite and strüverite. These Fe-, Ti- and Sb-phases are considered to have developed as a result of interaction of the pegmatite-forming melt with banded ironstones and Fe–Ti-rich metavolcanic host-rocks.

Keywords: columbite, tantalite, wodginite, ferrowodginite, titanowodginite, microlite, stibiomicrolite, stibiobetafite, nigerite, granitic pegmatite, tantalum, tin, Separation Rapids, Ontario.

SOMMAIRE

Le groupe de massifs de pegmatites granitiques dit de Separation Rapids, situé dans des roches hôtes mafiques métavolcaniques qui représenteraient l'aboutissement vers l'est de la ceinture métavolcanique et métasédimentaire de Bird River, contient les premières indications de la présence en Ontario des minéraux du groupe de la wodginite (surtout wodginite et ferrowodginite), du groupe du pyrochlore (stibiomicrolite, stibiobétafite et yttropyrochlore), de la ferrotapiolite, et tout probablement du premier exemple en Amérique du Nord de la nigérite dans un contexte pegmatitique. Ces exemples de pegmatites granitiques enrichies en éléments rares comprennent des cortèges à béryl et d'autres à pétalite. Columbite–tantalite et cassitérite en constituent les oxydes principaux. A la lumière des compositions de columbite–tantalite, les pegmatites définissent deux suites, une riche en Fe et l'autre, en Mn. On trouve des pegmatites à béryl et des pegmatites à pétalite dans chaque suite. Selon les compositions de la ferrocolumbite, le pluton de Separation Rapids, avoisinant, serait la source d'au moins la suite de pegmatites riche en Fe, dont les pegmatites à béryl à l'intérieur ou près du pluton contiennent la ferrocolumbite qui coexiste avec la ferrowodginite. Les faciès plus évolués, à pétalite, contiennent ferrotantalite et wodginite. Dans les venues pegmatitiques individuelles, la variation en composition de la columbite–tantalite implique surtout Ta/(Ta + Nb). Des traces de microlite, microlite stibié et stibiomicrolite remplacent en général les minéraux accessoires précoces. La cassitérite est le dernier

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oxyde porteur de Nb–Ta à cristalliser. Les pegmatites faisant partie de la suite enrichie en Mn suivent un schéma de cristallisation semblable, impliquant manganocolumbite suivie de manganotantalite, cette dernière en coexistence avec la wodginite. Dans un échantillon donné, la manganocolumbite varie de façon importante en Mn/(Mn + Fe), tandis que la variation dans la manganotantalite implique surtout Ta/(Ta + Nb). Dans des lentilles riches en "cleavelandite" et en mica lithinifère au sein d'une pegmatite à béryl, un enrichissement extrême en Mn a produit de la manganotantalite dont la composition est proche du pôle et de la wodginite riche en tungstène. Le microlite est une phase tardive importante, soit d'origine primaire ou bien en remplacement, surtout de la wodginite. La présence de microlite, mica lithinifère et topaze dans les pegmatites (et aplites) de la suite riche en Mn laisse entrevoir une cristallisation à partir d'un magma relativement enrichi en fluor comparé à celui qui a produit le cortège de pegmatites plutôt enrichies en Fe. L'albitisation est aussi plus évidente dans les pegmatites riches en Mn. La zone de paroi de la pegmatite de Marko, la plus volumineuse du groupe de l'est et faisant partie de ce groupe riche en Mn, semble la seule à contenir titanowodginite, "ferrotitanowodginite", stibiobétafite et strüverite. Ces phases riches en Fe, Ti et Sb seraient attribuables à une interaction entre le magma responsable de la pegmatite et les roches encaissantes, des formations de fer rubanées et des roches métavolcaniques riches en Fe–Ti.

(Traduit par la Rédaction)

Mots-clés: columbite, tantalite, wodginite, ferrowodginite, titanowodginite, microlite, stibiomicrolite, stibiobétafite, nigerite, pegmatite granitique, tantale, étain, Separation Rapids, Ontario.

INTRODUCTION

Zoned rare-element (*e.g.*, Be, Li, Nb, Ta, Sn) pegmatites represent the extremes of fractional crystallization of more primitive granitic magmas (Goad & Černý 1981, Shearer *et al.* 1992, Breaks & Moore 1992) and can give rise to major economic deposits of these elements. For instance, the Greenbushes pegmatite of Western Australia contains half the world's Ta resource (Partington *et al.* 1995), whereas other pegmatites such as Tanco, Manitoba (Crouse *et al.* 1979) and Bikita, Zimbabwe (Cooper 1964) are major Li producers. However, there are few examples where all components of the granite – pegmatite sequence are exposed, and consequently the processes of differentiation are poorly documented (with a few notable exceptions; *e.g.*, Greer Lake, Manitoba: Černý *et al.* 1986; Harney Peak, South Dakota: Shearer *et al.* 1992; Preissac – Lacorne, Quebec: Mulja *et al.* 1995). Specific factors responsible for the concentration of the rare metals still remain largely unknown, although this is as much a problem of experimental geochemistry as it is of field exposure.

The Separation Rapids Pegmatite Group offers the unusual opportunity to examine one of the chemically and mineralogically most evolved rare-element pegmatite groups *and* its potential source, the Separation Rapids pluton. In this paper, we document an electron-microprobe investigation of the oxide mineralogy, in particular the Nb–Ta oxides, as these have been shown to be especially responsive to fractionation and replacement effects (Černý *et al.* 1985, 1986, Abella *et al.* 1995, Černý & Němec 1995). The geology, geochemistry and K-feldspar mineral chemistry of the Treelined Lake granitic complex (the likely source of the Separation Rapids pluton) and the Separation Rapids Pegmatite Group will be described by Breaks *et al.* (in prep.), and details of the wodginite-group mineral chemistry are covered in a companion paper (Tindle *et al.* 1998).

TABLE 1. SEPARATION RAPIDS MINERALOGY

	1	2	3	4	5	6	7
Fe-columbite	•	•	•	•			(•)
Fe-tantalite			(•)	•			
Mn-columbite		(•)			•	•	•
Mn-tantalite		(•)			•	•	•
Fe-wodginite			•	•			(•)
Wodginite				(•)	•	•	•
FeTi wodginite							•
Ti wodginite							•
Microlite				•	•	•	•
Stibiomicrolite				•			•
Bismutomicrolite					(•)		
Uranmicrolite				•			
Stibiobetafite							(•)
Yttropyrochlore				(•)			
Ferrotapiolite			•				
Strüverite							•
Cassiterite	•	•	•	•	•	•	•
Ilmenite		(•)					
Scheelite				(•)			
Uraninite				•			
Gahnite	•	•	•				
Nigerite				•			
Sphalerite		•					
Pyrite		•	• ?	•		•	
Löllingite				•		•	
Arsenopyrite		•	• ?	•	• ?	•	•
Chalcopyrite		•	•	•			
Fluorapatite		•	•	•	•	•	•
Monazite		•	•	•		•	
Xenotime			•	• ?			
Alluaudite		•					
Purpurite				•			
Zircon		•	•	•		•	
Allanite		•					
Epidote		•		•		•	
Fluorite		•	• ?		•		
Topaz	•				•		
Beryl		•	•	•	•	•	•
Tourmaline	•	•		•	•	•	• ?
Garnet	•	•	•	•		•	
Cordierite	•	•					
Amphibole		•					
Biotite	•	•	(•)		• ?		
Zinnwaldite		• ?	• ?	• ?	• ?	• ?	
Li Mica		•	•	•	•	•	•
Muscovite	•	•	•	•	•	•	•
Albite	•	•	•	•	•	•	•
K feldspar	•	•	•	•	•	•	•
Quartz	•	•	•	•	•	•	•
Spodumene						•	
Petalite				•		•	
Pollucite				• ?		• ?	•

1 = Treelined Lake granitic complex, a potential source of the Separation Rapids pluton - also contains orthopyroxene, andalusite, anatase and rutile;
2 = Separation Rapids pluton; 3 = Beryl pegmatites (Fe-suite);
4 = Petalite pegmatites (Fe-suite); 5 = Beryl pegmatite (Mn-suite);
6 = Petalite pegmatites (Mn-suite);
7 = Wall zone of Marko's pegmatite - a Petalite pegmatite (Mn-suite).
(•) implies only found in one sample; ? questionable

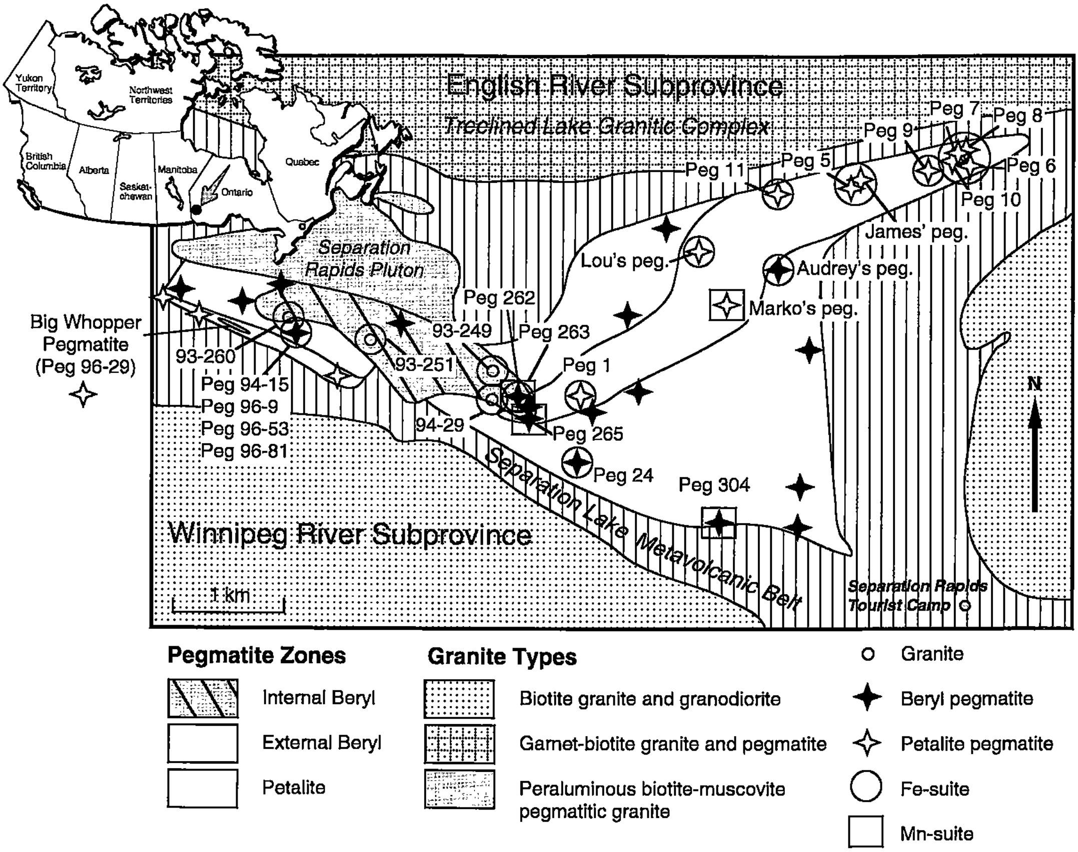


FIG. 1. General geology and location of rare-element mineral samples of the Separation Rapids Pegmatite Group.

THE SEPARATION RAPIDS PLUTON

The 4 km^2 Separation Rapids pluton (2643 ± 2 Ma, unpubl. U/Pb monazite age; Y. Larbi & R.K. Stevenson, GEOTOP Laboratory, Université du Québec à Montréal) is a fertile, peraluminous S-type granite dominated by distinct pegmatitic granite units that largely envelop a core of coarse-grained, K-feldspar porphyritic, garnet – muscovite – biotite granite. It is comparable in size and constituent granitic units to the fertile, peraluminous Greer Lake pegmatitic granite pluton, situated 55 km west–northwest in the Winnipeg River pegmatite district of southeast Manitoba (Černý *et al.* 1981). Similarities include the presence of cordierite, beryl, cassiterite and ferrocolumbite, and the primary layering and mineral assemblages of the associated pegmatites.

Notable petrographic features of the Separation Rapids pluton include: (1) widespread layering of pegmatitic leucogranite, sodic aplite, potassic pegmatite and coarse-grained granitic units, (2) beryl – garnet – muscovite – biotite pseudomorphs after cordierite megacrysts, (3) metasomatic reaction with widespread amphibolite enclaves, (4) sporadic ferrocolumbite and beryl in potassic pegmatite units, and gahnite and Cd-rich sphalerite in a foliated biotite – muscovite – garnet granite unit, (5) green muscovite – "cleavelandite" masses (pods) probably of replacement origin, hosting manganocolumbite, manganotantalite and alluaudite, $NaCaFe^{2+}(Mn,Fe,Mg)_2(PO_4)_3$, within potassic pegmatite units. On the basis of K-feldspar compositions and other geochemical evidence (Fig. 1, Table 1, columns 1 and 2), Breaks *et al.* (in prep.) suggest that the Separation Rapids pluton was derived from the Treelined Lake granitic complex, which outcrops immediately to the north.

REGIONAL PEGMATITE ZONATION

The Separation Rapids Pegmatite Group (Fig. 1) comprises two distinct pegmatite clusters (eastern subgroup, 7.5 km^2, and southwestern subgroup, 2.5 km^2) that are spatially related to the Separation

Rapids pluton and hosted by supracrustal rocks of the Separation Lake Metavolcanic Belt (Blackburn *et al.* 1992, Blackburn & Young 1994). The belt correlates with the 2.74 Ga Bird River Greenstone Belt of Manitoba (Timmins *et al.* 1985), the host of many rare-element pegmatites, including the Tanco pegmatite (Černý *et al.* 1981). On the basis of a striking similarity in geological setting, mineral assemblages and emplacement age with the various rare-element pegmatite groups commencing 40 km west in Manitoba, we consider that the Separation Rapids Pegmatite Group constitutes the eastern limits of the Cat Lake – Winnipeg River Pegmatite Field.

The southeastern margin of the Separation Rapids pluton and the proximal eastern subgroup pegmatites (Fig. 1; Peg. 1, Lou's, Marko's and Audrey's pegmatites) lie within the core and along the eastern flanks of the west-plunging Separation Narrows antiform (Blackburn & Young 1994). Pegmatites in the northeastern part of this subgroup (pegmatites 5–10 and James' pegmatite) are more deformed and hosted within a 100-m-wide high-strain zone in which the primary structural controls on pegmatite emplacement are uncertain. The southwestern pegmatite subgroup is a more recent discovery which comprises five relatively large petalite lenses and swarms of much thinner pegmatites, mostly 1 to 10 m in tickness, that occur *en échelon* to these lenses.

Three regional pegmatite zones have been recognized (Breaks 1993, Breaks & Tindle 1996). The first is a beryl (–columbite) zone within the southern half of the Separation Rapids pluton, beyond which there are two zoned pegmatite swarms that respectively extend east to northeast and southwest of the pluton. The external pegmatites are defined by an outer beryl (– columbite – cassiterite) zone that partially envelops a central petalite (– beryl – cassiterite) zone in the case of the eastern subgroup, whereas in the southwestern subgroup there is a systematic change from a beryl (– columbite – cassiterite) zone into a narrow petalite zone with increasing distance from the parent granite (Fig. 1).

The internal beryl zone contains sodic pegmatites in which green beryl, ferrocolumbite and cassiterite are important accessory phases. The external beryl zone comprises muscovite and biotite–muscovite-bearing sodic pegmatites and related aplites. They have essentially the same mineralogy as the interior pegmatites, and the two have therefore been grouped together in Table 1 (column 3).

Exceptional pegmatites within the beryl zone include a topaz-rich pegmatite (Peg. 304) and a layered, 8-m-thick fluorapatite – garnet – biotite – muscovite sodic pegmatite (Peg. 265) containing Li-mica-bearing "cleavelandite"-rich pods up to 1 by 10 m with minor white beryl, dark green tourmaline, purple lithian muscovite, cassiterite, microlite and wodginite. These pegmatites (and Peg. 263) contain no ferrocolumbite, but instead contain manganocolumbite or manganotantalite (or both). Table 1, column 5 summarizes the mineral assemblage in pegmatite Peg. 265.

The petalite zone comprises 19 petalite- and ferrowodginite-bearing internally zoned pegmatites approximately 1 meter in thickness or greater (11 in the eastern subgroup, 7 in the southwestern subgroup), hosting accessory beryl, cassiterite, microlite, ferrocolumbite, ferrotantalite, with rare nigerite, stibiomicrolite, uranmicrolite and yttropyrochlore occurring in some samples. In two of these pegmatites (Pegs. 5 and 7), albite-rich replacement patches within or cross-cutting large petalite crystals contain tungsteniferous wodginite (WO_3 in the range 2.4–17.3 wt %). The full mineral assemblage from these pegmatites is listed in Table 1, column 4.

One of the petalite-bearing pegmatites from the eastern subgroup (Marko's pegmatite) differs from the others in containing manganocolumbite, manganotantalite and wodginite rather than the "ferro-" varieties (Table 1, column 6). It is also the largest of the eastern subgroup pegmatites, at 8 by 130 m, and contains a well-developed internal zonation of five primary pegmatite units dominated by a blocky K-feldspar – petalite core mantled by a wall zone of beryl + muscovite + albite + quartz. Oxide minerals include cassiterite, wodginite, microlite and manganocolumbite – manganotantalite, which occur in all units, especially where secondary beryl – muscovite – albite units replace parts of the central petalite-rich core.

The wall zone of Marko's pegmatite hosts a unique mineral assemblage (Table 1, column 7) headed by stibiobetafite, stibiomicrolite and a wide range of wodginite species. Rarest of these is a Fe–Ti-rich mineral described by Ercit *et al.* (1992) as hypothetical "ferrotitanowodginite" on the basis of two samples too small to be adequately characterized. Titanowodginite, ferrowodginite and wodginite are also found in the wall zone of Marko's pegmatite. The Ti-rich wodginite species have been described previously only from the Tanco pegmatite, Manitoba (Ercit 1986, Ercit *et al.* 1992). Manganocolumbite, manganotantalite, microlite and strüverite (tantalian rutile) are the other Nb–Ta-bearing phases present.

Several large pegmatites comprise the petalite zone of the southwestern subgroup and vary in surface dimensions from 1 to 10 meters to the 60 by 450 meter Big Whopper Pegmatite, the latter being the largest complex-type pegmatite yet discovered in Ontario (Breaks & Tindle 1996, 1997).

Disseminated throughout the Separation Rapids Group pluton and pegmatites is a very minor sulfide assemblage, of which löllingite, arsenopyrite and Cd-rich sphalerite are perhaps the most notable.

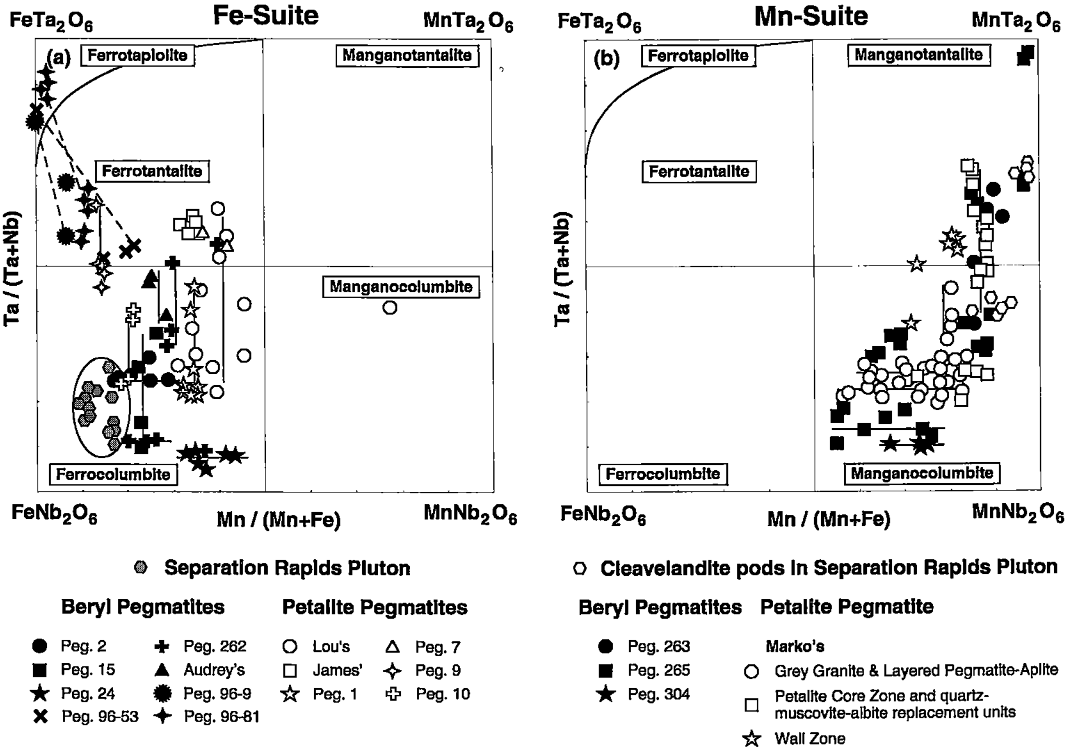


FIG. 2. Covariation of Mn/(Mn + Fe) *versus* Ta/(Ta + Nb) (at.) for (a) ferrocolumbite, ferrotantalite and ferrotapiolite from Fe-suite granitic pegmatites and the Separation Rapids pluton, and (b) manganocolumbite and manganotantalite from Mn-suite pegmatites. Solid vertical and horizontal lines indicate variation within individual samples. Dashed lines join coexisting ferrotapiolite – ferrotantalite pairs.

OXIDE MINERALOGY

Experimental methods

Mineral analyses were obtained at the Open University using a Cambridge Instruments Microscan 9 electron microprobe equipped with two wavelength-dispersion spectrometers. An operating voltage of 20 kV and a probe current of 30 nA (measured on a Faraday cage) were used. Further details of the methods can be found in Tindle *et al.* (1998). In addition, back-scattered electron images and further quantitative data (obtained under essentially identical conditions) were collected on a Cameca SX100 microprobe, also at the Open University. As most of this work was completed before the discovery of the southwestern pegmatite group, the majority of the data can be assumed to be from the eastern pegmatite subgroup unless otherwise specified.

Columbite–tantalite group

Columbite–tantalite is the "classic" Nb, Ta-bearing rare-element species in rare-element pegmatites, and at Separation Rapids it is perhaps the most useful monitor of fractionation effects. It is widely distributed, occurs in all rock types, and only the limitations of the sampling method (two polished thin sections or mineral separates were examined from each sample) are thought to have prevented it from being observed in all samples. Its modal abundance is highest in the distal petalite pegmatites, in particular in the early-crystallized part of Marko's pegmatite. It usually crystallizes earlier and occurs in lesser amounts than cassiterite.

In the Separation Rapids pluton, columbite–tantalite (Fig. 2a, Table 2, compositions 1 and 2) with primitive ferrocolumbite composition prevail [$0.1 < Mn/(Mn + Fe) < 0.2$, $0.1 < Ta/(Ta+Nb) < 0.3$]. They form small, subhedral, inclusion-free, lath-shaped or tabular crystals (0.2–0.6 mm in length) included within pale brown Li-rich mica (zinnwaldite?) or occasionally in zircon or quartz. Crystal size occasionally reaches 10 mm across. In one sample (96–260) from the southwestern margin of the pluton, muscovite–"cleavelandite" pods, containing alluaudite and considered to be of replacement origin, host manganocolumbite and manganotantalite (Fig. 2b).

TABLE 2. COLUMBITE-GROUP MINERALS FROM SEPARATION RAPIDS

	Separation Rapids Pluton		Fe-Suite Beryl Pegmatites				Fe-Suite Petalite Pegmatites				Fe-Suite Petalite Pegmatites (Sc-rich)		Mn-suite Beryl Pegmatites				Mn-suite Petalite Pegmatites				Wall Zone Marko's Pegmatite	
			Internal		External																	
	93-249	93-251	Peg. 262	Peg. 2	Peg. 24	Audrey's	Peg. 9	Peg. 10	James'	Peg. 7	Peg. 7	Lou's	Peg. 304	Peg. 265			Marko's				Marko's	
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
CaO	0.03	0.01	0.04	0.03	0.03	0.16	0.00	0.12	0.05	0.38	0.08	0.05	0.00	0.37	0.04	0.00	0.06	0.02	0.19	0.05	0.07	0.15
FeO	17.52	16.70	15.40	14.45	13.22	12.33	14.33	13.60	11.01	10.35	12.96	3.94	4.91	8.43	2.20	0.49	5.52	4.14	1.84	2.31	3.21	3.33
MnO	2.04	3.09	4.53	4.79	7.13	4.09	2.46	3.66	5.51	5.74	3.19	11.93	14.86	10.04	15.83	13.12	12.89	13.48	13.48	13.22	13.23	13.02
TiO2	0.64	0.57	0.57	1.27	1.15	1.70	0.99	1.20	1.95	1.09	2.81	4.83	1.98	0.54	0.29	0.11	0.91	3.62	1.64	1.22	1.06	1.46
Nb2O5	59.46	63.58	65.47	51.17	67.43	31.85	31.37	39.22	24.09	23.77	26.99	35.74	63.65	58.32	45.49	2.47	51.45	47.47	22.79	18.34	25.50	26.48
Ta2O5	18.77	15.99	13.13	27.47	7.44	48.16	49.62	40.29	53.42	52.97	50.43	40.23	12.53	19.75	36.47	82.99	28.68	29.72	57.72	64.42	56.07	53.38
SnO2	0.02	0.02	0.02	0.24	0.02	0.56	0.33	0.47	2.00	2.29	1.86	1.07	0.18	0.11	0.08	0.77	0.25	0.83	1.24	0.56	0.37	0.45
WO3	0.45	0.37	0.53	0.40	0.55	0.15	0.68	0.84	0.84	2.15	0.01	0.21	0.68	0.44	0.34	0.13	0.50	0.57	0.49	0.00	0.34	0.51
PbO	0.25	0.30	0.30	0.22	0.32	0.37	0.32	0.41	0.13	0.15	0.27	0.30	0.62	0.84	0.15	0.12	0.25	0.49	0.25	0.12	0.15	0.24
ThO2	0.00	0.00	0.00	0.01	0.01	0.02	0.02	0.00	0.00	0.00	0.00	0.00	0.04	0.00	0.00	0.00	0.00	0.01	0.00	0.02	0.01	0.00
UO2	0.00	0.00	0.00	0.03	0.02	0.10	0.11	0.11	0.00	0.14	0.08	0.10	0.73	0.60	0.00	0.02	0.19	0.37	0.00	0.08	0.09	0.24
Sb2O3	-	-	-	0.05	-	0.08	0.08	0.06	-	0.00	0.05	0.08	-	0.03	-	0.18	0.05	-	0.00	0.06	0.08	0.23
Bi2O3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.05	-	-	-	-	-	-
Sc2O3	-	-	-	0.35	-	0.49	0.05	0.03	-	0.24	0.80	1.30	0.02	0.02	-	0.11	0.10	0.05	0.06	0.02	0.05	0.02
Total	99.18	100.63	99.99	100.48	100.09	100.06	100.36	100.01	99.00	99.27	99.53	99.78	100.18	99.49	100.89	100.22	100.85	100.77	99.70	100.40	100.23	99.51
No. of oxygens	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6
Ca	0.002	0.001	0.003	0.002	0.002	0.012	0.000	0.009	0.004	0.030	0.006	0.004	0.000	0.025	0.003	0.000	0.004	0.001	0.015	0.004	0.005	0.012
Fe	0.899	0.833	0.765	0.758	0.639	0.714	0.838	0.763	0.668	0.631	0.762	0.216	0.243	0.436	0.120	0.034	0.290	0.217	0.112	0.145	0.193	0.200
Mn	0.106	0.156	0.228	0.254	0.349	0.240	0.146	0.208	0.339	0.354	0.190	0.664	0.745	0.526	0.872	0.927	0.686	0.716	0.834	0.839	0.808	0.793
Ti	0.030	0.026	0.025	0.060	0.050	0.089	0.052	0.061	0.106	0.060	0.149	0.239	0.088	0.025	0.014	0.007	0.043	0.171	0.090	0.069	0.057	0.079
Nb	1.650	1.715	1.757	1.451	1.833	0.997	0.992	1.190	0.790	0.783	0.858	1.061	1.704	1.630	1.337	0.093	1.461	1.346	0.753	0.621	0.831	0.860
Ta	0.313	0.259	0.212	0.469	0.118	0.907	0.944	0.735	1.054	1.049	0.964	0.719	0.202	0.332	0.645	1.883	0.490	0.507	1.147	1.312	1.099	1.043
Sn	0.000	0.000	0.000	0.006	0.000	0.015	0.009	0.013	0.058	0.067	0.052	0.028	0.004	0.003	0.002	0.026	0.006	0.021	0.036	0.017	0.011	0.013
W	0.007	0.006	0.008	0.007	0.008	0.003	0.012	0.015	0.016	0.041	0.000	0.004	0.010	0.007	0.006	0.003	0.008	0.009	0.009	0.000	0.006	0.009
Pb	0.004	0.005	0.005	0.004	0.005	0.007	0.006	0.007	0.003	0.003	0.005	0.005	0.010	0.014	0.003	0.003	0.004	0.008	0.005	0.002	0.003	0.005
Th	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.001	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
U	0.000	0.000	0.000	0.000	0.000	0.002	0.002	0.002	0.000	0.002	0.001	0.001	0.010	0.008	0.000	0.000	0.003	0.005	0.000	0.001	0.001	0.004
Sb	-	-	-	0.001	-	0.002	0.002	0.002	-	0.000	0.001	0.002	-	0.001	-	0.006	0.001	0.000	0.000	0.002	0.002	0.007
Bi	-	-	-	0.000	-	-	-	-	-	-	-	-	-	-	-	0.001	-	-	-	-	-	-
Sc	-	-	-	0.019	-	0.030	0.003	0.002	-	0.015	0.049	0.074	0.001	0.001	-	0.008	0.005	0.003	0.004	0.000	0.003	0.001
Total	3.011	3.001	3.003	3.031	3.006	3.017	3.006	3.006	3.038	3.034	3.039	3.017	3.019	3.006	3.000	2.990	3.002	3.004	3.005	3.012	3.020	3.026
Mn/(Mn+Fe)	0.105	0.158	0.230	0.251	0.353	0.251	0.148	0.214	0.336	0.360	0.200	0.754	0.754	0.547	0.879	0.964	0.703	0.767	0.881	0.853	0.807	0.798
Ta/(Ta+Nb)	0.160	0.131	0.108	0.244	0.061	0.476	0.488	0.382	0.572	0.573	0.529	0.404	0.106	0.169	0.325	0.953	0.251	0.274	0.604	0.679	0.569	0.548
Comments:	"-" = not analysed										Inclusions in cassiterite											

The Separation Rapids pegmatites have columbite and tantalite compositions that define two discrete groups, an Fe-suite and a Mn-suite. This grouping does not equate with beryl and petalite pegmatites, as both types occur in both groups. Indeed, there is no spatial discrimination between the groups, and members from each suite are mixed together in the field. Compositions of ferrocolumbite and ferrotantalite from selected beryl and petalite pegmatites of the eastern subgroup are listed in Table 2.

The *Fe-suite* contains the Separation Rapids pluton, all the internal beryl pegmatites, all but three of the eastern subgroup beryl pegmatites and all but one of the eastern subgroup petalite pegmatites. Apart from three beryl pegmatites discussed later in the ferrotapiolite section, columbite – tantalite from the southwestern subgroup pegmatites are not considered here, but preliminary results indicate little difference between the two subgroups, with Fe- and Mn-suites being represented in both regions. Tabular or elongate laths are most common, with grain size usually in the range 0.05–0.8 mm, but larger subhedral to anhedral crystals (up to 1.5 cm) are also found in both beryl and petalite pegmatites. Although most crystals are subhedral, it is unusual to find terminated crystals, as most have rounded ends, perhaps indicating minor resorption prior to final crystallization. Further evidence for resorption and crystallization is provided in Figure 3a, where the complex zonation observed is most likely to have developed as a result of these processes. Such patterns of zonation can, however, be generated if only a shallow subsurface of a crystal is revealed during sample preparation (P. Černý, pers. commun.). Inclusions are uncommon, and there is little evidence of alteration other than at the thin rim of some crystals. Ferrocolumbite – ferrotantalite may cluster with monazite or cassiterite, or occur as inclusions in garnet or cassiterite. More commonly, it occurs as inclusions in or around mica (both muscovite and zinnwaldite?), but its main occurrence is in the fine-grained albite-rich parts of the pegmatites, particularly within petalite-bearing pegmatites.

There is a clear relationship between ferrocolumbite from the Separation Rapids pluton and pegmatites from this suite. The internal beryl pegmatites contain ferrocolumbite (Fig. 2a), with compositions [Mn/(Mn + Fe) in the range 0.2–0.3], intermediate between those of the Separation Rapids pluton and the external beryl pegmatites [Mn/(Mn + Fe) in the range 0.3–0.4]. The ratio Ta/(Ta + Nb) is more variable; columbite from the external beryl pegmatites spans the range 0.08–0.48. The petalite pegmatites predominantly contain ferrotantalite [Mn/(Mn + Fe) in the range 0.15–0.4, Ta/(Ta + Nb) in the range 0.5–0.65], but ferrocolumbite with Ta/(Ta + Nb) values as low as 0.2 also occur in the petalite pegmatite closest to the Separation Rapids pluton. Individual pegmatites have limited variation in Mn/(Mn + Fe), but variation in Ta/(Ta + Nb) can be more pronounced and is responsible for the vertical evolution trends on Figure 2a. Horizontal variation within samples on Figure 2a is mainly from two bodies of beryl pegmatite with low Ta/(Ta + Nb) values (Pegs. 24, and part of 262).

In pegmatite Peg. 7, there appear to be two generations of ferrotantalite, an early generation occurring as inclusions in cassiterite (possibly ixiolite because of unusually high Sc and Ti contents: Table 2, composition 11), and a later, euhedral, partially altered generation that forms elongate terminated prismatic crystals (Fig. 3b, Table 2, composition 10). Another unusual phase, found as inclusions in a single large grain of cassiterite within Lou's pegmatite (Fig. 3c), may also be ixiolite on the basis of its high content of divalent and tetravalent elements (mainly Ti and Sc; Table 2, composition 12). It plots in the manganocolumbite field on Figure 2a.

The *Mn-suite* is defined on the basis of manganocolumbite – manganotantalite data from four eastern subgroup pegmatites, an internal beryl pegmatite (Peg. 263), two external beryl pegmatites (one proximal, Peg. 265, and one distal, Peg. 304, from the Separation Rapids pluton), one petalite pegmatite (Marko's pegmatite) and the "cleavelandite" pods at the margin of the Separation Rapids granite (93–260). Crystal form is not dissimilar to the Fe-suite, but there is perhaps a greater tendency to form euhedral, terminated crystals, which are both larger (0.2–3 mm) and modally more abundant. This is particularly so in parts of Marko's pegmatite. There is also a tendency for the manganocolumbite to be associated with the finer-grained, quartz–albite (locally "cleavelandite") portions of the pegmatites, although in one sample from Marko's pegmatite, manganocolumbite occurs mainly as inclusions in spessartine.

In Marko's pegmatite, manganocolumbite is found in the outer, earlier crystallized parts of the pegmatite, such as the layered pegmatite–aplite unit, and compositionally spans a wide range of Mn/(Mn + Fe), from 0.6 to 0.85, but with a restricted Ta/(Ta + Nb) of 0.2–0.3 (decreasing to 0.1 in other pegmatites of the group). Table 2 lists representative data for manganocolumbite and manganotantalite from this suite. These samples do not contain wodginite, and garnet is generally uncommon, although locally abundant. Pegmatite 304 is unusual in containing topaz in addition to manganocolumbite and spessartine. The flat trend in Figure 2b generated by these early units [as a result of varying Mn/(Mn + Fe) at constant Ta/(Ta + Nb)] changes direction sharply upward in the later-crystallized core regions of the pegmatites, comparable to trends reported from lepidolite pegmatites (Černý & Ercit 1985, Spilde & Shearer 1992, Černý & Němec 1995). Manganotantalite with extreme compositions occurs here, approaching end-member manganotantalite [Mn/(Mn + Fe) = 0.98, Ta/(Ta + Nb) = 0.97], either in "cleavelandite" – Li

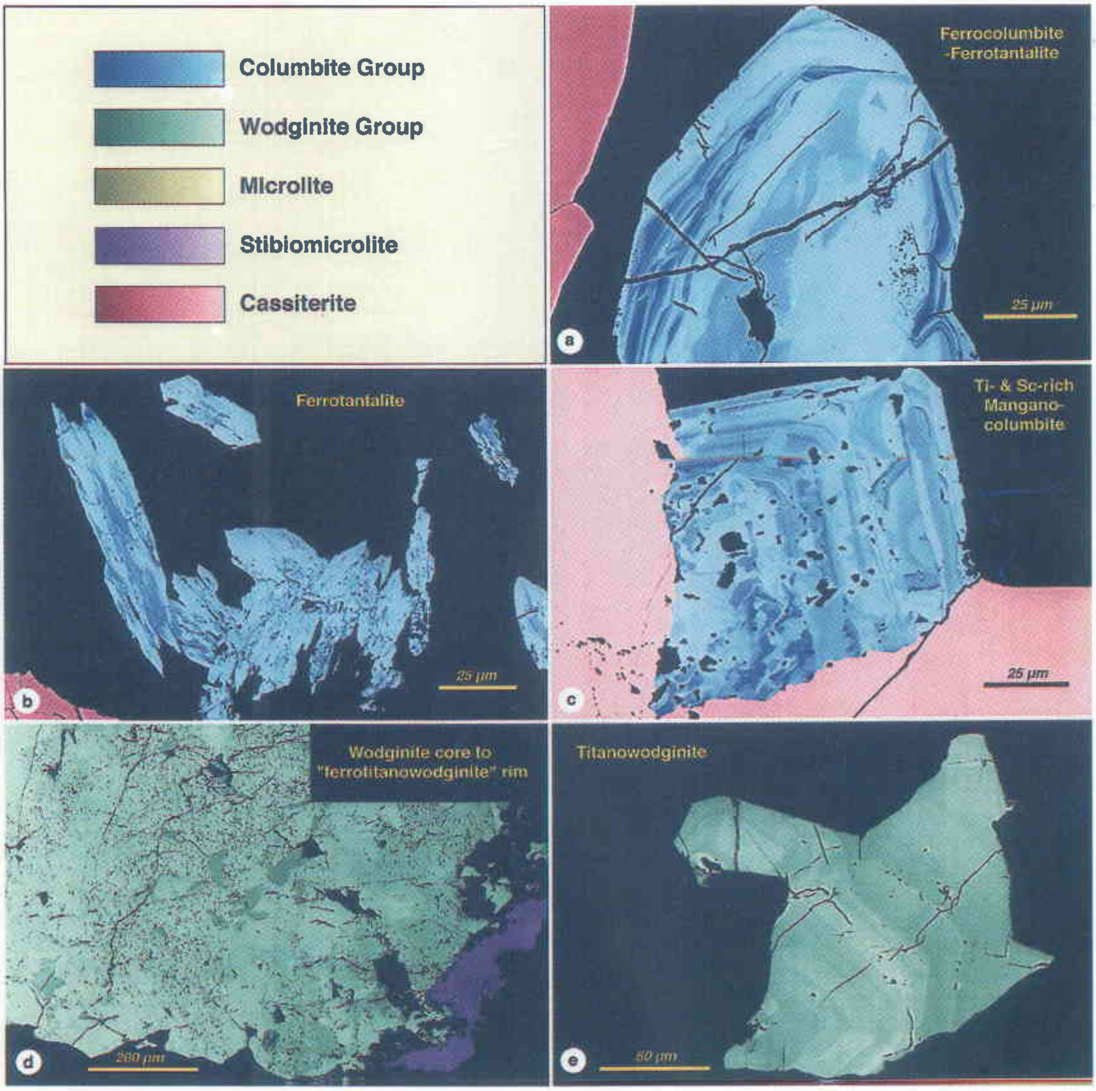


FIG. 3. False-color back-scattered electron images of Nb–Ta oxides from Separation Rapids. (a) Complex zonation in ferrocolumbite – ferrotantalite from Lou's (petalite) pegmatite. (b) Partially altered, late-crystallizing ferrotantalite from petalite pegmatite (Peg. 7). (c) Complexly zoned Sc-rich manganocolumbite (ixiolite?) in cassiterite. From Lou's (petalite) pegmatite. (d) Wodginite core patchily zoned to a "ferrotitanowodginite" composition in the rim. The dark mineral at bottom right is stibiomicrolite. Sample from the wall zone of Marko's pegmatite. (e) Oscillatorily zoned inclusion of titanowodginite in cassiterite from the wall zone of Marko's pegmatite. Color key applies to Figures 3, 6 and 7.

mica-rich pods within beryl pegmatite or as inclusions in microlite (also associated with Li-bearing mica) within petalite pegmatite. Wodginite is found alongside the manganotantalite in the evolved parts of these pegmatites, but spessartine is absent. These samples have also clearly undergone postmagmatic albitization, and this has produced albite-rich veins cross-cutting petalite, small masses and veins of apatite – tourmaline – quartz – albite that replace petalite, and "cleavelandite" replacing margins of blocky K-feldspar. At Marko's pegmatite, more extensive replacement occurs in two stages: (i) development of beryl – Li mica – albite patches, and (ii) development of saccharoidal albite. The implication of this replacement origin is important, as the Ta-rich (and Sn-rich) oxide minerals are strongly concentrated within many of the albite-replacement pods and therefore must also be postmagmatic. A replacement origin for

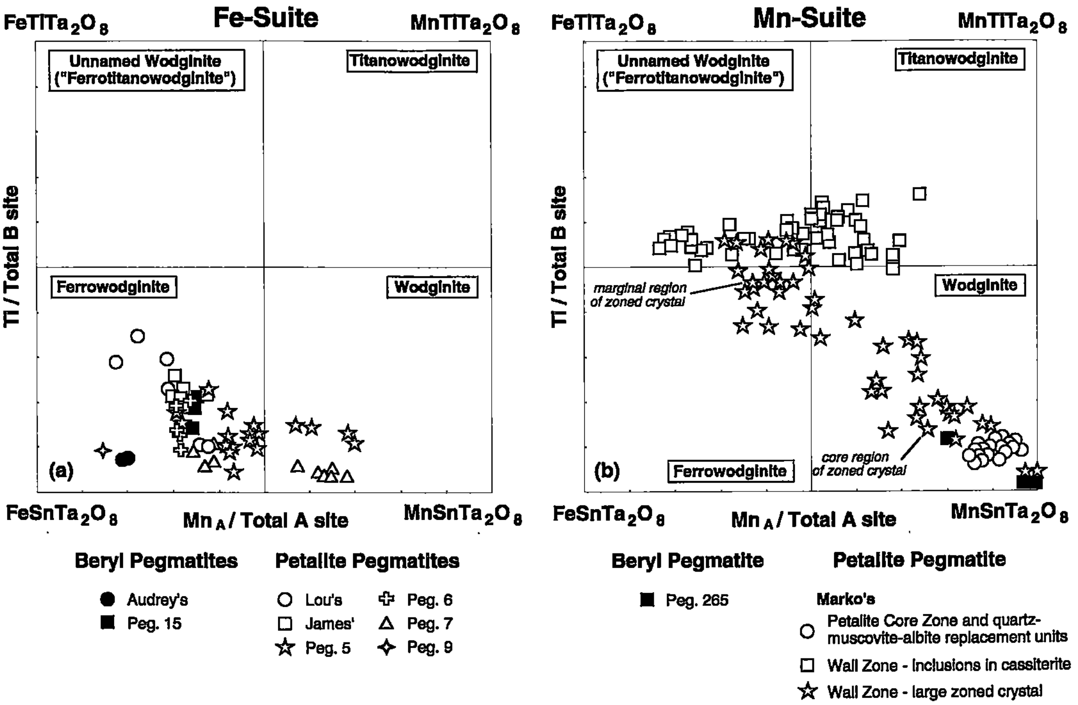


FIG. 4. Covariation of Mn/total *A* site *versus* Ti/total *B* site for wodginite-group minerals from the Separation Rapids pluton and pegmatites. (a) Fe-suite and (b) Mn-suite. *A*-site elements include Mn_A, Fe^{2+} and Li; *B*-site elements include Mn_B, Fe^{3+}, Ti, Sn, Sc, Sb, Bi, Th, U and Ta_B.

the "cleavelandite" pods occurring at the margin of the Separation Rapids pluton is also proposed.

Wodginite-group minerals

Wodginite-group minerals from the eastern subgroup pegmatites are the subject of a companion paper (Tindle *et al.* 1998), and so are not discussed at length here. However, important points relevant to the forthcoming discussion are repeated here for clarity:

(i) A classification diagram for wodginite-group minerals (Mn_A/total *A* site *versus* Ti/total *B* site) has been devised akin to the Mn/(Mn + Fe) *versus* Ta/(Ta + Nb) classification diagram used widely for columbite–tantalite minerals. The diagram is suitable for most wodginite-group minerals with the exception of lithiowodginite. Data for wodginite-group minerals from Separation Rapids are plotted on it (Figs. 4a, b) and indicate that for the Fe-suite of pegmatites, ferrowodginite is the dominant species. There is also an increase in Mn that parallels that observed in coexisting ferrocolumbite. For two evolved samples from pegmatites 5 and 7, this Mn-enrichment extends into the wodginite field and tentatively offers the possibility that the Fe- and Mn-suites may be part of a continuum of compositions.

(ii) Ferrowodginite from individual samples has a tendency to vary more in terms of Ti/(total *B* site) than in terms of Mn_A/(total *A* site). A similar pattern was noted for ferrocolumbite and ferrotantalite from individual samples, where appreciably more variation in Ta/(Ta + Nb) was measured compared to Mn/(Mn + Fe).

(iii) The Mn-suite pegmatites show the greatest variation in composition, and Marko's pegmatite alone contains four wodginite-group species (Fig. 4b). Wodginite is by far the most common species found in this suite and reaches near-end-member compositions in pegmatite Peg. 265. A large crystal (2 mm across) with complex internal zonation and replacement varies in composition from a wodginite core to a ferrowodginite and "ferrotitanowodginite" rim (Fig. 3d). Other data for "ferrotitanowodginite" and titanowodginite (Fig. 3e) were obtained from euhedral to anhedral zoned inclusions in cassiterite crystals. The increased Fe and Ti required to produce these latter wodginite-group species is not consistent with processes of magmatic fractionation, as these elements would be

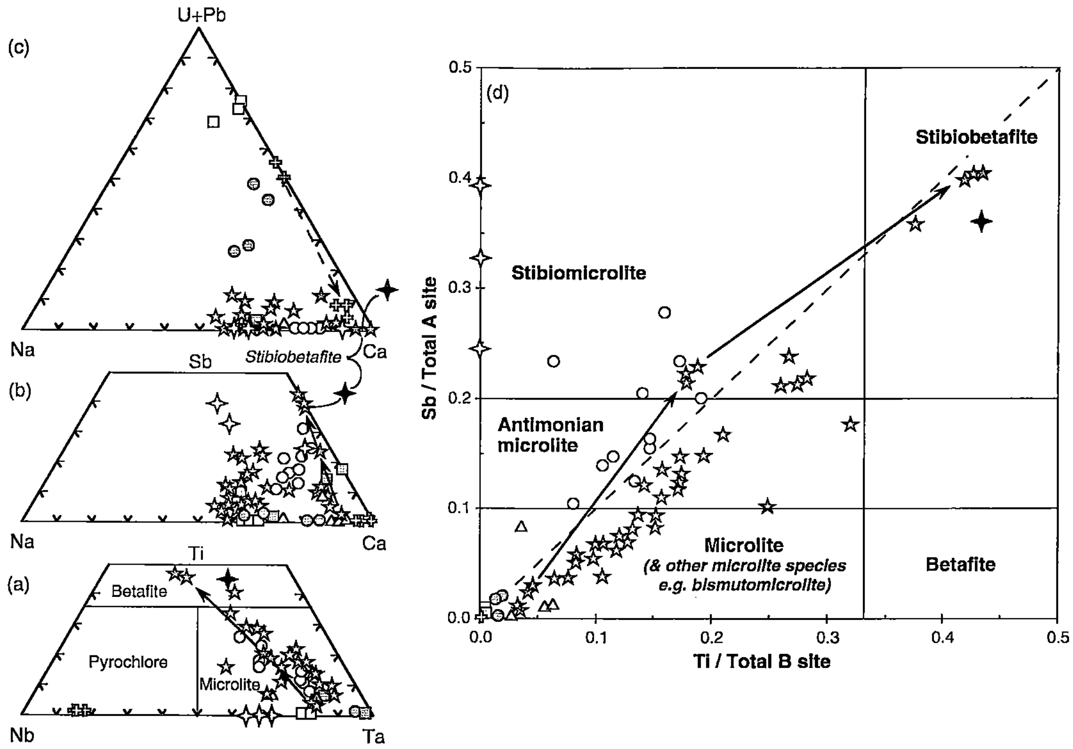


FIG. 5. Variation in composition of pyrochlore-group minerals from Separation Rapids. (a) Classification diagram for the pyrochlore group (Ti–Nb–Ta) illustrating the three main subgroups. (b) Sb–Na–Ca variation diagram. (c) U + Pb–Na–Ca variation diagram. (d) Covariation of Ti/total *B* site with Sb/total *A* site (at.). Open square: microlite from Mn-suite beryl pegmatite (Peg. 265). Open triangle: microlite from Fe-suite petalite pegmatite (Peg. 7). Open circle: antimonian microlite and stibiomicrolite from Fe-suite petalite pegmatite (Lou's). Open five-pointed star: antimonian microlite and stibiomicrolite from the wall zone of Mn-suite pegmatite (Marko's). Shaded five-pointed star: microlite – stibiomicrolite – stibiobetafite from single crystal in the wall zone of Mn-suite pegmatite (Marko's) at the host-rock contact; solid arrows point to later compositions. Shaded circle: uranmicrolite from Fe-suite petalite pegmatite (Peg. 5). Shaded square: bismutomicrolite from Mn-suite beryl pegmatite (Peg. 265). Shaded cross: yttropyrochlore from Fe-suite petalite pegmatite (Peg. 96–29). Open cross: yttrian pyrochlore from Fe-suite petalite pegmatite (Peg. 96–29). Dashed arrow shows replacement trend. Open four-pointed star: stibiomicrolite from the type locality at Varuträsk, Sweden (Groat *et al.* 1987). Solid four-pointed star: stibiobetafite from the type locality at Věžná, Czechoslovakia (Černý *et al.* 1979). For clarity, not all data for microlite are plotted.

expected to be depleted early in the pegmatite's evolution. An influx of these elements from adjacent host-rocks is proposed.

(iv) High but variable levels of W are recorded in many ferrowodginite and wodginite crystals from Separation Rapids. In the Fe-suite, there is an indication that W increases with Mn_A/(total *A* site), especially in the evolved parts of pegmatites 5 and 7 (*e.g.*, in Peg. 5, WO_3 reaches 17.36 wt.%). Marko's pegmatite is unusual in having low W contents (ranging from below detection to 1.02 wt.% WO_3), but very high levels are recorded in the Mn-suite pegmatite Peg. 265, where WO_3 reaches 34.63 wt.%. High Bi (up to 2.35 wt.% Bi_2O_3) is also found in wodginite from this pegmatite. Such "wolframowodginite" contains sufficient W to be classified as a new species, but as with "ferrotitanowodginite" (Ercit *et al.* 1992), it must remain hypothetical until sufficient material is found to characterize it fully.

Pyrochlore-group minerals

The pyrochlore group consists of three subgroups, pyrochlore, betafite and microlite (Fig. 5a). Members of all three subgroups are represented at Separation Rapids; these include four members of the microlite subgroup, *microlite* $(Ca,Na)_2Ta_2O_6(O,OH,F)$, *stibiomicrolite* $(Sb,Ca,Na)_2(Ta,Nb)_2O_6(O,OH,F)$, *uranmicrolite*

TABLE 3. PYROCHLORE GROUP MINERALS FROM SEPARATION RAPIDS

	Microlite					Antimonian microlite			Stibiomicrolite			U microlite	Bi microlite	Stibiobetafite	Y pyrochlore	Yttrian pyrochlore
	Peg. 7	Peg. 265	Marko's			Lou's	Marko's Wall Zone		Lou's	Marko's Wall Zone		Peg. 5	Peg. 265	Marko's Wall-Zone	96-29	96-29
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
TiO2	2.16	0.11	0.63	0.75	0.66	3.32	5.38	6.49	4.88	12.63	8.97	0.34	0.12		0.09	0.00
Nb2O5	13.61	0.97	2.55	2.56	3.73	7.11	6.19	5.63	11.40	9.91	10.00	1.46	6.32	14.88	36.44	44.33
Ta2O5	60.68	78.19	73.22	76.53	72.53	63.04	58.13	55.61	47.40	35.03	46.65	65.29	42.21	16.50	12.79	12.95
WO3	0.12	0.00	0.00	0.00	0.03	0.00	0.00	0.07	0.00	0.48	0.00	0.00	2.18	3.74	1.44	1.81
SnO2	3.34	0.55	1.21	1.20	1.20	1.37	1.75	1.90	3.10	3.11	1.60	1.03	4.52	21.79	0.00	0.07
Al2O3	0.10	0.00	0.00	0.00	0.00	0.00	0.19	0.00	0.06	0.38	0.00	0.00	3.83	0.49	0.00	0.77
PbO	0.28	0.40	0.12	0.59	0.05	0.64	0.05	0.07	0.58	0.13	0.07	3.05	4.82	0.50	0.97	0.37
ThO2	0.03	0.02	0.04	0.02	0.02	0.00	0.03	0.04	0.00	0.05	0.00	0.00	0.00	0.14	0.39	0.62
UO2	0.55	1.91	0.64	1.53	0.00	0.12	0.15	0.17	0.11	0.21	0.00	13.24	2.10	0.00	2.18	2.00
Sb2O3	0.58	0.19	1.12	0.62	1.04	7.23	7.88	9.90	15.75	20.91	14.22	0.67	0.21	0.02	0.03	0.01
Bi2O3	0.04	0.02	0.04	0.04	0.09	0.03	0.00	0.00	0.02	0.07	0.00	0.08	9.63	24.86	0.00	0.00
Sc2O3	0.00	0.02	0.00	0.00	0.02	0.00	0.05	0.08	0.03	0.10	0.00	0.02	0.02	0.00	0.08	0.27
FeO	0.34	1.23	0.01	0.02	0.01	0.39	0.07	0.07	1.71	0.67	0.18	1.24	4.75	0.75	0.00	0.81
MnO	0.39	0.32	0.07	0.23	0.11	0.59	0.28	0.43	0.99	1.08	0.65	0.45	2.77	0.58	0.00	1.10
CaO	14.87	9.91	11.78	9.94	12.14	12.72	13.04	12.81	12.54	12.78	12.50	6.27	0.47	13.08	0.58	6.90
Na2O	0.65	3.24	5.36	3.51	5.32	1.99	4.99	3.19	0.47	0.00	4.00	1.96	0.15	0.04	0.00	0.12
Cs2O	-	-	-	-	-	-	-	-	-	-	-	-	-	0.26	0.05	0.02
BaO	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.00	0.06
K2O	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.03	0.22
Y2O3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	20.92	5.15
SiO2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.00	4.67
F	1.94	2.51	4.09	3.08	3.96	2.00	3.45	2.86	0.93	0.44	1.92	1.06	0.05	0.00	0.26	0.24
Subtotal	99.68	99.59	100.88	100.62	100.91	100.55	101.63	99.32	99.97	97.98	100.76	96.16	84.15	97.62	76.22	82.49
O=F	0.82	1.06	1.72	1.30	1.67	0.84	1.45	1.20	0.39	0.19	0.81	0.45	0.02	0.00	0.11	0.10
Total	98.86	98.53	99.16	99.32	99.24	99.71	100.18	98.12	99.58	97.79	99.95	95.71	84.13	97.62	76.11	82.39
Ti	0.126	0.008	0.043	0.049	0.044	0.213	0.343	0.418	0.319	0.750	0.549	0.027	0.008	0.000	0.006	0.000
Nb	0.478	0.040	0.105	0.101	0.151	0.275	0.237	0.218	0.448	0.354	0.368	0.069	0.268	0.627	1.616	1.606
Ta	1.281	1.933	1.808	1.809	1.762	1.465	1.341	1.297	1.120	0.753	1.032	1.861	1.077	0.418	0.341	0.282
W	0.002	0.000	0.000	0.000	0.001	0.000	0.000	0.002	0.000	0.010	0.000	0.000	0.053	0.090	0.037	0.038
Sn	0.103	0.020	0.044	0.042	0.043	0.047	0.059	0.065	0.107	0.098	0.052	0.043	0.169	0.810	0.000	0.002
Al	0.009	0.000	0.000	0.000	0.000	0.000	0.019	0.000	0.006	0.035	0.000	0.000	0.424	0.054	0.000	0.073
Sum B	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.000	2.000		
Pb	0.006	0.010	0.003	0.014	0.001	0.015	0.001	0.002	0.014	0.003	0.002	0.086	0.122	0.013	0.026	0.008
Th	0.001	0.000	0.001	0.000	0.000	0.000	0.001	0.001	0.000	0.001	0.000	0.000	0.000	0.003	0.009	0.011
U	0.010	0.039	0.013	0.030	0.000	0.002	0.003	0.003	0.002	0.004	0.000	0.309	0.044	0.000	0.048	0.036
Sb	0.019	0.007	0.042	0.022	0.038	0.255	0.276	0.350	0.564	0.681	0.477	0.029	0.008	0.001	0.001	0.000
Bi	0.001	0.000	0.001	0.001	0.002	0.001	0.000	0.000	0.000	0.001	0.000	0.002	0.233	0.598	0.000	0.000
Sc	0.000	0.002	0.000	0.000	0.002	0.000	0.004	0.006	0.002	0.007	0.000	0.002	0.002	0.000	0.006	0.019
Fe	0.022	0.093	0.001	0.001	0.001	0.028	0.005	0.005	0.124	0.044	0.012	0.109	0.373	0.058	0.000	0.054
Mn	0.026	0.025	0.005	0.017	0.008	0.043	0.020	0.031	0.073	0.072	0.045	0.040	0.220	0.046	0.000	0.075
Ca	1.237	0.965	1.146	0.926	1.162	1.165	1.185	1.177	1.167	1.082	1.089	0.704	0.047	1.307	0.060	0.592
Na	0.098	0.571	0.944	0.591	0.921	0.330	0.821	0.530	0.079	0.000	0.631	0.398	0.027	0.007	0.000	0.019
Cs	-	-	-	-	-	-	-	-	-	-	-	-	-	0.010	0.002	0.001
Ba	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.000	0.002
K	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.003	0.022
Y	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1.092	0.220
Si	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.000	0.374
Sum A	1.418	1.712	2.156	1.602	2.136	1.838	2.315	2.105	2.026	1.895	2.255	1.679	1.076	2.042	1.247	1.433
F	0.476	0.721	1.175	0.847	1.118	0.541	0.926	0.776	0.256	0.110	0.494	0.351	0.015	0.000	0.079	0.060
Total	3.894	4.434	5.331	4.449	5.254	4.378	5.241	4.881	4.282	4.005	4.749	4.030	3.091	4.042	4.574	4.926
Comment:	Incl. in cassiterite Replaces Fe wodginite	Possibly primary	Replaces wodginite		Possibly primary	Incl. in cassiterite Possibly primary	Primary		Possibly primary		Replaces FeTi-wodginite	Incl. in wodginite Primary	Replaces wodginite	After stibiomicrolite & microlite		Replaces Y pyrochlore

$(U,Ca)_2(Ta,Nb)_2O_6(O,OH,F)$ and *bismutomicrolite* $(Bi,Ca)_2(Ta,Nb)_2O_6(OH)$. *Antimonian microlite* intermediate in composition between *microlite* and *stibiomicrolite* also is found. In addition, *stibiobetafite* $(Sb,Ca)_2(Ti,Nb,Ta)_2O_6(OH)$ and *yttropyrochlore* $(Y,Na,Ca,U)_{1-2}(Nb,Ta,Ti)_2O_6(OH)$ also are found. These minerals do not occur in the Separation Rapids pluton or those pegmatite bodies with ferrocolumbite or ferrotantalite whose Mn/(Mn + Fe) values are less than 0.3, and their occurrence does not correlate with the presence of either beryl or petalite. Of all the microlite subgroup species, *microlite* is the one found most commonly, such as in a Fe-suite petalite pegmatite (Peg. 7) and two bodies of Mn-suite pegmatite (especially Marko's, but also beryl pegmatite Peg. 265). *Stibiomicrolite* is a very rare mineral found in a Fe-suite petalite pegmatite (Lou's) and in the wall zone of Marko's pegmatite. *Bismutomicrolite* is only found in a Mn-suite beryl pegmatite Peg. 265, whereas *uranmicrolite* and *yttropyrochlore* are restricted to Fe-suite petalite pegmatite Peg. 5 and the southwestern subgroup "Big Whopper" pegmatite (Peg. 96–29), respectively. *Stibiobetafite* has the most restricted occurrence of all, being found in the wall zone of Marko's pegmatite at the actual contact with its host rock, a metasomatized garnet – biotite – plagioclase metavolcanic rock. Table 3 lists representative compositions of pyrochlore-group minerals from Separation Rapids, with structural formulae calculated on the basis of the *B* site summing to 2.00 atoms per formula unit.

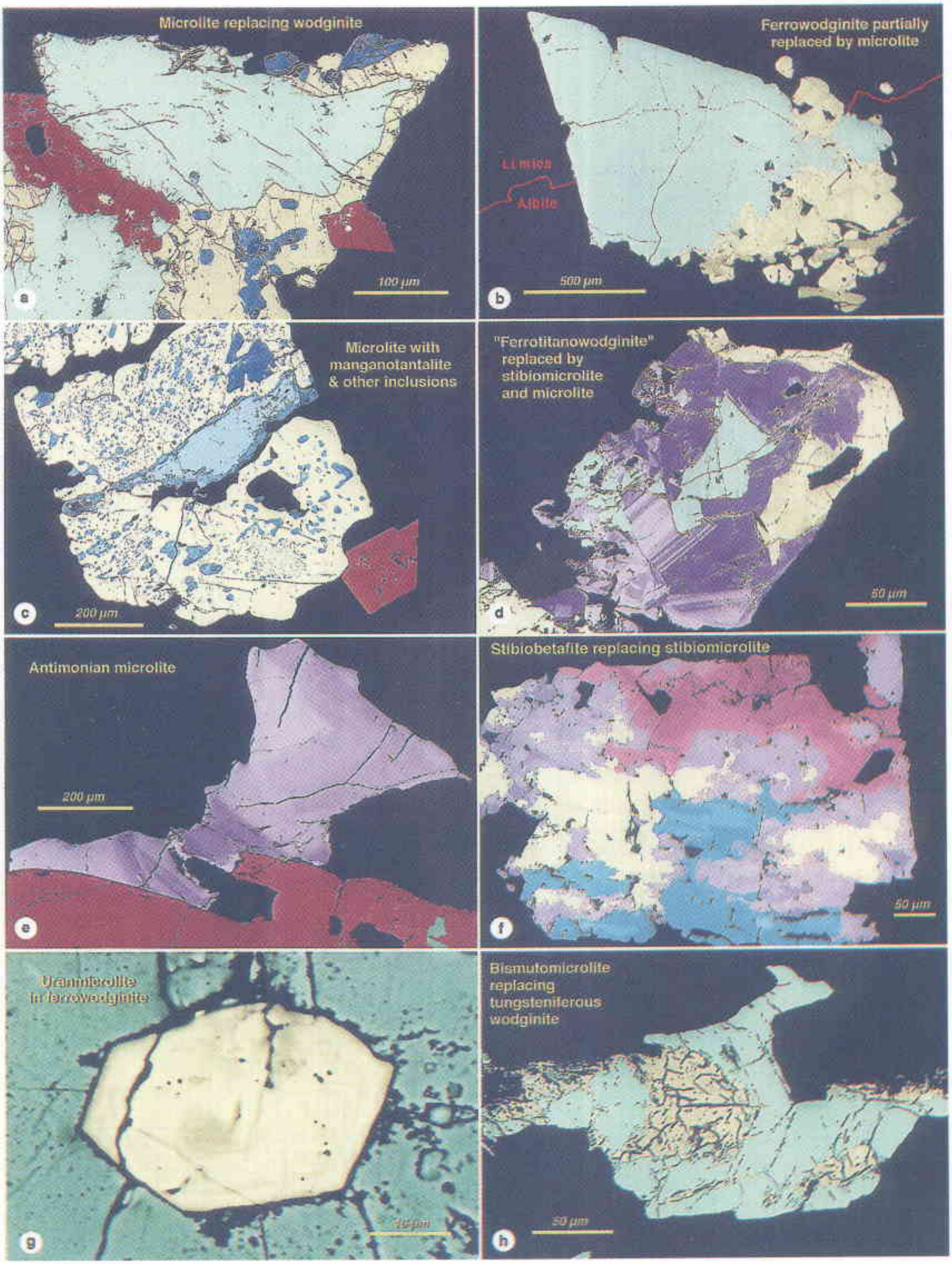
Microlite is a strongly associated with Li-bearing mica (zinnwaldite?), with most *microlite* crystals being included within, in contact with, or adjacent to such mica. *Microlite* typically forms mottled, irregularly shaped, inclusion-rich crystals, which partially or totally replace earlier ferrowodginite, or wodginite (rarely manganocolumbite). In Marko's pegmatite, the presence of *microlite* after wodginite (Fig. 6a) and small grains of inclusion-free cassiterite growing from within the *microlite* outward beyond the original margin of the wodginite suggests a reaction involving the following exchange:

$$\underset{MnSnTa_2O_8}{\text{wodginite}} + \text{Ca,Na,F-rich fluid} \rightarrow \underset{SnO_2}{\text{cassiterite}} + \underset{(Ca,Na)_2Ta_2O_6F}{\textit{microlite}}$$

Evidence that the Ca, Na and F components of this fluid became sufficiently concentrated to react late in the crystallization sequence is provided in Figure 6b, where part of a euhedral crystal of wodginite, jutting out from the edge of a large grain of Li-bearing mica (zinnwaldite?), has been totally replaced by *microlite* prior to being partially included within albite. One sample from a muscovite–albite replacement unit within the petalite-rich core zone of Marko's pegmatite hosts abundant *microlite* with a particularly complex history. Large anhedral crystals of *microlite* up to 2.5 mm in length are found containing elongate inclusions of manganotantalite (the most manganiferous from this pegmatite) rimmed by a ferrocolumbite alteration rim (Fig. 6c). Such *microlite* crystals often contain numerous oriented inclusions of ferrocolumbite that form fan-shaped patterns or a symplectitic texture. Certain zones of these crystals are either porous and rich in inclusions, or non-porous, with larger, more discrete inclusions. Most inclusions show some oscillatory zoning.

Whereas *microlite* is closely associated with a Li-rich mica, *stibiomicrolite* and *antimonian microlite* tend to occur associated with cassiterite and wodginite. *Stibiomicrolite* is pale yellow and usually found as inclusions in, or in contact with, cassiterite. Exceptions include a *stibiomicrolite* at the margin of a "ferrotitanowodginite" crystal and a *stibiomicrolite* partially replacing a wodginite crystal included in quartz. A *stibiomicrolite* inclusion in cassiterite from

FIG. 6. False-color back-scattered electron images of pyrochlore-group minerals from Separation Rapids. (a) Microlite (yellow) after wodginite (green) from the albite replacement unit of Marko's pegmatite. Note the small crystals of cassiterite (red) growing out from within the microlite and the patchily zoned ferrocolumbite (blue) inclusions in the microlite. (b) Subhedral wodginite jutting out from the edge of a large grain of lithium-rich mica (zinnwaldite?), in part replaced by microlite prior to being partially included within untwinned plagioclase. Sample taken from a muscovite–albite replacement unit within the petalite core-zone of Marko's pegmatite. (c) Large grain of microlite (yellow) containing an elongate inclusion of manganotantalite (pale blue) rimmed by and including numerous oriented inclusions of ferrocolumbite (ixiolite?) (blue). A euhedral crystal of cassiterite (red) grows from its margin. Sample taken from a muscovite–albite replacement unit within the petalite core-zone of Marko's pegmatite. (d) "Ferrotitanowodginite" inclusion (center) in cassiterite partially replaced by stibiomicrolite (purple and zoned in parts), which itself is partially replaced by microlite (yellow region, top right). From the wall zone of Marko's pegmatite. (e) Zoned antimonian microlite growing along margin of large grain of cassiterite. The cassiterite hosts many inclusions of titanowodginite (two at bottom right) and "ferrotitanowodginite". From the wall zone of Marko's pegmatite. (f) Ferrocolumbite – manganocolumbite replaced in a three-step process starting with microlite, then stibiomicrolite and, finally, stibiobetafite (pink). From the wall zone of Marko's pegmatite. (g) Euhedral inclusion of uranmicrolite in large grain of ferrowodginite from petalite pegmatite (Peg. 5). (h) Replacement of tungsteniferous wodginite by bismutomicrolite in beryl pegmatite Peg. 265. For color key, see Fig. 3.


Microlite replacing wodginite
100 µm
a
Ferrowodginite partially replaced by microlite
Li mica
Albite
500 µm
b
Microlite with manganotantalite & other inclusions
200 µm
c
"Ferrotitanowodginite" replaced by stibiomicrolite and microlite
50 µm
d
Antimonian microlite
200 µm
e
Stibiobetafite replacing stibiomicrolite
50 µm
f
Uranmicrolite in ferrowodginite
10 µm
g
Bismutomicrolite replacing tungsteniferous wodginite
50 µm
h

the wall zone of Marko's pegmatite clearly replaces "ferrotitanowodginite" (Fig. 6d), but in another sample from the same unit, a large (0.5 mm), oscillatorily zoned *antimonian microlite* crystal has grown against an earlier cassiterite containing titanowodginite and "ferrotitanowodginite" inclusions, and thus is most likely primary (Fig. 6e). This crystal has an Sb-rich core (9.99 wt.% Sb_2O_3), and the Sb content decreases outward to *antimonian microlite* compositions with 3.6 wt.% Sb_2O_3 (Table 3, compositions 7 and 8). Other crystals belonging to the microlite subgroup have much higher Sb contents (*e.g.*, Marko's pegmatite, with 20.91 wt.% Sb_2O_3; Table 3, anal. 10), which exceeds the necessary 20 at.% Sb in the *A* site required to name the mineral as true *stibiomicrolite* (Hogarth 1977, Groat *et al.* 1987). In the Fe-suite petalite pegmatite (Lou's), oscillatorily zoned inclusions of *stibiomicrolite* in cassiterite are nearly euhedral, with a form reminiscent of wodginite (see Dunn *et al.* 1978, Fig. 5). These too are most likely primary occurrences.

Microlite-subgroup minerals show little systematic variation within an individual pegmatite, but there are differences among pegmatites (Table 3); however, the wall zone of Marko's pegmatite hosts a suite with compositions ranging from *microlite* (0.2 – 4.6 wt.% Sb_2O_3) to *antimonian microlite* (3.8 – 10.5 wt.% Sb_2O_3) and *stibiomicrolite* (9.9 – 12.7 wt.% Sb_2O_3). It is apparent from these data and, to a lesser extent, those from Lou's pegmatite, that there is a continuum of compositions from microlite to stibiomicrolite (Figs. 5b, d) and that where the Ti content is high, so too is the Sb content. This systematic behavior is more complex than first appears, as petrographic observations indicate that stibiomicrolite can crystallize earlier (Fig. 6e) or later (Fig. 6f) than microlite, and further suggest that the initial concentration of Ti and Sb is more related to the local availability of these elements rather than simply to magmatic fractionation processes.

Uranmicrolite has been observed in two samples from a petalite pegmatite (Peg. 5), where it occurs as small (approximately 50 μm), weakly zoned inclusions in ferrowodginite (Fig. 6g). The pegmatites are assumed to belong to the Fe-suite, although columbite–tantalite was not found in either sample. Uranmicrolite has a euhedral form, is yellowish brown in color, and appears to be an early-crystallizing primary phase. The low analytical total (Table 3, composition 12; see also Fig. 6g) is considered to indicate a high H_2O content. Petrographic evidence in the form of radial fractures typical of radiation damage due to volume expansion indicates that the crystal is metamict or nearly so. An alpha-decay dose of approximately 1.1×10^{18} α/mg (based on equation 1 of Lumpkin *et al.* 1994) is consistent with this interpretation. Because of their rarity, little can be said about how *uranmicrolite* (or *bismutomicrolite*) are related to other microlite-subgroup minerals (Fig. 5c).

Bismutomicrolite is a rare mineral that occurs in the most evolved, Mn-suite beryl pegmatite (Peg. 265) in association with large pods enriched in "cleavelandite" and Li-rich mica. It forms as a product of the late-stage alteration of tungsteniferous wodginite, where it selectively replaces zone boundaries and individual zones of the wodginite. The mineral has a mottled appearance and is dissected by many cracks (Fig. 6h). Although the mineral contains much less U than *uranmicrolite* (Table 3; composition 13), it appears to be affected by metamictization. Yet, calculations of alpha-decay (approximately 1.8×10^{17} α/mg based on equation 1 of Lumpkin *et al.* 1994) indicate only moderate damage. The very low analytical total is considered to reflect a high H_2O content. Because the major *A*-site cations, Bi and Fe are not plotted on Figure 5c, the *bismutomicrolite* data plot at anomalously high U + Pb levels on this diagram. Table 3, composition 13 shows that the elements U, Pb, Na and Ca are all in low abundance.

Stibiobetafite has the highest Sb concentrations of all the pyrochlore-group minerals found at Separation Rapids (20.9–25.8 wt.% Sb_2O_3), but is only found in the wall zone of Marko's pegmatite. It occurs as a diffuse inclusion in a large crystal of wodginite that exhibits complex internal zonation or replacement and whose composition varies from a wodginite core to a ferrowodginite and "ferrotitanowodginite" rim. Figure 6f illustrates a second occurrence of *stibiobetafite* in which crystallization of the pyrochlore-group mineral appears to have been a three-step process. Manganocolumbite has been replaced by *microlite*, which in turn is replaced by *stibiomicrolite*. The final step of the process is the crystallization of oscillatorily zoned *stibiobetafite* on the *stibiomicrolite*. Perhaps because of a change in volume as manganocolumbite was replaced by *microlite* or because some *stibiomicrolite* was dissolved away first, much of the *stibiobetafite* was able to grow in open spaces to form subhedral crystals. The change in composition of the pyrochlore-group minerals in this crystal are shown by an arrow in Figures 5a, b and d.

As with the transformation of wodginite to microlite, the sequence of reactions leading to crystallization of *stibiobetafite* requires an initial influx of Ca, Na and F, which is followed later by an influx or exchange with a source rich in Ti and Sb:

$$\begin{array}{l}\text{manganocolumbite} + \text{Ca,Na,F-rich fluid}\\ (Mn,Fe)(Nb,Ta)_2O_6\\ \qquad\qquad \rightarrow \textit{microlite}\\ \qquad\qquad (Ca,Na)_2(Ta,Nb)_2O_6(OH,F)\\ \qquad\qquad + (Fe+Mn) \qquad\qquad (2)\end{array}$$

$$\begin{array}{ll}\textit{microlite} + \text{Ti, Sb source} \rightarrow & \textit{stibiomicrolite}\\ (Ca,Na)_2(Ta,Nb)_2O_6(F) & (Sb,Ca)_2(Ta,Nb,Ti)_2O_6(OH,F)\\ \qquad + (Ca,Na,F) & \qquad\qquad (3)\end{array}$$

$$\begin{aligned} &\textit{stibiomicrolite} + \text{Ti, Sb source} \\ &(Sb,Ca)_2(Ta,Nb,Ti)_2O_6(OH,F) \\ &\quad \rightarrow \textit{stibiobetafite} \\ &\quad (Sb,Ca)_2(Ti,Nb,Ta)_2O_6(OH) \\ &\quad + (Ca,Na,F) \end{aligned} \tag{4}$$

A (Ca,Na,F)-bearing fluid is a likely constituent contributing to albitization, which probably developed as the pegmatite-forming melt evolved and conditions changed from magmatic to hydrothermal. However, as the stibiobetafite (only?) occurs at the actual margin of the wall zone of Marko's pegmatite, it is reasonable to assume the Ti and Sb came from the host rocks.

Yttropyrochlore has been found in only one sample, a petalite pegmatite (96–29) from the southwestern subgroup. A 150-µm-long relict core of yttropyrochlore composition (20.92 wt.% Y_2O_3) is mantled by an alteration rim with a much lower yttrium content (5.15 wt.% Y_2O_3) and high Si content (4.67 wt.% SiO_2) (Table 3, compositions 15 and 16, and Fig. 7a). The source of the Y is unknown, but possibilities include its concentration during magmatic fractionation to form primary yttropyrochlore or crystallization of xenotime from the pegmatite-forming melt, later to be dissolved by albitizing fluids, with the mobilized Y then available to form yttropyrochlore. Further discoveries are required before this can be unraveled.

Ferrotapiolite

Ferrotapiolite, $Fe(Ta,Nb)_2O_6$, is an uncommon oxide that is spatially restricted to a few Fe-suite beryl pegmatites of the southwestern subgroup. Influx of fluids from these pegmatites into a small area (200 m^2) led to metasomatic alteration of metavolcanic host-rocks, resulting in elevated Li contents, up to 245 ppm, compared to "background" levels of 16 ppm (Breaks & Tindle 1997). Ferrotapiolite was first found in a sodic pegmatite dyke (Peg. 96–53) as a 30-µm inclusion associated with ferrotantalite (Fig. 7b), both within a larger grain of cassiterite. Ferrotapiolite from a second sodic pegmatite dyke (Peg. 96–9) appears to replace earlier-formed patchily zoned ferrotantalite (Fig. 7c), and in a third occurrence from a narrow, deformed garnet–albite pegmatite (Peg. 96–81), ferrotapiolite forms large clusters of grains (600 µm across) intimately associated with, but later than, ferrotantalite. Table 4 lists analytical data from three coexisting pairs from the above samples. These coexisting pairs are similar in composition to those from a pegmatite of the beryl–columbite subtype from Spittal a.d. Drau, Austria (Černý *et al.* 1989), but crossing tie-lines and compositions within the two-phase field (Fig. 2a) indicate localized disequilibrium crystallization and possibly metastable conditions (Černý *et al.* 1992). Ferrotapiolite from Separation Rapids can also contain higher levels of Sc (up to 0.72 wt.% Sc_2O_3) and W (up to 0.61 wt.% WO_3) than the compiled worldwide dataset reported by Wise & Černý (1996).

Ferrotapiolite has not been found in the eastern subgroup pegmatites. However, a ferrotapiolite-like phase occurs in one part of the wall zone of Marko's pegmatite. Its unusual composition is attributable to partial replacement of ferrotapiolite along cleavage planes by microlite (Fig. 7d, Table 4, compositions 7 and 8). Only Pough (1945) and Eid & von Knorring (1976) appear to have reported this process previously. As with replacement of wodginite and manganocolumbite by microlite described above, replacement of ferrotapiolite by microlite requires interaction with a (Ca,Na,F)-rich fluid.

Strüverite

This rare mineral, containing 23.85–42.49 wt.% Ta_2O_5 (Table 4, compositions 3–5), has been found only in the wall zone of Marko's pegmatite. It occurs as a 0.3-mm anhedral inclusion in coarse-grained albite, where it appears to be replacing manganotantalite. In a second occurrence, a titanowodginite inclusion in a large grain of cassiterite that also hosts "ferrotitanowodginite" inclusions has been partially replaced by patchily zoned strüverite. The strüverite has continued growing out from the margin of the cassiterite and has developed both a euhedral habit and well-defined oscillatory zonation (Fig. 7e).

Cassiterite

Cassiterite is common at Separation Rapids, occurring in all types of granitic pegmatite. Although its modal abundance is quite variable, it is in many cases the most common oxide present. In much of the Separation Rapids pluton, it is either absent or rare, although crystals up to 3 mm across are found in the more evolved part of the pluton. The beryl-bearing pegmatites contain relatively little or no cassiterite, the exception being pegmatite Peg. 265, which shows other mineralogical differences with most of the other beryl pegmatites (Table 1, column 5). Cassiterite in pegmatites from the southwestern subgroup is not discussed here.

It is in the petalite-bearing pegmatites that cassiterite occurs in the greatest abundance. Pegmatite 7 is particularly enriched, with visual estimates of modal abundance reaching 3–5% in zones rich in fluorapatite and zircon. Noteable enrichment also occurs in both Lou's and Marko's pegmatites. However, no cassiterite has been found in pegmatites 1 or 10, and abundances are quite low in pegmatites 6, 8, 9 and 11.

Cassiterite commonly forms euhedral, diamond-shaped crystals up to 1.2 cm in length (but more commonly 0.2 – 0.4 mm in pegmatites 6, 8, 9, and 11), and exhibits a wide variety of colors in thin section, from dark brown (almost black) through deep reddish

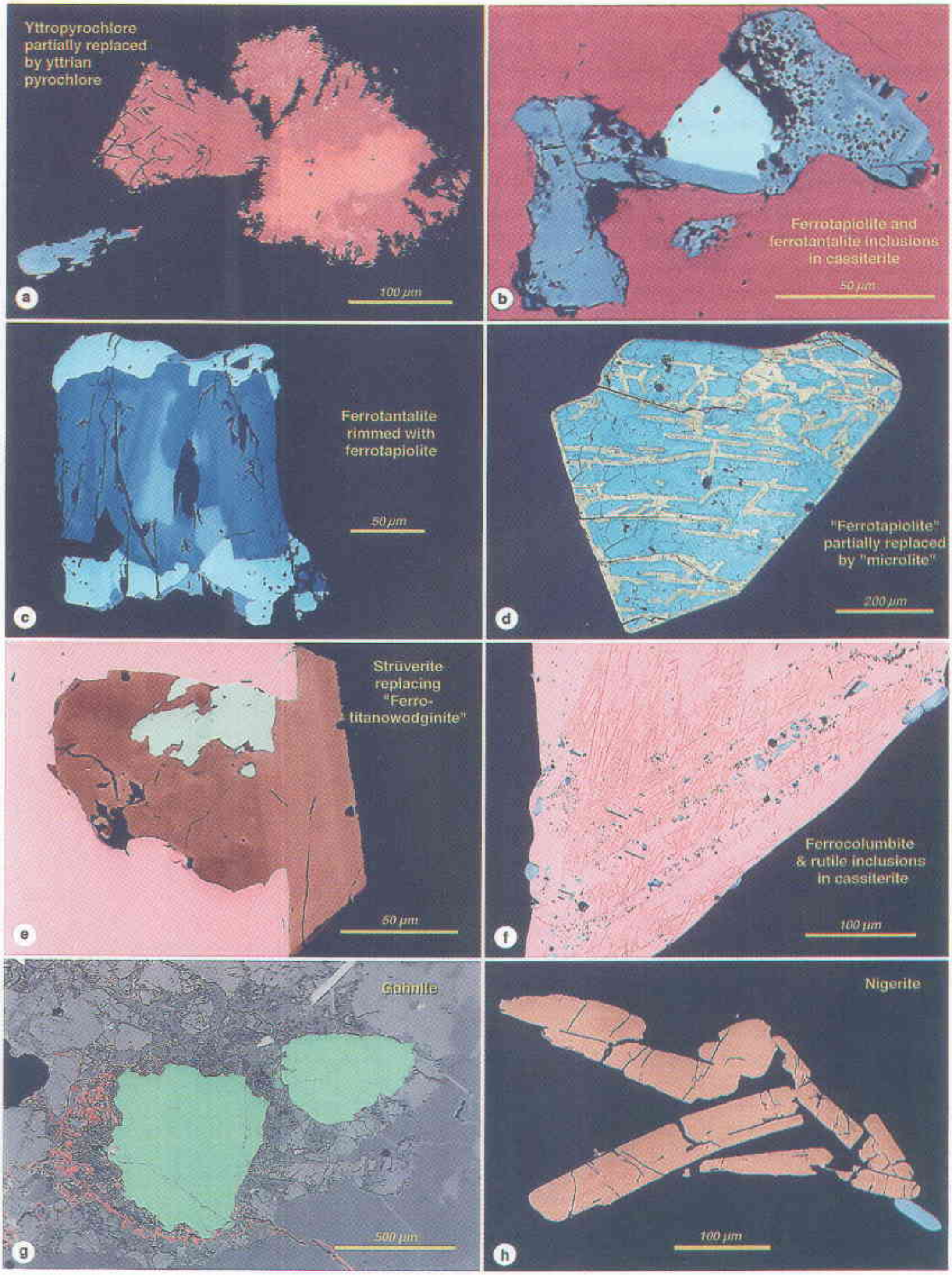

Yttropyrochlore partially replaced by yttrian pyrochlore
100 µm
a
Ferrotapiolite and ferrotantalite inclusions in cassiterite
50 µm
b
Ferrotantalite rimmed with ferrotapiolite
50 µm
c
"Ferrotapiolite" partially replaced by "microlite"
200 µm
d
Strüverite replacing "Ferro-titanowodginite"
50 µm
e
Ferrocolumbite & rutile inclusions in cassiterite
100 µm
f
Gahnite
500 µm
g
Nigerite
100 µm
h

TABLE 4. FERROTAPIOLITE-FERROTANTALITE PAIRS AND RELATED MINERALS FROM SEPARATION RAPIDS

	Fe tantalite	Ferrotapiolite	Fe tantalite	Ferrotapiolite	Fe tantalite	Ferrotapiolite
	96-53		96-9		96-81	
	1	2	3	4	5	6
CaO	0.03	0.00	0.00	0.03	0.00	0.00
FeO	13.13	14.38	13.87	14.48	12.96	13.57
MnO	3.60	0.09	1.01	0.02	1.69	0.24
TiO2	0.74	0.69	1.71	3.40	0.11	0.00
Nb2O5	27.49	8.22	25.32	9.60	18.97	4.72
Ta2O5	54.71	74.61	55.21	70.83	64.71	79.02
SnO2	0.53	0.99	1.15	0.54	0.56	1.12
WO3	0.14	0.22	0.49	0.36	0.60	0.09
PbO	0.08	0.00	0.16	0.00	0.28	0.62
ThO2	0.00	0.00	0.00	0.00	0.00	0.00
UO2	0.00	0.00	0.00	0.02	0.00	0.00
Sb2O3	0.00	0.00	0.00	0.00	0.20	0.20
Bi2O3	0.00	0.00	0.00	0.06	0.44	0.00
Sc2O3	0.00	0.04	0.25	0.08	0.00	0.06
Total	100.44	99.23	99.17	99.41	100.52	99.65
No. of oxygens	6	6	6	6	6	6
Ca	0.002	0.000	0.000	0.002	0.000	0.000
Fe	0.784	0.973	0.840	0.942	0.822	0.947
Mn	0.218	0.006	0.062	0.001	0.109	0.017
Ti	0.040	0.042	0.093	0.199	0.006	0.000
Nb	0.888	0.301	0.829	0.338	0.650	0.178
Ta	1.063	1.642	1.087	1.499	1.334	1.792
Sn	0.015	0.032	0.033	0.017	0.017	0.037
W	0.003	0.005	0.009	0.007	0.012	0.002
Pb	0.002	0.000	0.003	0.000	0.006	0.014
Th	0.000	0.000	0.000	0.000	0.000	0.000
U	0.000	0.000	0.000	0.000	0.000	0.000
Sb	0.000	0.000	0.000	0.000	0.006	0.007
Bi	0.000	0.000	0.000	0.001	0.009	0.000
Sc	0.000	0.003	0.015	0.005	0.000	0.004
Total	3.014	3.002	2.973	3.012	2.970	2.998
Comments:	both are inclusions in cassiterite		formed early	formed later	formed early	formed later

	Ferrotapiolite-like	Microlite-like
	Marko's	
	7	8
Na2O	0.47	3.60
CaO	1.55	10.67
Al2O3	0.00	0.00
FeO	15.39	1.10
MnO	0.03	0.05
TiO2	0.00	0.00
Nb2O5	0.90	0.85
Ta2O5	70.60	74.11
SnO2	1.04	1.02
WO3	0.36	0.27
PbO	0.06	0.12
ThO2	0.05	0.02
UO2	0.99	1.02
Sb2O3	0.80	0.61
Bi2O3	0.07	0.04
Sc2O3	0.87	0.07
Cs2O	0.07	0.08
BaO	0.32	0.05
SiO2	2.60	1.41
F	0.00	2.33
Subtotal	96.18	97.41
O=F	0.00	0.98
Total	96.18	96.43
Ti	0.000	0.000
Nb	0.041	0.036
Ta	1.909	1.918
W	0.009	0.007
Sn	0.041	0.039
Al	0.000	0.000
Fe	1.280	0.088
Mn	0.002	0.004
Ca	0.165	1.088
Na	0.091	0.665
Ba	0.012	0.002
Pb	0.002	0.003
Th	0.001	0.000
U	0.022	0.022
Sb	0.033	0.024
Bi	0.002	0.001
Cs	0.003	0.003
Sc	0.075	0.006
Si	0.258	0.135
F	0.000	0.700
Total	3.946	4.739

Formulas based on sum B site = 2.00

FIG. 7. False-color back-scattered electron images of oxide minerals from Separation Rapids. (a) Yttropyrochlore (orange) replaced by yttrian pyrochlore (brownish orange). From petalite pegmatite Peg. 96–29. (b) Ferrotapiolite (cyan) and ferrotantalite (blue) inclusions in cassiterite. From beryl pegmatite 96–53. (c) Ferrotapiolite along margins of earlier-crystallized ferrotantalite. From beryl pegmatite Peg. 96–9. (d) "Ferrotapiolite" replaced along cleavages and fractures by "microlite". From Marko's pegmatite. (e) Patchy zoned strüverite replacing an inclusion of titanowodginite in cassiterite in the wall zone of Marko's pegmatite. Continued crystallization has produced oscillatorily zoned strüverite that grows out from the cassiterite margin. The dark region on the right is quartz. (f) Euhedral grain of cassiterite containing ferrocolumbite- and pore-rich zones alternating with inclusion-free zones criss-crossed with needle-shaped products of exsolution (rutile?). From beryl pegmatite Peg. 15. (g) Cuspate gahnite (mid-green) partially replaced by zinnwaldite? and cut by later veinlets of (red) secondary Fe-oxides (Separation Rapids pluton, 94–29). (h) Elongate tabular crystals of nigerite (pale brown) showing minor zonation, inclusions of cassiterite, and evidence of later deformation that has broken up and bent some of the crystals. The isolated blue crystal is ferrotantalite. From petalite pegmatite Peg. 10. For other colors, see key to Fig. 3.

TABLE 5. STRUVERITE FROM SEPARATION RAPIDS

	Mn-suite Wall Zone of Marko's Pegmatite		
	1	2	3
FeO	7.85	7.07	5.25
MnO	0.07	1.21	5.23
TiO2	58.28	55.51	34.39
Nb2O5	7.77	4.53	8.80
Ta2O5	23.85	30.55	42.49
SnO2	1.40	1.47	3.63
WO3	0.02	0.01	0.00
PbO	0.03	0.02	0.02
ThO2	0.02	0.01	0.00
UO2	0.09	0.02	0.00
Sc2O3	0.04	-	-
Total	99.42	100.40	99.81
No. of oxygens	4	4	4
Fe	0.218	0.201	0.172
Mn	0.002	0.035	0.173
Ti	1.455	1.421	1.011
Nb	0.117	0.070	0.156
Ta	0.215	0.283	0.452
Sn	0.019	0.020	0.057
W	0.000	0.000	0.000
Pb	0.000	0.000	0.000
Th	0.000	0.000	0.000
U	0.001	0.000	0.000
Sc	0.001	-	-
Total	2.027	2.030	2.021

brown, medium brown, pale brown, to a straw-yellow brown. In some samples, all these colors occur in a single crystal in a series of variably spaced concentric zones. Cassiterite from the Separation Rapids pluton and Fe-suite beryl pegmatites is relatively free of inclusions, but in the Fe-suite petalite-bearing pegmatites, it is not unusual for the darker core-regions to be relatively inclusion-free and for a later, lighter brown zone to contain trains of small inclusions of ferrocolumbite near the boundary between the two zones. Where multiple zones occur (such as in beryl pegmatite Peg. 15), there is alternation between ferrocolumbite- and pore-rich zones and other zones lacking inclusions but full of dark, needle-shaped products of exsolution (possibly rutile?). Figure 7f illustrates such an example, in which larger inclusions stand out from the edges of the crystal, having possibly been preferentially left behind after an episode of dissolution.

Cassiterite in the petalite-bearing pegmatites of the Fe-suite hosts inclusions of ferrocolumbite, ferrowodginite, Sc-rich manganocolumbite (or possibly ixiolite?: Fig. 3c), *stibiomicrolite*, rare löllingite and uraninite; usually it is one of the last, if not the last oxide mineral, to crystallize. In pegmatite 7, microlite and a second generation of ferrotantalite (slightly more Mn-rich than the first) seem to be later. In the wall zone of Marko's pegmatite (Mn-suite), cassiterite hosts inclusions of titanowodginite and "ferrotitanowodginite". Elsewhere in Marko's pegmatite, cassiterite is inclusion-free and forms euhedral crystals commonly associated with manganocolumbite and, less commonly, *microlite*.

Overall, cassiterite has a limited range of compositions, and there is considerable overlap in FeO, MnO, Nb_2O_5 and Ta_2O_5 contents among rock types (Table 5). Of these elements, Ta concentrations are highest and range from 0.6 to 1.6 wt.% Ta_2O_5 in the pluton, 0.5 to 2.7 wt.% in the beryl pegmatites, and 0.3 to 4.4 wt.% in most of the petalite pegmatites. Marko's pegmatite has a slightly wider range, 0.2–6.2 wt.%, whereas pegmatite Peg. 6 has Ta values up to 8.6 wt.% Ta_2O_5. Chemical composition seems related to the color of the cassiterite; in the petalite pegmatites, the darker zones typically have $Nb_2O_5 + Ta_2O_5$ contents of 4.5 wt.%, whereas in the lighter zones, they are nearer 1.5 wt.%. There is no systematic variation in cassiterite composition among rock types, but all the data fall along a linear array, with Nb + Ta and Fe + Mn predominantly varying in the ratio of 2:1 corresponding to the coupled substitution: $3\ Sn^{4+} \Leftrightarrow 2(Nb,Ta)^{5+} + (Fe,Mn)^{2+}$. This relationship is common to cassiterite from many rare-element granites and pegmatites worldwide, and distinctly different to those from epithermal and hydrothermal vein deposits (Fig. 8). In addition, cassiterite compositions approaching the 1:1 substitution line suggest the presence of a $Fe^{3+}(Nb,Ta)O_4$ component in some of the cassiterite, consistent with the substitution: $2Sn^{4+} = Fe^{3+} + Ta^{5+}$ (Černý & Ercit 1989).

Ilmenite

The occurrence of ilmenite is restricted to the non-pegmatitic part of the Separation Rapids pluton, where it is rarely found in foliated muscovite – biotite – (garnet) granodiorite, associated with chloritized biotite. Columbite–tantalite-group minerals are, in fact, much more common throughout the Separation Rapids pegmatite field.

Scheelite

Scheelite is found only in one petalite pegmatite (Peg. 7), where it forms very rare 0.1 mm anhedral crystals associated with cassiterite. A high tungsten-carrying capacity in the pegmatite-forming melt or fluid of this and two other petalite pegmatites (Pegs. 5 and 6) is also indicated by the presence of associated tungsteniferous columbite or wodginite (or both).

Uraninite

Geiger counter tests indicate that pegmatite Peg. 7, and in particular its cassiterite-rich zones, is significantly more radioactive than the other pegmatites. Only in this pegmatite and Peg. 265 has uraninite been positively

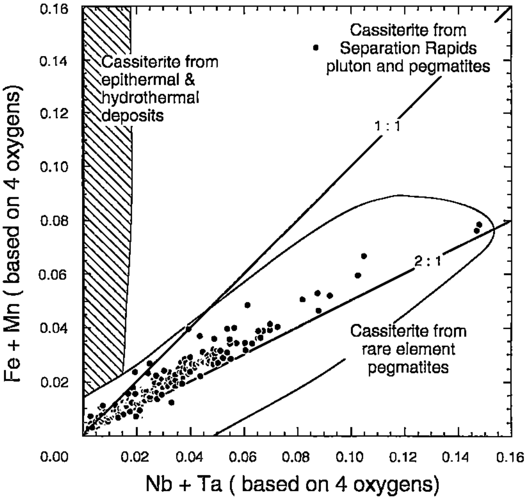


FIG. 8. Covariation of Nb + Ta with Fe + Mn in cassiterite from the Separation Rapids pluton and associated bodies of granitic pegmatite. The rare-element pegmatite field is drawn using data from Beauvoir, France (Wang *et al.* 1988), Jihalva, Czech Republic (Černý & Němec 1995), Pyrenees, Spain (Abella *et al.* 1995), Ririwai, Nigeria (Ixer *et al.* 1987), Sinceni, Swaziland (Trumbull 1995), Rondonia, Brazil (Oen *et al.* 1982), Greer Lake, Manitoba (Černý *et al.* 1986), Tanco, Manitoba (Černý *et al.* 1981), Peerless, South Dakota (Černý *et al.* 1985), and Bob Ingersoll, South Dakota (Spilde & Shearer 1992). The epithermal and hydrothermal field uses data from Cirotan, West Java, Indonesia (Marcoux *et al.* 1993), Rooiberg, South Africa (Ollila 1986) and Cornwall, England (Moore & Howie 1979).

identified. In pegmatite Peg. 265, it occurs as euhedral inclusions in manganocolumbite, whereas in Peg. 7, it occurs as inclusions in cassiterite, where it has caused major radiation-induced damage resulting in radial fracturing and the development of a uraniferous diffusion front. Uraninite may have crystallized in other pegmatites, for relict U–Th–Pb (± Nb–Ta)-rich phases are observed, but these are so affected by metamictization that conclusive identification is impossible.

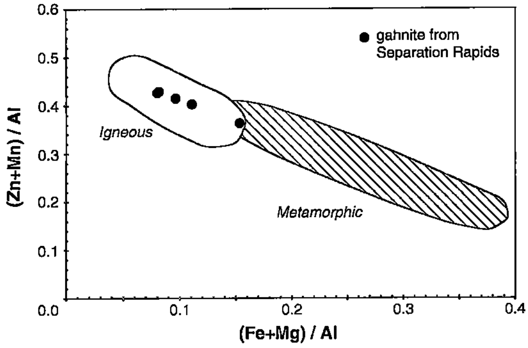


FIG. 9. Covariation of (Fe + Mg)/Al with (Zn + Mn)/Al (atomic ratios) for gahnite from Separation Rapids plotted with igneous and metamorphic gahnite from worldwide localities. Worldwide data from Ontario (Spry 1982), Namaqualand (Moore & Reid 1989), Australia (Parr 1992), Glen Muick, Scotland (Goodman 1993), Poland (Cook & Dudek 1994), in addition to the compilation of Batchelor & Kinnaird (1984).

TABLE 6. CASSITERITE FROM SEPARATION RAPIDS

	Separation Rapids Pluton	Fe-Suite Beryl Pegmatites		Fe-Suite Petalite Pegmatites			Mn-suite Beryl Pegmatites		Mn-suite Petalite Pegmatites		Wall Zone Marko's Pegmatite	
	93-260	Peg. 15	Audrey's	Lou's	James'	Peg. 6	Peg. 265		Marko's		Marko's	
	1	2	3	4	5	6	7	8	9	10	11	12
FeO	0.52	0.19	0.57	0.23	0.51	1.73	0.11	0.71	0.13	0.60	0.06	0.21
MnO	0.05	0.04	0.07	0.03	0.08	0.07	0.10	0.04	0.03	0.20	0.04	0.11
Nb2O5	0.75	0.03	0.21	0.09	0.16	1.19	0.06	0.96	0.07	0.12	0.00	0.40
Ta2O5	0.98	0.57	1.48	1.00	2.33	8.68	1.18	2.85	0.85	4.39	0.37	1.37
SnO2	97.10	99.20	97.97	98.64	96.52	88.07	98.23	95.08	98.76	94.24	99.67	97.96
Total	99.40	100.03	100.30	99.99	99.60	99.74	99.68	99.64	99.84	99.55	100.14	100.05
No. of oxygens	4	4	4	4	4	4	4	4	4	4	4	4
Fe	0.022	0.008	0.024	0.009	0.022	0.073	0.005	0.030	0.005	0.025	0.003	0.009
Mn	0.002	0.002	0.003	0.001	0.003	0.003	0.004	0.002	0.001	0.009	0.002	0.005
Nb	0.017	0.001	0.005	0.002	0.004	0.027	0.001	0.022	0.002	0.003	0.000	0.009
Ta	0.013	0.008	0.020	0.014	0.032	0.120	0.016	0.039	0.012	0.060	0.005	0.019
Sn	1.950	1.985	1.955	1.975	1.943	1.778	1.974	1.908	1.980	1.904	1.992	1.959
Total	0.055	2.003	2.007	0.026	0.061	0.223	0.026	0.092	0.020	0.097	0.009	0.041

Gahnite

Gahnite, $ZnAl_2O_4$, is a widespread accessory mineral in rare-element pegmatites including Greer Lake, Manitoba (Černý *et al.* 1986), central Nigeria (Batchelor & Kinnaird 1984), Greenbushes, Western Australia (Palache *et al.* 1944), Preissac – Lacorne, Quebec (Mulja *et al.* 1995), northern Moravia, Czech Republic (Černý *et al.* 1992) and the Pyrenees, northern Spain (Abella *et al.* 1995). It is also reported from a peraluminous granite in New Zealand (Tulloch 1981). Batchelor & Kinnaird (1984) noted differences in composition between such examples of gahnite and those with a metamorphic origin. The mineral may therefore be an important indicator of hidden rare-element pegmatites in areas of glaciated (or other) terranes where exposure is limited.

At Separation Rapids, gahnite (Table 6, compositions 1 and 2) is found in the Treelined Lake granitic complex and the Separation Rapids pluton, where it occurs as small (0.5–1.5 mm), bright green, subrounded crystals, commonly associated with mica (muscovite or zinnwaldite? or both) and in some cases accompanied by Cd-rich sphalerite (3 wt.% Cd). A few crystals show replacement textures, being totally surrounded and partially replaced by muscovite, or having a cuspate margin (Fig. 7g). There is some evidence that gahnite originally formed larger grains (4–5 mm) and that deformation resulted in break-up of the crystals with growth of a lithium-rich mica (zinnwaldite?) in the resulting pressure-shadows. Perhaps surprisingly, gahnite has not been observed in any of the pegmatites. Gahnite from the Separation Rapids pluton (Fig. 9) is Fe-rich and falls within the igneous field of Batchelor & Kinnaird (1984).

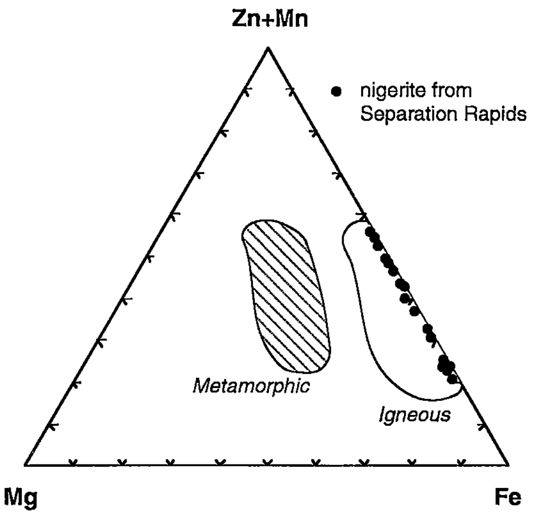


FIG. 10. Triangular diagram showing the composition of nigerite from Separation Rapids, plotted in terms of Zn+Mn – Mg – Fe with igneous and metamorphic nigerite from worldwide localities. Worldwide data from Nigeria (Bannister & Stadler 1947), Siberia (Ginsburg *et al.* 1961), Portugal (van Tassel 1965), Brazil (Kloosterman 1974), Finland (Burke *et al.* 1977), Czechoslovakia (Čech *et al.* 1978), Australia (Grey & Gatehouse 1979), Ontario (Spry 1982, Petersen 1986), Sweden (Schumacher *et al.* 1987) and Tsomtsaub, Omaruru River, Namibia (Tindle, unpubl. data).

TABLE 7. GAHNITE AND NIGERITE FROM SEPARATION RAPIDS

	Separation Rapids Pluton		Mn-suite Petalite Pegmatites			
	Gahnite		Nigerite			
	94-29		Peg. 1	Peg. 9	Peg. 1	Peg. 10
	1	2	3	4	5	6
Al2O3	55.96	55.21	54.14	54.32	55.85	54.80
FeO	11.58	6.01	11.13	11.98	9.47	11.32
MnO	0.37	0.14	0.61	0.40	0.46	0.56
MgO	0.28	0.14	0.00	0.12	0.08	0.08
TiO2	0.02	0.02	0.09	2.46	0.07	0.42
Nb2O5	0.02	0.02	0.32	0.37	0.14	0.36
Ta2O5	0.02	0.08	0.38	0.82	0.21	0.88
SnO2	0.05	0.05	28.60	24.71	21.40	20.00
WO3	0.01	0.00	0.44	0.21	0.19	0.21
ZnO	32.14	37.80	3.29	3.73	12.64	10.69
Total	100.46	99.46	99.00	99.12	100.51	99.32
No. of oxygens	32	32	24	24	32	32
Al	15.846	15.894	11.613	11.478	15.705	15.589
Fe	2.327	1.227	1.694	1.796	1.890	2.285
Mn	0.075	0.029	0.094	0.061	0.093	0.114
Mg	0.100	0.051	0.000	0.032	0.028	0.029
Ti	0.004	0.004	0.012	0.332	0.013	0.076
Nb	0.002	0.002	0.026	0.030	0.015	0.039
Ta	0.001	0.005	0.019	0.040	0.014	0.058
Sn	0.005	0.005	2.075	1.766	2.036	1.925
W	0.001	0.000	0.021	0.010	0.012	0.013
Zn	5.702	6.816	0.442	0.494	2.227	1.905
Total	24.062	24.033	15.997	16.038	22.032	22.033

Structural formula calculated assuming analyses 3, 4 and 5, 6 represent respectively, the 6H and 24R polytypes of nigerite (Grey & Gatehouse 1979)

Nigerite

The rare mineral *nigerite* $(Zn,Fe^{2+})(Sn,Zn)_2(Al,Fe^{3+})_{12}O_{22}(OH)_2$ (6*H* polymorph) was first described by Jacobson & Webb (1947) from quartz–sillimanite rocks associated with tin-bearing pegmatites of the Kabba province, central Nigeria, where it occurs with andalusite, muscovite, gahnite, garnet (probably spessartine–almandine), cassiterite, columbite–tantalite, chrysoberyl and apatite. At Lixa, in the Douro Litoral province of Portugal, nigerite is closely associated with muscovite in a cassiterite-bearing pegmatite that also hosts lithiophilite, apatite and vivianite (van Tassel 1965). Ginsburg *et al.* (1961) described it from a granitic pegmatite vein in eastern Siberia, Kloosterman (1974), from pegmatitic tin–tantalum veins from central Amapá, Brazil, and Burke *et al.* (1977), from granitic pegmatite and associated aplite veins from Kemiö

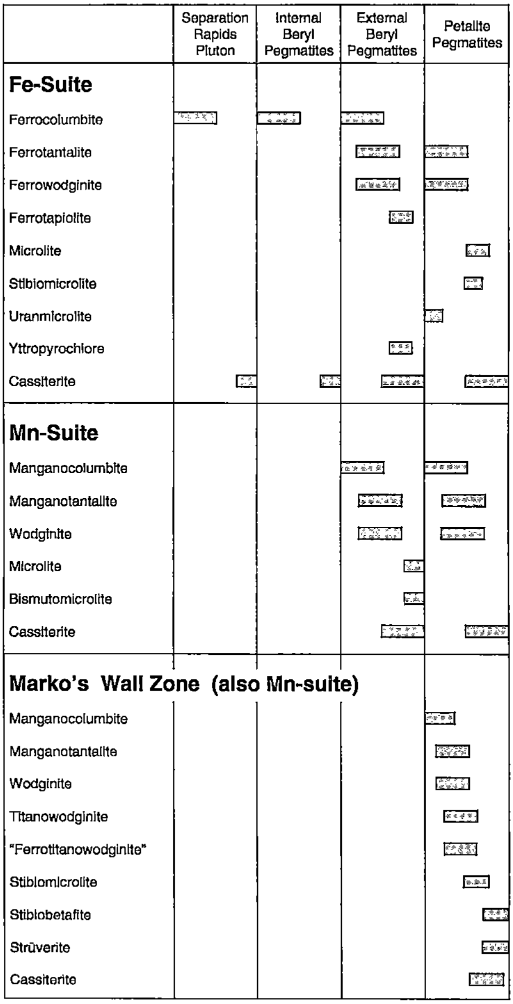


FIG. 11. Paragenesis of Nb–Ta oxides from Separation Rapids.

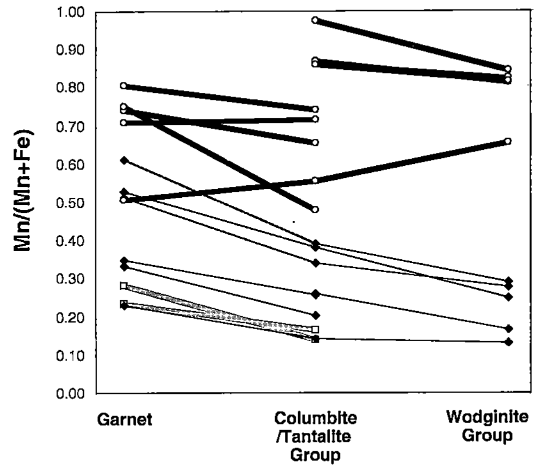


FIG. 12. Covariation of Mn/(Mn + Fe) in garnet, columbite and wodginite-group minerals from Separation Rapids. Shaded line and open squares: Separation Rapids pluton; thin solid line and filled diamonds: Fe-suite pegmatites; thick solid line and open circles: Mn-suite pegmatites.

Island, southwestern Finland. Commonly associated accessory minerals include cassiterite, columbite–tantalite, tourmaline, chrysoberyl, and occasionally andalusite, sillimanite, corundum and garnet. At Amapá, nigerite and cassiterite commonly occur together, but not in the presence of gahnite, whereas at Kemiö Island, nigerite forms oriented overgrowths on gahnite, or in spinel-free samples, the nigerite represents a stage of complete replacement. A lack of gahnite in the Separation Rapids pegmatites may indicate total replacement by nigerite; however, most nigerite forms euhedral or subhedral crystals that seem primary.

Separation Rapids probably represents the first occurrence in North America of nigerite from a granitic pegmatite. It has been found in four of the Fe-suite petalite pegmatites, where it has a distinctive golden brown color and forms small elongate tabular crystals with rounded margins or, less commonly, hexagonal platy crystals (Fig. 7h). In Peg. 1, crystals reach over 4 mm in length, but in other pegmatites, grain sizes are smaller (less than 1 mm). Muscovite or "squi" (spodumene – quartz intergrowth, alteration products after petalite) are commonly associated phases. More rarely, nigerite occurs amongst clusters of ferrocolumbite–ferrotantalite crystals (Peg. 10). In some cases, it is accompanied by cassiterite or spessartine garnet (or both).

Two distinct compositions of nigerite are recognized, a low-Zn variety with 3.2–6.2 wt.% ZnO (Table 6, compositions 3 and 4), and a high-Zn variety, with 10.7–13.0 wt.% ZnO (Table 6, compositions 5 and 6). We tentatively equate these with the 6*H* and 24*R* polytypes (Grey & Gatehouse 1979). Our analytical data indicate that both varieties occur in individual samples.

As with gahnite, nigerite from rare-element pegmatites is distinct from that occurring in metamorphic environments (Fig. 10), such as at the Geco Cu–Zn volcanogenic massive sulfide deposit, Manitouwadge district, Ontario (Petersen 1986).

DISCUSSION

An examination of the oxide minerals in the Separation Rapids pluton and pegmatites, and in particular columbite–tantalite, has revealed two major groupings, an Fe-suite and a Mn-suite. Both beryl- and petalite-bearing granitic pegmatites are found in each suite. The wall zone of one of the Mn-suite pegmatites (Marko's pegmatite) has a sufficiently different assemblage of minerals that it is described separately.

Fe-suite

The Fe-suite includes the Separation Rapids pluton and the majority of beryl- and petalite-bearing granitic pegmatites. The Zn oxides gahnite and nigerite are also restricted to this suite. Zn^{2+} predominantly substitutes for Fe^{2+}, and usually both elements decrease in abundance with progressing igneous fractionation. Nonetheless, gahnite and nigerite still constitute minor accessory minerals, probably in response to the low concentration of ferromagnesian minerals and to the availability of excess Al^{3+} in the Separation Rapids pluton and pegmatites. Černý & Hawthorne (1982) noted that as the fugacity of S^{2-} regulates the form of Zn precipitation, gahnite and sphalerite are mutually exclusive in most of their pegmatite occurrences. In the Separation Rapids pluton, however, they may occur together. The irregular shape of gahnite crystals (Fig. 7g) indicates local disequilibrium and could point to a xenocrystic origin, the gahnite being derived from partial melting of the Treelined Lake granitic complex, where it also is found. However, another possibility is that the shape of the gahnite may have been produced by replacement of mica, which is almost a rule in "pegmatite-born" gahnite. The intergranular fluid is necessarily alkali-bearing in feldspar-dominated pegmatites, and these alkalis vigorously react with any peraluminous minerals (P. Černý, pers. comm.).

Although a distinct succession of Nb–Ta minerals can be recognized within each pegmatite, there is a general sequence from ferrocolumbite to ferrotantalite throughout the suite (Fig. 11). Ferrocolumbite from the Separation Rapids pluton has the lowest Mn and Ta content, and is considered the most primitive. As with the internal beryl pegmatites (which have ferrocolumbite with slightly higher Mn contents), the Separation Rapids pluton does not contain other Nb–Ta oxide species than minor cassiterite.

In most external beryl pegmatites, ferrocolumbite has higher Mn and Ta contents than in the Separation Rapids pluton, and it coexists with ferrowodginite in a few cases. Ferrowodginite becomes a characteristic mineral in the petalite pegmatites, where it coexists with either ferrocolumbite or ferrotantalite, and wodginite is also present in the evolved parts of two of these pegmatites.

There is some petrographic evidence that *microlite* is a primary crystallizing phase, but the mineral also occurs as a replacement product after ferrowodginite or manganocolumbite. The other minerals of the microlite subgroup, *uranmicrolite*, *antimonian microlite* (with one exception: Fig. 6f) and *stibiomicrolite* only occur as inclusions (the first in ferrowodginite, the Sb-bearing species in cassiterite) and are therefore difficult to place in a paragenetic sequence. The *uranmicrolite* and some *antimonian microlite* display primary textural features, such as euhedral habit and oscillatory zoning.

If the oxides had been significantly disturbed by fluids, it might be expected that they would exhibit a variable composition. It was therefore decided to analyze garnet (chosen as a relatively stable, primary mineral that most likely crystallized directly from a pegmatite-forming melt) and compare Mn/(Mn + Fe) values for garnet-, columbite–tantalite- and wodginite-group minerals (Fig. 12). With little deviation, the Nb–Ta oxides and the garnet change composition systematically; there can be little doubt that these minerals crystallized in local equilibrium under identical (magmatic) conditions, and that they were not seriously affected by later fluids. Even albite-rich units in the Separation Rapids pluton and pegmatites from this suite show little evidence of replacement features, and all members of the Fe-suite are therefore interpreted to consist of primary magmatic assemblages.

Mn-suite

Two large bodies of pegmatite from the eastern subgroup essentially define this suite, one an external beryl-bearing pegmatite (Peg. 265), the other a petalite-bearing pegmatite (Marko's). Two other external beryl pegmatites are included in the Mn-suite, but neither the internal beryl pegmatites, nor the Separation Rapids pluton (apart from the "cleavelandite"-rich pods) are represented.

The suite is characterized by early-formed (petalite-free) units of individual pegmatites containing manganocolumbite with appreciable variation in Mn/(Mn + Fe) within individual samples, followed by manganocolumbite and manganotantalite with appreciable variation in Ta/(Ta + Nb) in more evolved (petalite-rich in Marko's pegmatite) units. In these later units, wodginite coexists with manganocolumbite and manganotantalite, but spessartine (which can be locally abundant in the early crystallized units) is absent in the later units.

Microlite is restricted to Marko's pegmatite, where it locally occurs in abundance. As with the Fe-suite, most microlite is secondary after wodginite, but where no evidence exists of a possible precursor phase, it is likely that this microlite is primary. *Bismutomicrolite* in pegmatite Peg. 265 is, however, secondary, clearly replacing earlier (tungsteniferous) wodginite. Cassiterite is the final oxide to crystallize. The positive correlation of Na with F noted at Separation Rapids, particularly in the Mn-suite pegmatites and also reported in microlite from the Harding pegmatite, New Mexico, suggests systematic variation due to magmatic fractionation or a degree of primary alteration of the microlite itself during the late magmatic to hydrothermal stages of pegmatite emplacement (Lumpkin & Ewing 1992).

A comparison of garnet with Nb–Ta oxides does not reveal the same systematic variation as that observed in the Fe-suite (Fig. 12), even though many samples do define broadly similar trends. This may be related to albitization, which is particularly noticeable in the manganotantalite-bearing samples and discussed further in Tindle *et al.* (1998). Although there is petrographic evidence to suggest that albitization is a metasomatic phenomenon capable of producing subsolidus replacement-induced textures, such as albite-rich pods replacing earlier petalite crystals, we agree with London *et al.* (1989) and Černý & Lenton (1995) that most late albite-rich and micaceous units probably represent the very last residual melt and should be considered part of the fractionation sequence (*i.e.*, are magmatic). However, it is not possible to consistently distinguish assemblages formed as late magmatic replacements from those that may be entirely subsolidus, as a spectrum of textural features is evident in the field.

The rapid nucleation and resulting fine grain-size of these late albite-rich units can be triggered by nucleation of B, P, Li and F-bearing minerals (London 1990), which drastically reduces the solubility of H_2O in the residual melt and leads to exsolution of a supercritical fluid. Topaz-rich aplitic dykes associated with pegmatite 304 are a clear example of where this is likely to have occurred.

We note that although primary albite-bearing assemblages are common to both Fe- and Mn-suite pegmatites, it is only in the Mn-suite pegmatites that albite-rich replacement units are well developed. We speculate that this was a consequence of differences in F contents of the parental pegmatite-forming melts. Pegmatite-forming melts can, however, evolve toward an alkaline, silica-depleted composition rich in Na, F and P, and become vapor-saturated, with resulting increase in solidus temperatures and the formation of fine-grained, aplitic albite–mica bodies (London 1990). The Mn-suite pegmatites are most likely to have achieved vapor saturation, but there is less evidence for it in the Fe-suite pegmatites. This could, therefore, be another factor contributing to the differences between the two suites.

On the basis of all our observations, the parent of the Fe-suite pegmatites is considered to be the Separation Rapids pluton. Those pegmatites containing ferrocolumbite–ferrotantalite with low Mn/(Mn + Fe) values (such as pegmatites 9 and 10) are considered to be early. As the Separation Rapids magma fractionated, Mn/(Mn + Fe) increased in both pluton and later pegmatites. Columbite–tantalite variation within individual pegmatites mainly involves Ta/(Ta + Nb), and this is ascribed to *in situ* fractionation.

The Mn-suite pegmatites are more difficult to explain, as there is no obvious parent. Spatially, the suite is intimately associated with the Fe-suite pegmatites. The diverging columbite–tantalite trends and the lack of a continuum of K-feldpar compositions (Breaks *et al.* in prep.) preclude a simple fractionation model linking the two suites. There is, however, the possibility that the Mn-suite pegmatites were derived from a thermogravitationally separated F-rich layer within the Separation Rapids magma chamber, as K-feldpar compositions (Breaks *et al.*, in prep.) indicate that the Separation Rapids granite is compositionally layered, with extreme Cs (> 6000 ppm) and high Rb (>150 ppm) values along the southwestern margin. In this situation, columbite compositions from pegmatites 24 and 262 (Fig. 2a) could be interpreted as being derived from a boundary layer between the sources of the relatively F-poor Fe-suite and the relatively F-rich Mn suite.

At this time, there is insufficient evidence to discriminate between a thermogravitationally layered magma chamber and one where batches of more evolved, F-rich, sodic residual melt ponded in the apical part of the Separation Rapids pluton. It is also not possible to discount the possibility that the Mn-suite pegmatites were derived from a totally independent F-rich source, although there is no field evidence to support the existence of such a second pluton.

The wall zone of Marko's pegmatite, a petalite-absent pollucite – beryl – muscovite – albite – quartz unit, is considered here separately from the main Mn-suite, because of its unique assemblage of minerals. It also differs from other Mn-suite pegmatites and the remainder of Marko's pegmatite in not containing any albite-replacement pods or other macro-scale replacement assemblages. Manganocolumbite and manganotantalite are both present, but show little systematic variation. However, wodginite in the wall zone shows evidence of alteration and replacement, with one large crystal displaying a texturally complex titanowodginite and "ferrotitanowodginite" rim (Fig. 3d) comparable to that reported in secondary columbite from Finland (Lahti 1987), and secondary columbite–tantalite from the Czech Republic (Černý *et al.* 1992). One inclusion of titanowodginite at the edge of a large grain of cassiterite has been partially replaced by strüverite (tantalian rutile). These features cannot be explained by normal processes of fractionation (although extreme fractionation is required to crystallize pollucite). We thus propose that there also was interaction with the host rocks (banded iron-formation and Fe–Ti tholeiitic metavolcanic rocks), which led to an influx of Fe and Ti into the pegmatite-forming melt. We speculate that this influx occurred whilst the pegmatite was crystallizing, as other titanowodginite and "ferrotitanowodginite" crystals found in wallrock samples occur as inclusions in what would appear to be magmatic cassiterite. Host rocks may also have contributed to the Sb and Ti enrichment of pyrochlore-group minerals on a localized scale. Our conclusions are supported by those of Abella *et al.* (1995), who considered the restricted occurrence of niobian and tantalian rutile in the border and wall zones of pegmatites from the Cap de Creus pegmatite field, eastern Pyrenees, as being due to host-rock assimilation.

The evidence presented above suggests that at Separation Rapids, physicochemical variations due to halogen and alkali enrichment at the end of magmatic differentiation, coupled with interaction of magmatic and extraneous fluids, led to conditions of non-equilibrium crystallization (Ohnenstetter & Piantone 1992) in the Mn-suite pegmatites. Locally there also was interaction with host rocks.

CONCLUSIONS

Oxide minerals have been characterized from the Separation Rapids pluton and its associated pegmatites. Compositions of the Nb–Ta minerals, associated minerals (petalite, beryl, gahnite, nigerite, zircon, albite, Li-rich mica, garnet) and other geological and petrological data (Breaks *et al.* in prep., Tindle *et al.* 1998) confirm these pegmatites belong to the rare-element class, complex type, petalite subtype.

The following oxide minerals were identified: ferrocolumbite, ferrotantalite, manganocolumbite, manganotantalite, wodginite, ferrowodginite, titanowodginite, "ferrotitanowodginite", microlite, antimonian microlite, stibiomicrolite, uranmicrolite, bismutomicrolite, stibiobetafite, yttropyrochlore, ferrotapiolite, strüverite, cassiterite, ilmenite, scheelite, uraninite, gahnite and nigerite. Ixiolite also was tentatively identified.

On the basis of compositions of columbite-group minerals, the pegmatites can be divided into an Fe-suite and a Mn-suite, both containing beryl and petalite pegmatites. The suites are not spatially separable in the field.

The Separation Rapids pluton has primitive compositions of ferrocolumbite consistent with it being the parent to at least the Fe-suite of pegmatites. The ferrocolumbite–ferrotantalite trends, as well as the restricted occurrence of microlite-group minerals and other F-rich minerals, indicate a rather F-poor environment.

Late-stage alteration of this suite is considered to be minimal, as coexisting garnet, columbite–tantalite and wodginite-group minerals show systematic variation inconsistent with major influx of late fluids.

The Mn-suite has quite different characteristics indicative of a more F-rich environment. These include a greater abundance of microlite, the presence of topaz in metasomatic selvedges (up to 8.5 wt.% F), associated topaz-rich aplite dykes, concentrations of Li–F micas in evolved pegmatites, and field and textural evidence for replacement and albitization.

The mineral assemblage found in the wall zone of Marko's pegmatite provides evidence for local interaction of the pegmatite-forming magma with Fe-, Ti- and probably Sb-rich host rocks.

In summary, the Nb–Ta oxides are shown to be excellent indicators of the chemical evolution of the leucogranitic and pegmatite-forming melts. Unfortunately, experimental data essential for a more detailed interpretation of the conditions of crystallization are lacking, but they are very desirable.

ACKNOWLEDGEMENTS

Kay Chambers, Brian Ellis and John Watson are thanked for producing innumerable polished thin sections and blocks for electron-microprobe analysis. Thanks are also due to Mrs. Saga von Knorring for providing nigerite-bearing samples from the collection of her late husband (Dr. Oleg von Knorring). Petr Černý is thanked for his indirect influence on this line of research. He, Greg Lumpkin and Peter Webb are thanked for very thorough reviews of earlier versions of this manuscript. AGT thanks the Mineralogical Society for a Senior Travel Bursary, and the Ontario Geological Survey for support in the field.

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Received July 8, 1996, revised manuscript accepted December 16, 1997.