

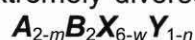
# Plumbomicrolite from the Heftetjern granitic pegmatite, Tørdal, Telemark, south Norway

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## Introduction

A lead-rich microlite was described from an alluvial cassiterite deposit in Kivu, Congo, by Safiannikoff & Van Wambeke (1961). Although they use the name plumbomicrolite (in French) in two places, they suggest that the Commission on New Minerals of the International Mineralogical Association (IMA) ought to examine the nomenclature of the pyrochlore-microlite family and decide if the mineral they describe should receive a specific name. Based on the preliminary naming of the mineral, Hey (1964, p. 1147) introduced the name plumbomicrolite which has since persisted in the literature. Back & Mandarino (2008) report a more exact type locality: Mumba tin gravels, Masisi, Kivu Province, Democratic Republic of the Congo. 'Mumbite' (Van Wambeke 1970, p. 121) is an obsolete synonym of plumbomicrolite (Hogarth 1977).

Pyrochlore supergroup minerals are extremely diverse in composition and have the general formula



where **A** is typically a large eight-coordinated cation (Na, Ca, Fe<sup>2+</sup>, Sn<sup>2+</sup>, Pb, Y, Ce and other REEs, U<sup>4+</sup>, etc.),

**B** is a smaller six-coordinated cation (Nb, Ta, Ti, Sn<sup>4+</sup>, Fe<sup>3+</sup>, Al, etc.),

**X** is mainly O and **Y** is typically OH, F or O.

H<sub>2</sub>O can be incorporated at the **A** and **Y** sites, and the formula indicates the possibility of incomplete occupancy of the **A**, **X** and **Y** sites. Consequently, the empirical formulae of pyrochlore minerals should be calculated on the basis of  $B = 2$ .

A paper describing the currently accepted classification and nomenclature of pyrochlore minerals was published by Hogarth (1977). Three subgroups were established, based on the atomic proportions of the **B** atoms:

- Pyrochlore subgroup with  $Nb + Ta > 2Ti$  and  $Nb > Ta$ ;
- Microlite subgroup with  $Nb + Ta > 2Ti$  and  $Ta > Nb$ ;
- Betafite subgroup with  $2Ti \geq Nb + Ta$ .

This tripartite division is not consistent with the general IMA guidelines for mineral nomenclature (Nickel 1992).

Within the subgroups, species are defined with respect to the content of **A** atoms:

- If the sum of Na + Ca exceeds 20% of the total **A** atoms, no prefix is added to the root name. This means that Hogarth's (1997) system does not differentiate between occupation by Ca and Na.
- For other species, one or more **A** atoms other than Na and Ca shall exceed 20% of the total **A** atoms (for purposes of species definition, the sum of Y + REEs counts as one **A** atom). The species is named by adding the appropriate prefix to the root name, e.g. plumbomicrolite.

An atomic content of 20% at the **A** site corresponds to 0.4 apfu (atoms per formula unit) of a fully occupied site. Obviously, the official pyrochlore classification system of Hogarth (1977) does not comply with the IMA rules (Hatert & Burke 2008). To rectify the situation, the Commission on New Minerals, Nomenclature and Classification (CNMNC) of the IMA has established a subcommittee to deal with the classification and nomenclature of the (much expanded) pyrochlore supergroup. The CNMNC

members have, during 2009, commented and voted on the proposal presented by the subcommittee. However, the proposed naming scheme (cf. Andrade & Atencio 2008) was not approved, and the report is suspended at the present time. At any rate, it is clear that the subgroup division and the 20% rule of Hogarth (1977) have to be abandoned.

### Analytical details

Wavelength-dispersive analyses were performed in September 1999 with a Cameca Camebax electron microprobe housed at the Mineralogical-Geological Museum in Oslo, using 15 kV operating voltage and 17.4 nA probe current. The counting time was 10 s for peak and background, except for Al (20 s) and Fe (20 s using a pulse-height analyser). The probe standards and measured peaks were: albite (Na  $K\alpha$ ), wollastonite (Ca  $K\alpha$ ), PbS (Pb  $M\alpha$ ),  $Al_2O_3$  (Al  $K\alpha$ ),  $Fe_2O_3$  (Fe  $K\alpha$ ),  $MnTiO_3$  (Ti  $K\alpha$ ),  $SnO_2$  (Sn  $L\alpha$ ), a Ca-Al-silicate glass with 15%  $UO_2$  (U  $M\alpha$ ), Nb metal (Nb  $L\alpha$ ) and Ta metal (Ta  $M\alpha$ ). The elements K, Sc, Y, W and F were sought but found to be below the detection limit.

### Results

The analysed samples occur in the Heftetjern granitic pegmatite as brownish to greenish octahedra, less than 1 mm in size, often displaying etched surfaces. Sample HF53-98 was provided by Roy Kristiansen; the analytical data are presented in Table 1, and back-scattered electron (BSE) images are shown in Fig. 1a-c. Sample AH1 was supplied by Astrid Haugen; analytical data for this sample are given in Table 2, and a BSE image is displayed in Fig. 1d. The occurrence of plumbomicrolite in the Heftetjern pegmatite was briefly mentioned by Raade & Kristiansen (2000).

Iron is assumed to be trivalent ( $Fe_2O_3$  in Tables 1 and 2). It is known that divalent tin ( $Sn^{2+}$ ) may enter the *A* site in pyrochlore group minerals (stannomicrolite). However, tin may also be quadrivalent ( $Sn^{4+}$ ) and located at the *B* site. The latter situation is most likely for pyrochlore group minerals from the Heftetjern occurrence, where cassiterite has been found (Bergstøl & Juve 1988), and a triclinic titanite is reported to contain 5.87 wt%  $SnO_2$  (Lussier et al. 2009).

Low summations are quite normal in EMP analyses of pyrochlore minerals, mainly due to the lack of  $H_2O$  determination. Empirical cation compositions have been calculated on the basis of  $\Sigma (Al + Fe^{3+} + Ti + Sn^{4+} + Nb + Ta) = 2$  (Tables 1 and 2). The resulting atom contents of the *A* position are well below the theoretical value of 2, indicating the presence of  $H_2O$  and vacancies at this site.

#### Sample HF53-98

Fig. 1a shows that this sample is extremely inhomogeneous. By higher magnification, the left, nearly black part is seen to consist of dark patches surrounded by lighter veins (Fig. 1b). EDS and WDS scans of these dark areas indicate the same elements as found in the lighter, right parts of the crystal (Ta, Nb, Ti, Ca, Al, Fe and U) plus appreciable amounts of Si.

The right part of the crystal shows three distinct zones, numbered I to III from the interior to the rim (Fig. 1c). Analytical data from each zone are presented in Table 1. With high Ta contents, all these analyses correspond to members of the microlite subgroup. Zone I is patchily inhomogeneous, the darker areas (points 7 and 9) showing lower PbO contents than the lighter areas (points 8 and 10). All four points of zone I correspond to plumbomicrolite following the Hogarth (1977) classification, but this would not be so if a 'modern' classification were used, Ca being the most

abundant element at the A site. Zone II is also inhomogeneous, on a finer scale than zone I. The nine analyses of zone II are arranged in increasing order of PbO content in Table 1. With all of them having Pb > 0.4 apfu, they classify as plumbomicrolites according to Hogarth (1977). Only the points 2, 13, 6, 12 and 11 have Pb > Ca. Also zone III shows fine-scaled inhomogeneity, the dark parts (points 14 and 17) corresponding to microlite and the light spots (points 15 and 16) to plumbomicrolite. All A site sums being significantly greater than 1 apfu, zero-valent constituents (H<sub>2</sub>O and vacancies) are not dominant. Zone III is relatively enriched in Sn, whereas zone I and parts of zone II are enriched in U. There seems to be an erratic variation in Ti among the three zones.

#### Sample AH1

Three zones can be distinguished in this sample (Fig. 1d); the analytical data are presented in Table 2. All analyses show a dominance of Ta at the B site. Zone I (point 18 from the upper dark area and point 19 from the lower dark area) is very low in PbO; the analyses correspond to microlite. Point 18 has an A site content of only 0.50 apfu and must be zero-valent-dominated. Zone II is also microlite with slightly higher PbO contents than zone I; it is quite homogeneous in composition. It is peculiar in having relatively high contents of Na. Zone III is inhomogeneous, the darker patches (point 25) being microlite, whereas the light parts (points 23 and 24) are plumbomicrolite with Pb > Ca. Zone III is depleted in U relative to zones I and II and is enriched in Ti.

### **Discussion**

#### World-wide occurrences

Plumbomicrolite seems to be reported from only a restricted number of localities. From its type locality at Mumba, Kivu, Congo, the mineral was reanalysed by Eid & von Knorring (1977), with similar results. Eid & von Knorring (1977) also presented analytical data on microlites from several African granitic pegmatites; these are mostly 'normal' Ca-Na-dominant microlites. Analytical data for a sample of plumbomicrolite from "Africa (Zaire)" are given by Voloshin & Pakhomovskii (1986). Judging from the similar composition, this is probably the Mumba locality. Plumbomicrolites from pegmatites in the central part of Madagascar were analysed by Eremenko et al. (1987, Table 3). Granitic pegmatites of the Kafubu area, Zambia, are hosts of plumbomicrolite and several other Nb-Ta minerals (Seifert et al. 2004).

The first find of plumbomicrolite in the former USSR was made in an amazonite pegmatite at Mount Ploskaya, Kola Peninsula (Stepanov et al. 1982). New analytical data and a crystal structure refinement of the Ploskaya mineral were published by Bindi et al. (2006). The results of several analyses of Nb-rich plumbomicrolite from the Kola Peninsula are found in Voloshin & Pakhomovskii (1986); a sample of plumbomicrolite from the Urals is also included.

Plumbomicrolite with 21.2 wt% PbO occur in a pegmatite on Utö in the Stockholm archipelago, Sweden (Smeds & Černý 1989). Another locality of the mineral is the Prašivá field of granitic pegmatites, Slovakia (Uher et al. 1998). Plumbomicrolite and several other pyrochlore group minerals are reported from the Borborema pegmatitic province, northeast Brazil (Beurlen et al. 2005).

According to the survey presented above, plumbomicrolite is almost exclusively reported from granitic pegmatites. Its alluvial occurrence at the type locality could possibly be derived from a similar source. The forthcoming introduction of a new classification of the pyrochlore supergroup will undoubtedly change the status of the minerals described as plumbomicrolite in the literature; they will in the future be classified as several different species.

### Pyrochlore group minerals at Heftetjern

Bergstøl & Juve (1988) described several pyrochlore group minerals from Heftetjern as alteration products of Sc-rich ixiolite with up to 18.80 wt% Sc<sub>2</sub>O<sub>3</sub>. These include microlites rich in Sc, Y, Sn and U, scandian ytropyrochlore and uranian ytrobetafite. Their analysis No. 1 in Table 3 (uranian "scandium microlite") is in fact zero-valent-dominated. The alteration of ixiolite to pyrochlore is described as a "late magmatic-hydrothermal event".

Plumbomicrolite from Heftetjern is not Sc-bearing and is not connected with the alteration of ixiolite. It occurs as discrete crystals or clusters of crystals in vugs of albite. The time of formation of these crystals relative to the alteration process of ixiolite is difficult to constrain.

### Discussion of zoning

The pervasively altered part of sample HF53-98 (Fig. 1a and 1b) probably originated in a late-hydrothermal phase from percolating, Si-rich fluids; owing to a strong inhomogeneity, it was not studied in detail. The sample displays a conspicuous zoning (Fig. 1a and 1c), and zone III undoubtedly corresponds to the outer rim of the crystal. All three zones are rich in Pb, especially the light part of zone III (Table 1). Whether the mottled appearance of the three zones, and especially that of zone III, is a primary or secondary feature, is hard to tell.

Zone III of sample AH1 (Fig. 1 d) is interpreted as the outer rim of the crystal. It is the only part of the crystal that is enriched in Pb (Table 2). The darker, relatively Pb-poor areas of zone III (cf. point 25 in Table 2), occurring closest to the border of the crystal, may be the result of secondary alteration.

It might be worthwhile to mention that in a study of pyrochlore from the Gjerdingen ekerite, Oslo Region, plumbopyrochlore was shown to be confined to the outer rim of the crystal, whereas, in this case, the inner parts correspond to ceriopyrochlore-(Ce) (Raade 2009). This is similar to the Pb enrichment at the rims of the Heftetjern microlite crystals.

### **Acknowledgements**

Muriel Erambert is thanked for her proficient assistance with the microprobe analyses. Astrid Haugen and Roy Kristiansen kindly provided material for investigation. Roy Kristiansen has been very helpful with literature information.

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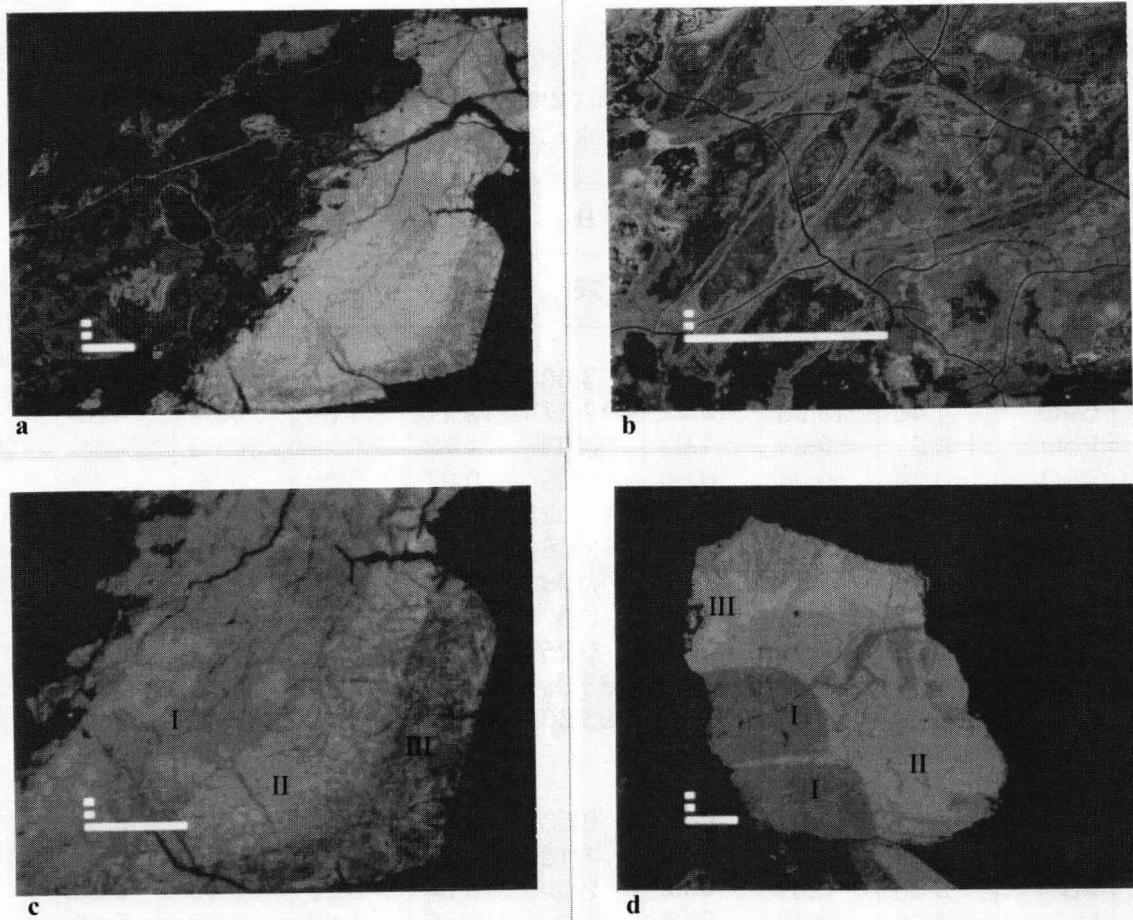
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**Table 1.** Electron-microprobe analytical data (wt%) of microlite minerals from Heftetjern, sample HF53-98, and formula calculation based on  $\Sigma B = 2$ .

Zone	I	I	I	I	II	II	II	II	II	II	II	II	II	III	III	III	III
No.	7	9	8	10	5	4	3	1	2	13	6	12	11	14	17	15	16
Na <sub>2</sub> O	0.17	0.04	0.55	0.09	0.15	0.10	0.07	0.06	0.12	0.11	0.07	0.13	0.12	0.27	0.18	0.09	0.09
CaO	6.80	6.46	6.66	7.05	6.59	6.91	6.59	5.88	5.37	6.01	4.88	4.81	4.09	9.36	8.83	4.75	3.68
PbO	15.77	18.13	22.53	25.15	15.33	18.37	20.13	22.80	23.80	25.29	30.57	38.00	39.55	12.13	12.70	34.64	39.16
Al <sub>2</sub> O <sub>3</sub>	0.00	0.00	0.00	0.05	0.01	0.00	0.00	0.02	0.04	0.04	0.02	0.02	0.04	0.02	0.04	0.00	0.03
Fe <sub>2</sub> O <sub>3</sub>	0.04	0.22	0.17	0.35	0.16	0.24	0.16	0.24	0.18	0.10	0.27	0.18	0.23	0.07	0.10	0.26	0.16
TiO <sub>2</sub>	1.87	2.29	2.34	2.59	1.92	2.40	2.96	2.25	3.95	5.04	4.95	4.06	4.40	6.63	6.75	4.95	4.25
SnO <sub>2</sub>	1.12	1.24	1.10	1.51	0.95	1.29	0.95	1.14	1.62	2.09	2.17	1.89	2.05	3.16	3.31	2.59	2.96
UO <sub>2</sub>	2.69	3.08	3.01	2.08	2.97	2.72	2.97	2.21	0.45	0.06	0.00	0.01	0.00	0.14	0.28	0.11	0.00
Nb <sub>2</sub> O <sub>5</sub>	4.75	5.18	5.17	5.66	5.09	5.25	5.65	4.97	5.44	7.67	7.70	6.24	7.14	13.39	13.35	9.81	7.86
Ta <sub>2</sub> O <sub>5</sub>	49.38	46.66	51.18	43.42	50.18	51.86	48.18	51.25	45.52	39.76	33.89	39.46	36.45	40.62	39.91	32.10	33.93
Sum	82.59	83.30	92.71	87.95	83.35	89.14	87.66	90.82	86.49	86.17	84.52	94.80	94.07	85.79	85.45	89.30	92.12
Na	0.04	0.01	0.11	0.02	0.03	0.02	0.01	0.01	0.03	0.02	0.02	0.03	0.03	0.04	0.03	0.02	0.02
Ca	0.83	0.80	0.77	0.88	0.79	0.78	0.77	0.68	0.62	0.68	0.60	0.59	0.50	0.86	0.81	0.56	0.46
Pb	0.49	0.56	0.65	0.79	0.46	0.52	0.59	0.66	0.69	0.72	0.94	1.17	1.22	0.28	0.29	1.03	1.22
U	0.07	0.08	0.07	0.05	0.07	0.06	0.07	0.05	0.01	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00
$\Sigma A$	1.43	1.45	1.60	1.74	1.35	1.38	1.44	1.40	1.35	1.42	1.56	1.79	1.75	1.18	1.14	1.61	1.70
Al	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00
Fe <sup>3+</sup>	0.00	0.02	0.01	0.03	0.01	0.02	0.01	0.02	0.01	0.01	0.02	0.02	0.02	0.00	0.01	0.02	0.01
Ti	0.16	0.20	0.19	0.22	0.16	0.19	0.24	0.18	0.32	0.40	0.43	0.35	0.38	0.43	0.43	0.41	0.37
Sn <sup>4+</sup>	0.05	0.05	0.05	0.07	0.04	0.05	0.04	0.05	0.07	0.09	0.10	0.09	0.09	0.11	0.11	0.12	0.14
Nb	0.25	0.27	0.25	0.30	0.26	0.25	0.28	0.24	0.26	0.36	0.40	0.32	0.37	0.52	0.52	0.49	0.41
Ta	1.54	1.46	1.50	1.37	1.53	1.49	1.43	1.50	1.33	1.14	1.05	1.22	1.13	0.94	0.93	0.96	1.07
$\Sigma B$	2.00	2.00	2.00	2.00	2.00	2.00	2.00	1.99	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00

**Table 2.** Electron-microprobe analytical data (wt%) of microlite minerals from Heftetjern, sample AH1, and formula calculation based on  $\Sigma B = 2$ .

Zone	I	I	II	II	II	III	III	III
No.	18	19	21	20	22	25	23	24
Na <sub>2</sub> O	0.04	0.04	3.14	3.08	3.12	2.35	0.06	0.36
CaO	4.40	13.08	12.02	12.37	12.47	11.54	5.30	4.67
PbO	0.21	0.24	1.03	0.93	1.73	9.20	30.90	31.05
Al <sub>2</sub> O <sub>3</sub>	0.36	0.24	0.05	0.03	0.07	0.08	0.03	0.10
Fe <sub>2</sub> O <sub>3</sub>	0.71	2.57	0.07	0.02	0.03	0.04	0.06	0.26
TiO <sub>2</sub>	3.17	2.93	2.47	2.46	2.45	4.95	6.78	5.88
SnO <sub>2</sub>	3.44	3.11	2.75	2.78	2.42	4.90	2.21	2.30
UO <sub>2</sub>	5.42	5.11	3.14	3.86	2.94	0.00	0.99	1.29
Nb <sub>2</sub> O <sub>5</sub>	15.06	15.96	5.30	5.25	5.93	12.31	9.43	6.98
Ta <sub>2</sub> O <sub>5</sub>	46.17	38.67	62.20	62.90	62.43	42.79	31.10	37.10
Sum	78.98	81.95	92.17	93.68	93.59	88.16	86.86	89.99
Na	0.01	0.01	0.54	0.53	0.54	0.40	0.01	0.07
Ca	0.39	1.20	1.15	1.18	1.18	1.08	0.60	0.53
Pb	0.00	0.01	0.02	0.02	0.04	0.22	0.89	0.88
U	0.10	0.10	0.06	0.08	0.06	0.00	0.02	0.03
$\Sigma A$	0.50	1.32	1.77	1.81	1.82	1.70	1.52	1.51
Al	0.04	0.02	0.01	0.00	0.01	0.01	0.00	0.01
Fe <sup>3+</sup>	0.04	0.16	0.00	0.00	0.00	0.00	0.01	0.02
Ti	0.20	0.19	0.17	0.17	0.16	0.32	0.54	0.47
Sn <sup>4+</sup>	0.11	0.11	0.10	0.10	0.09	0.17	0.09	0.10
Nb	0.57	0.62	0.21	0.21	0.24	0.48	0.45	0.33
Ta	1.04	0.90	1.51	1.52	1.50	1.01	0.90	1.07
$\Sigma B$	2.00	2.00	2.00	2.00	2.00	1.99	1.99	2.00



**Fig. 1.** Back-scatter electron (BSE) images of microlite minerals from Heftetjern: sample HF53-98 (**a-c**) and sample AH1 (**d**). Image **b** shows a detail of the dark part of image **a**. Image **c** is an enlargement of the zoned, right part of image **a**. The scale bar is 100  $\mu\text{m}$ . Analytical data from zones I-III are given in Table 1 for sample HF53-98 and in Table 2 for sample AH1.