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Composition and probable ore igneous rocks source of columbite from alluvial deposits of Mayoko district (Republic of the Congo)

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The article presents the results of optical, electron microscopic and electron microprobe studies of columbite group minerals, collected during heavy mineral concentrate sampling of alluvial deposits in the Mayoko region (Republic of the Congo). The aim of the study is revealing tantalum niobates ore body in this region. We found that these minerals in loose deposits are represented by two grain-size groups: less than 1.6 mm (fine fraction) and 1.6-15 mm (coarse fraction). The grains of both fractions belong mainly to columbite-(Fe), less often to columbite-(Mn), tantalite-(Mn) and tantalite-(Fe), contain impurities of Sc, Ti, and W. The crystals have micro-scaled zoning (zones varies slightly in the Ta/Nb ratio values) and contains a lot of mineral inclusions and veins represented by zirc con, pyrochlore supergroup minerals and others. Columbite-(Fe) and columbite-(Mn) are characterized by an increased content of Ta_2O_5 up to the transition to tantalite-(Fe) and tantalite-(Mn). This allows us to exclude the formation of sub-alkaline rare-metal granites, their metasomatites (albitites and greisenes) and carbonatites, from the list of possible columbite ore rocks source in the Mayoko district. Thus, beryl type and complex spodumene subtype rare-element pegmatites of the mixed petrogenetic family LCT-NYF (according to P.Černý) should be considered as a probable root source. The results of the research should be taken into account when developing the methodology for prospecting in this area.

Keywords: columbite group minerals; columbite-(Fe) – ferrocolumbite; columbite-(Mn) – manganocolumbite; tantalite-(Mn) – manganotantalite and tantalite-(Fe) – ferrotantalite; alluvial placers; rare metal pegmatites; Mayoko area; Republic of the Congo

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Introduction. In 1945, during the gold placers development, columbite^{*} grains along with gold were found in the heavy fraction of alluvial deposits of modern river valleys in the Mayoko region, located in the south-west of the Republic of the Congo. Since then, no data on the ore igneous rocks source of this tantalum-niobium-bearing mineral have appeared in the materials of geological organizations of the Republic of the Congo. This find has stand out for a long time in connection with the problem of the rare metal deposits exploration facing the geological service of the Republic of the Congo. Major niobium and tantalum deposits within the African continent are known in neighboring territories with a similar geological structure – in the Democratic Republic of the Congo, Rwanda and other countries. Therefore, the study results presented in this article correspond to the quite urgent task of determining the nature of the probable ore body of this very valuable mineral raw material and taking these data into account when choosing the optimal exploration methodology in this area.

The studies are based on the results of grain-size classification, analysis of chemical composition of columbite and its grains heterogeneity in ten stream sediment samples, collected during heavy mineral concentrate sampling and geological traversing of the area.

Location and geology of Mayoko area. Mayoko district is located in the Niari department in the rainforest area covering the Chaillu granitoid massif. This massif is a part of the Congo Craton and can be traced in the territories of both the Republic of the Congo and neighboring Gabon. The geology of this massif is poorly studied. Route exploration of Cosson, Devigne, Donnot, Boineau,

 $^{^{*}}$ Columbite in this article refers to the following mineral species of columbite and euxenite groups: columbite-(Fe) – ferrocolumbite, columbite-(Mn) – manganocolumbite, tantalite- (Mn) – manganotantalite, and tantalite-(Fe) – ferrotanthalite (in according to the modern minerals classification adopted by the International Mineralogical Association).



Nicolini, Novikoff, and Kessi, described in the N.Watha-Ndoudy^{*} geological report, allowed to outline this array and build a schematic geological map. It was established that biotite and amphibole granodiorites, quartz diorites, and to a lesser extent charnockitoids are widespread within the massif. Biotite varieties of granodiorite are characterized by a high content of microcline and Na-rich plagioclase. The age of the rocks is estimated as Late Archean [10].

The late phases of granitoid magmatism are represented by evengrained granites with porphyry alkaline feldspar phenocrysts. These granites form small massifs and vein bodies, accompanied by aplites and pegmatites dykes [10]. Also, there are picrites, pyroxenites and dolerites^{*} bodies.

The pegmatites of the Chaillu massif are associated with the second phase of granitoid magmatism, represented by leucocratic granites. There are two pegmatites groups:

• P_1 – complex shaped stocks and veins located within granites and consisting of microcline, quartz, biotite, as well as chlorite and hematite^{*}.

• P_2 – stocks and veins located in major metamorphic rocks with a thickness of up to 10 m of mostly concordant bedding forms and consisting of plagioclase, microcline, quartz, biotite and muscovite with such minor minerals as tourmaline (schorl) and chlorite. Block and graphic structures are very common. The size of feldspar crystals reaches 30 cm across. In coarse and giant-grained structural varieties of these rocks, plagioclase predominates over the microcline. In graphic pegmatite, on the contrary, microcline is the main mineral. The P₂-pegmatites bodies are associated with quartz veins.

The rocks of the Chaillu massif in several regions have overlaid Proterozoic sedimentary rocks – conglomerates, arkose sandstones, mudstones, dolomites, clay and bituminous schist [10].

The granitoid fields of the Chaillu massif include metamorphic rocks arrays of the pre-granite migmatite gneiss complex [10], which belong to two greenstone belts – Zanaga and Mayoko. Both greenstone belts are located in the Republic of the Congo. Amphibolites, gneisses, ferruginous quartzites (itabirites and jespilites) are a part of the greenstone belts strata. These rocks are laterally limited from all sides by tectonic contacts with host granitoids. Like xenoliths, such bodies have an acute-angled bedding in the plain view, but unlike ordinary xenoliths, they are characterized by very significant size of up to 150 km². Obviously, such formations are parts of embedded metamorphic strata.

The rocks of the Mayoko greenstone belt are represented by amphibolites; amphibole, biotite and bi-mica gneisses and ferruginous quartzites. These rocks mass is crumpled into linear folds of northeastern strike and can be traced in the northeast direction in the form of a strip 20 km long and 5 km wide. Their age is estimated as Archean in the range of 3000-3100 billion years^{*}.

Mayoko rocks are believed to be para-metamorphic, formed under the conditions of amphibolite facies of regional metamorphism, although the primary sedimentary origin of the amphibolites is considered unproven^{*}.

Metamorphic rocks are split by the P_2 pegmatites vein bodies. P_1 -pegmatites were identified on the border of metamorphic rocks of Mayoko district with granites of the Chaillu massif in the Leyou river valley^{*}.

In practical terms, the Mayoko district is known for manifestations its iron deposits and placer gold [13]. Findings of diamond, corundum, ilmenite, rutile, and chromite are known in river sediments. The discovery of columbite group minerals in alluvial deposits, make this area promising for the exploration of tantalum and niobium primary ore deposits.

Until recently, many researchers of the Mayoko area believed that P_2 -pegmatites were the columbite parent rocks in this area. The materials of the meeting of the UN Economic and Social

^{*} Noël Watha-Ndoudy. Caratéristiques morphologiques et géochimiques des grains d'or: application a la prospection des placers de Mayoko (Congo).1993. 1993 INPL113N.

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Council held on February 5-10, 1968 in Addis Ababa^{*}, preserved information that in the early 60's of the last century, exploration work on this ore type was carried out in Mayoko region. 214 pits and 66 linear meters of prospecting trenches were passed. As a result of sampling and laboratory study of the selected samples, signs of columbite mineralization in P₂-pegmatites were not detected. Such minerals as beryl, monazite, zircon, rutile, and martite were revealed in P₂-pegmatites^{**}.

Research methods. We collected 10 stream sediment samples weighing from 62 to 500 g. The primary samples processing was carried out according to a standard scheme, including separation of grain fractions by size, further magnetic, electromagnetic and non-electromagnetic fractioning and, finally, separation of single mineral fractions by handpicking.

The EDS analysis of columbite group minerals was carried out using electron microscope equipped with microanalyzer JEOL-JSM-6510LA (IPGG RAS, Saint Petersburg, operator O.L.Galankina), CamScan MV2300 (VSEGEI, Saint Petersburg), Camebax SX50 (Lomonosov MSU, Moscow, operator D.A.Khanin). Samples for microprobe studies (polished section) were made by the authors on the basis of a protocryl bonding mass with diamond polishing.

We have studied the chemical composition of 32 grains of columbite fine fraction (91 measurements) and 15 grains of columbite coarse fraction (128 measurements). On average, it was planned to provide 2-3 measurements in one grain of a fine fraction, and 3-5 measurements in one grain of a coarse fraction. The composition of the largest columbite grains was determined in each growth zones (8 measurements).

Granulometric characteristic of columbite. The columbite grain size in all stream sediment samples varies from 0.2 to 15 mm across. We revealed two columbite groups (fractions) which differ in granulometric characteristics. The first fraction is represented by semi-rounded and rounded crystal fragments of 1.6-15 mm in size. The second fraction is characterized by euhedral, less rounded crystals of predominantly flattened, less often elongated habit, as well as crystal fragments of 0.2-1.5 mm in size. Such differences served as the basis for the columbite samples separation into two fractions differ in grain size: coarse (more than 1.6 mm) and fine (less than 1.6 mm).

Columbite chemical composition. It was found that the columbite crystals are heterogeneous. They are characterized by zoning and sector zoning, mineral micro inclusions and veins, appeared due to the secondary alterations (Fig.1). Zoning is appears due to a change in the Nb₂O₅ and Ta₂O₅ ratio and does not correlate with a change in FeO and MnO content. Mineral inclusions are represented by ilmenorutil, scheelite, and komarovite. Zircon and pyrochlore supergroup minerals were found in mineralized fissures and veins.

The content of major and trace elements in the columbite grains of both grain-sized fractions (wt.%, see table) varies in the following ranges:

• fine fraction: $Nb_2O_5 = 5,82-73,11$ %; $Ta_2O_5 = 3,28-77,79$ %; FeO = 2,28-18,08 %; MnO = 1,98-14,35 %; $Sc_2O_3 = 0,08-1,01$ %; $WO_3 = 0,06-2,04$ %; $TiO_2 = 0-5,3$ %; CaO = 0-3,35 %; $SiO_2 = 0-4,32$ %; MgO = 0,54 H 0,78 % (2 measurements);

• coarse fraction: $Nb_2O_5 = 30,69-72,79$ %; $Ta_2O_5 = 6,97-51,61$ %; FeO = 3,56-16,99 %; MnO = 3,18-13,79 %; $Sc_2O_3 = 0,36-0,84$ %; $WO_3 = 0,51-3,04$ %; $TiO_2 = 0,33-5,04$ %; CaO = 0-3,34 %; $SiO_2 = 0,45$ % (1 measurement); MgO = 0,02-0,83 %.

By the content of the major components, columbite of both fractions mainly refers to the ferrocolumbite (columbite-(Fe). Ferrotantalite, manganocolumbite, and manganotantalite are also found in the fine fraction; manganocolumbite is noted among the grains of the coarse fraction (Fig.2).

^{*} Apercu documentaire sur la situation du pays pour les minéraux suivant (Communication présentée par le Gouvernement de la République du Congo).

^{**} Noël Watha-Ndoudy. Caratéristiques morphologiques et géochimiques des grains d'or: application a la prospection des placers de Mayoko (Congo).1993. 1993 INPL113N.





Fig.1. BSE image of minerals

 $Mcl-mangano columbite,\ Mc-microlite,\ Fcl-ferrocolumbite,\ Ftn-ferrotantalite,\ Zr-zircon,\ Pc-pyrochlore,\ Sh-scheelite,\ Sh-scheelite,\$



Fig.2. The mineral composition of the columbite group minerals of the Mayoko region, presented on the Ta/(Nb + Ta) and Mn/(Mn + Fe) diagrams: a - fine fraction; b - coarse fraction

There are such trace elements as Sc, W, Ti, Ca, Mg, and Si. The grains of the coarse fraction are enriched in W (Fig.3, *a*) and depleted in Sc (Fig.3, *c*, *d*) in comparison with fine grains. Both fractions do not differ in the Ca and Ti content (Fig.3, *b*, *e*, *f*). However, the grains of the coarse fraction differ in the contents of Ti and Sc (Fig.3, *c*, *f*): some are depleted in Ti and Sc – these are ferrocolumbite and manganocolumbite with high Ta_2O_5 content (44-51.62 wt.%); others are enriched in Ti and Sc – ferrocolumbite and manganocolumbite with a low Ta_2O_5 content (up to 40%). The latter, in turn, are divided into two subgroups according to the Sc content: up to 0.3 and 0.3-0.6 wt.%.

The presence of Si is observed in fine columbite fraction only (Fig.3, g), and the Mg traces, on the contrary, are mainly found in coarse grains (Fig.3, h).



The average content of chemical elements in the Mayoko columbite group minerals (coarse and fine fraction), wt.%

Sample num- ber (grains)	1	2	3	8	7	6	9	10	11	12	13	14
Number of measurements	5	4	3	8	4	3	5	4	4	5	3	7
TiO ₂	0.56	0.57	2.94	3.47	0.49	3.13	3.28	4.62	3.91	3.36	3.91	2.95
Al_2O_3	-	-	-	-	-	-	-	-	—	-	-	-
Cr_2O_3	- 8 25	3 71	1/ 98	13 78	- 11.46	10.21	13.36	_ 14.07	13.76	10.48	13.87	14.26
MnO	8.23 9.41	13.59	4.11	5.29	5.83	8.74	5.42	5.11	5.02	8.37	4.86	3.83
MgO	-	-	-	-	-	-	-	-	-	-	-	-
CaO	-	0.13	-	-	-	-	-	-	0.20	0.07	-	_
Na ₂ O	-	-	-	-	-	-	-	_	-	-	-	-
Sc_2O_3	-	22.01	0.66	0.65	-	0.78	0.6	0.67	0.70	0.56	0.75	0.58
ND_2O_5 TapOr	37.07 45.03	52.81 48.87	00.41 17.31	16.09	37.02 45.47	01.5	00.14 17.13	01.38	02.15 12.77	05.14	01.80	20 57
WO ₃	-		-	0.29	-	1.72	0.51	0.46	1.48	1.9	1.86	0.82
Bi ₂ O ₃	-	-	-	-	-	-	-	-	_	-	-	_
Total amount	100.31	99.68	100.39	100.37	100.26	100.53	100.43	99.61	99.99	99.65	100.53	100.01
Sample num- ber (grains)	9-2a	01-3	9-1b	8-1a	8-2a	22.2	21.1	21.2	21.3	21.4	5*1	20.2
Number of measurements	2	3	10	10	10	7	3	2	2	9	1	1
TiO ₂	2.59	0.66	3.06	2.05	2.24	2.68	1.56	1.99	0.92	1.21	1.13	0.87
Al_2O_3	-	-	0.02	-	0.05	-	-	-	_	-	-	-
Cr_2O_3	-	11.12	- 11.86	15.05	- 7 37	12.84	-	-	-	13.68	-	-
MnO	4.08	4.08	7.51	4.29	10.67	3.99	3.74	4.36	4.34	4.62	4.13	4.88
MgO	-	-	0.13	0.63	0.07	-	_	-	_	_	-	-
CaO	-	-	0.08	0.04	1.73	1.84	-	-	-	0.62	-	0.14
Na ₂ O	-	-	-	-		-	-	-	—	-	-	-
Sc_2O_3 Nb ₂ O ₂	0.55	0.00	0.55	0.51 63.05	0.75	63 30	66.23	62 20	65 59	- 64.60	0.21	- 64 78
Ta_2O_5	23.48	28.66	17.37	14.60	12.38	14.09	11.37	15.96	15.16	14.82	58.46	14.98
WO ₃	-	-	-	-	-	0.83	1.47	1.17	0.40	0.41	-	-
Bi ₂ O ₃	-	-	0.04	-	0.05	-	-	-	-	-	-	-
l otal amount	100	100.01	99.29	100.21	98./1	100.16	100	100	101.26	99.96	100.82	100.49
0 1												
Sample num- ber (grains)	20.3	20.4	6*2	19.1	19.2	19.3	19.4	18.1	18.2	18.3	18.4	17
Sample num- ber (grains) Number of measurements	20.3	20.4 3	6*2 1	19.1	19.2	19.3 2	19.4 2	18.1	18.2	18.3 2	18.4	17 4
Sample num- ber (grains) Number of measurements TiO ₂	20.3 2 0.78	20.4 3 1.08	6*2 1 1.92	19.1 1 1.73	19.2 2 1.24	19.3 2 3.75	19.4 2 2.22	18.1 1 4.59	18.2 1 1.25	18.3 2 1.08	18.4 1 1.09	17 4 1.31
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃	20.3 2 0.78 -	20.4 3 1.08 -	6*2 1 1.92 -	19.1 1 1.73 -	19.2 2 1.24 -	19.3 2 3.75 -	19.4 2 2.22 -	18.1 1 4.59 0.66	18.2 1 1.25 -	18.3 2 1.08 -	18.4 1 1.09 -	17 4 1.31 -
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ E ₆ O	20.3 2 0.78 - -	20.4 3 1.08 - 15.44	6*2 1 1.92 - 5.27	19.1 1 1.73 - -	19.2 2 1.24 - - 15.56	19.3 2 3.75 - - 13.85	19.4 2 2.22 - 15.78	18.1 1 4.59 0.66 0.22 10.85	18.2 1 1.25 - 16.43	18.3 2 1.08 - -	18.4 1 1.09 - - 18.08	17 4 1.31 - -
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO	20.3 2 0.78 - 14.33 4.12	20.4 3 1.08 - 15.44 4.76	6*2 1 1.92 - 5.27 12.28	19.1 1 1.73 - 14.92 4.74	19.2 2 1.24 - 15.56 4.37	19.3 2 3.75 - 13.85 4.94	19.4 2 2.22 - 15.78 3.09	18.1 1 4.59 0.66 0.22 10.85 2	18.2 1 1.25 - 16.43 3.17	18.3 2 1.08 - - 14.38 4.63	18.4 1 1.09 - - 18.08 1.98	17 4 1.31 - 14.96 3.90
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO	20.3 2 0.78 - 14.33 4.12 -	20.4 3 1.08 - 15.44 4.76 -	6*2 1 1.92 - 5.27 12.28 -	19.1 1 1.73 - 14.92 4.74 -	19.2 2 1.24 - 15.56 4.37 -	19.3 2 3.75 - - 13.85 4.94 -	19.4 2 2.22 - 15.78 3.09 0.68	18.1 1 4.59 0.66 0.22 10.85 2	18.2 1 1.25 - 16.43 3.17 -	18.3 2 1.08 - - 14.38 4.63 -	18.4 1 1.09 - 18.08 1.98 -	17 4 1.31 - 14.96 3.90 -
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO	20.3 2 0.78 - 14.33 4.12 - -	20.4 3 1.08 - 15.44 4.76 - -	6*2 1 1.92 - 5.27 12.28 - -	19.1 1 1.73 - 14.92 4.74 - -	19.2 2 1.24 - 15.56 4.37 -	19.3 2 3.75 - - 13.85 4.94 - -	19.4 2 2.22 - 15.78 3.09 0.68	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61	18.2 1 1.25 - 16.43 3.17 -	18.3 2 1.08 - - 14.38 4.63 - -	18.4 1 1.09 - 18.08 1.98 - - -	17 4 1.31 - - 14.96 3.90 - -
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O So	20.3 2 0.78 - 14.33 4.12 - - -	20.4 3 1.08 - 15.44 4.76 - - - -	6*2 1 1.92 - 5.27 12.28 - - - - - - - - - - - - -	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51	19.2 2 1.24 - 15.56 4.37 - -	19.3 2 3.75 - 13.85 4.94 - - - - - - - - - - - - -	19.4 2 2.22 - 15.78 3.09 0.68 - -	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61	18.2 1 1.25 - 16.43 3.17 - - - -	18.3 2 1.08 - - 14.38 4.63 - - -	18.4 1 1.09 - 18.08 1.98 - - - -	17 4 1.31 - 14.96 3.90 - - -
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O.	20.3 2 0.78 - 14.33 4.12 - - - - - - - - - - - - -	20.4 3 1.08 - 15.44 4.76 - - - 70.01	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22	19.2 2 1.24 - 15.56 4.37 - - - 59.87	19.3 2 3.75 - - 13.85 4.94 - - 0.74 59.04	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - - 63.84	18.2 1 1.25 - 16.43 3.17 - - - - - - - - - - - - -	18.3 2 1.08 - - 14.38 4.63 - - - 63.11	18.4 1 1.09 - 18.08 1.98 - - - - - - - - - - - - -	17 4 1.31 - 14.96 3.90 - - - 56.96
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅	20.3 2 0.78 - 14.33 4.12 - - 44.68 35.43	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47	19.2 2 1.24 - - 15.56 4.37 - - 59.87 18.97	19.3 2 3.75 - 13.85 4.94 - - 0.74 59.04 16.96	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11	18.4 1 1.09 - - 18.08 1.98 - - - 71.15 8.61	17 4 1.31 - 14.96 3.90 - - 56.96 23.12
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅ WO ₃	20.3 2 0.78 - 14.33 4.12 - - 44.68 35.43 -	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 -	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87 -	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 -	19.2 2 1.24 - 15.56 4.37 - - 59.87 18.97 -	19.3 2 3.75 - 13.85 4.94 - - 0.74 59.04 16.96 0.73	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77 1.53	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71	18.4 1 1.09 - - 18.08 1.98 - - - 71.15 8.61 -	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 -
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅ WO ₃ Bi ₂ O ₃ Total amount	20.3 2 0.78 - 14.33 4.12 - 44.68 35.43 - 99.32	20.4 3 1.08 - - 15.44 4.76 - - 70.01 9.63 - 100.92	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87 - 100	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100	19.2 2 1.24 - 15.56 4.37 - - 59.87 18.97 - 100.01	19.3 2 3.75 - - 13.85 4.94 - - 0.74 59.04 16.96 0.73 - 100	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77 1.53 - 100	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 100.01	18.4 1 1.09 - - 18.08 1.98 - - - 71.15 8.61 - 100.91	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Nb ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅ WO ₃ Bi ₂ O ₃ Total amount Sample num- ber (grains)	20.3 2 0.78 - 14.33 4.12 - 44.68 35.43 - 99.32 16.1	20.4 3 1.08 - 15.44 4.76 - 70.01 9.63 - 100.92 16.2	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87 - 100 16.3	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99	18.2 1 1.25 - 16.43 3.17 - 70.85 6.77 1.53 - 100 26	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 100.01	18.4 1 1.09 - 18.08 1.98 - - 71.15 8.61 - 100.91 13*4	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅ WO ₃ Bi ₂ O ₃ Total amount Sample num- ber (grains) Number of measurements	20.3 2 0.78 - 14.33 4.12 - 44.68 35.43 - 99.32 16.1 2	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 - 100.92 16.2 2	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87 - 100 16.3 1	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4 1	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2 2	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3 3	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99 24 1	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77 1.53 - 100 26 2	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 100.01 29 3	18.4 1 1.09 - 18.08 1.98 - 71.15 8.61 - 100.91 13*4 1	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅ WO ₃ Bi ₂ O ₃ Total amount Sample num- ber (grains) Number of measurements TiO ₂	20.3 2 0.78 - 14.33 4.12 - 44.68 35.43 - 99.32 16.1 2 1.25	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 - 100.92 16.2 2 1.36	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87 - 100 16.3 1 0.98	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4 1 1.26	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1 1 1.62	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2 2 1.93	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3 3 3.06	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99 24 1 2.07	18.2 1 1.25 - 16.43 3.17 - 70.85 6.77 1.53 - 100 26 2 0.00	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 00.01 29 3 1.97	18.4 1 1.09 - 18.08 1.98 - 71.15 8.61 - 100.91 13*4 1 1.22	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1 1.83
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅ WO ₃ Bi ₂ O ₃ Total amount Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₄ Cr ₂ O ₅	20.3 2 0.78 - 14.33 4.12 - - 44.68 35.43 - 99.32 16.1 2 1.25 0.16	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 - 100.92 16.2 2 1.36 -	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87 - 100 16.3 1 0.98 -	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4 1 1.26 -	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1 1 1.62 -	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2 2 1.93 -	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3 3 3.06	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99 24 1 2.07 -	18.2 1 1.25 - 16.43 3.17 - 70.85 6.77 1.53 - 100 26 2 0.00 -	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 100.01 29 3 1.97 -	18.4 1 1.09 - 18.08 1.98 - - 71.15 8.61 - 100.91 13*4 1 1.22 -	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1 1.83 -
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅ WO ₃ Bi ₂ O ₃ Total amount Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO	20.3 2 0.78 - 14.33 4.12 - - 44.68 35.43 - 99.32 16.1 2 1.25 0.16 - 12.86	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 - 100.92 16.2 2 1.36 - 14.52	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87 - 100 16.3 1 0.98 - 15.71	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4 1 1.26 - - 11.94	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1 1 1.62 - - 16.66	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2 2 1.93 - - 11.20	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3 3 3.06 - 15.17	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99 24 1 2.07 - 14.20	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77 1.53 - 100 26 2 0.000 - - 3.12	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 100.01 29 3 1.97 - - 14.77	18.4 1 1.09 - 18.08 1.98 - - 71.15 8.61 - 100.91 13*4 1 1.22 - 2.78	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1 1.83 - 12.03
Sample number (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅ WO ₃ Bi ₂ O ₃ Total amount Sample number (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MnO MnO Mumber MnO Mago CaO Number Mumber Mago CaO Number Mago CaO Number Mago CaO Number Mago CaO Number Mum	20.3 2 0.78 - 14.33 4.12 - - 44.68 35.43 - 99.32 16.1 2 1.25 0.16 - 12.86 4.32	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 - 100.92 16.2 2 1.36 - 14.52 5.34	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87 - 100 16.3 1 0.98 - 15.71 4.22	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4 1 1.26 - - 11.94 7.84	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1 1 1.62 - 16.66 3.61	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2 2 1.93 - 11.20 8 70	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3 3 3.06 - 15.17 3.97	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99 24 1 2.07 - 14.20 5.20	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77 1.53 - 100 26 2 0.000 - - 3.12 12.50	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 100.01 29 3 1.97 - 14.77	18.4 1 1.09 - 18.08 1.98 - - 71.15 8.61 - 100.91 13*4 1 1.22 - 2.78 13.13	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1 1.83 - 12.03 5.80
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅ WO ₃ Bi ₂ O ₃ Total amount Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ FeO MnO MgO	20.3 2 0.78 - 14.33 4.12 - - 44.68 35.43 - 99.32 16.1 2 1.25 0.16 - 12.86 4.32 -	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 - 100.92 16.2 2 1.36 - 14.52 5.34 -	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87 - 100 16.3 1 0.98 - 15.71 4.22 -	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4 1 1.26 - - 11.94 7.84	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1 1 1.62 - - 16.66 3.61	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2 2 1.93 - 11.20 8.79	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3 3 3.06 - 15.17 3.97	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99 24 1 2.07 - 14.20 5.29	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77 1.53 - 100 26 2 0.000 - - 3.12 12.50	18.3 2 1.08 - 14.38 4.63 - - 63.11 15.11 1.71 - 100.01 29 3 1.97 - 14.77 4.47	18.4 1 1.09 - 18.08 1.98 - - 71.15 8.61 - 100.91 13*4 1 1.22 - 2.78 13.13	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1 1.83 - 12.03 5.80 -
Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ Cr ₂ O ₃ FeO MnO MgO CaO Na ₂ O Sc ₂ O ₃ Nb ₂ O ₅ Ta ₂ O ₅ WO ₃ Bi ₂ O ₃ Total amount Sample num- ber (grains) Number of measurements TiO ₂ Al ₂ O ₃ FeO MnO MgO CaO	20.3 2 0.78 - 14.33 4.12 - 44.68 35.43 - 99.32 16.1 2 1.25 0.16 - 12.86 4.32 - - -	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 - 100.92 16.2 2 1.36 - 14.52 5.34 - - -	6*2 1 1.92 - 5.27 12.28 - 0.55 52.22 27.87 - 100 16.3 1 0.98 - 15.71 4.22 - - - - - - - - - - - - -	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4 1 1.26 - - 11.94 7.84 - -	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1 1 1.62 - - 16.66 3.61 -	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2 2 1.93 - - 11.20 8.79 - -	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3 3 3.06 - 15.17 3.97 - -	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99 24 1 2.07 - 14.20 5.29 - -	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77 1.53 - 100 26 2 0.000 - - 3.12 12.50 -	18.3 2 1.08 - 14.38 4.63 - - 63.11 15.11 1.71 - 100.01 29 3 1.97 - 14.77 4.47 -	18.4 1 1.09 - 18.08 1.98 - - 71.15 8.61 - 100.91 13*4 1 1.22 - 2.78 13.13 0.32	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1 1.83 - 12.03 5.80 - 0.13
$\frac{\text{Sample number (grains)}}{\text{Number of measurements}}$ $\overline{TiO_2}$ Al_2O_3 Cr_2O_3 FeO MnO MgO CaO Na_2O Sc_2O_3 Nb_2O_5 Ta_2O_5 WO_3 Bi_2O_3 $Total amount$ $\overline{Sample number of measurements}}$ $\overline{TiO_2}$ Al_2O_3 Cr_2O_3 FeO MnO MgO CaO Na_2O	20.3 2 0.78 - 14.33 4.12 - 44.68 35.43 - 99.32 16.1 2 1.25 0.16 - 12.86 4.32 - - - - - - - - - - - - -	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 - 100.92 16.2 2 1.36 - 14.52 5.34 - - - - - - - - - - - - -	6*2 1 1.92 - 5.27 12.28 - - 0.55 52.22 27.87 - 100 16.3 1 0.98 - - 15.71 4.22 - - - - - - - - - - - - -	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4 1 1.26 - - 11.94 7.84 - - -	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1 1 1.62 - - 16.66 3.61 - -	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2 2 1.93 - - 11.20 8.79 -	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3 3 3.06 - 15.17 3.97 - - -	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99 24 1 2.07 - - 14.20 5.29 - - - - - -	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77 1.53 - 100 26 2 0.00 - - 3.12 12.50 - - -	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 100.01 29 3 1.97 - 14.77 4.47 - -	18.4 1 1.09 - 18.08 1.98 - - 71.15 8.61 - 100.91 13*4 1 1.22 - 2.78 13.13 - 0.32 -	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1 1.83 - 12.03 5.80 - 0.13 - 0.13 -
$\frac{\text{Sample number (grains)}}{\text{Number of measurements}}$ $\overline{TiO_2}$ Al_2O_3 Cr_2O_3 FeO MnO MgO CaO Na_2O Sc_2O_3 Nb_2O_5 WO_3 Bi_2O_3 $Total amount$ $\overline{Sample number (grains)}$ $\overline{TiO_2}$ Al_2O_3 TiO_2 Al_2O_3 Cr_2O_3 FeO MnO MgO CaO Na_2O Sc_2O_3 FeO MnO MgO CaO Na_2O Sc_2O_3	20.3 2 0.78 - 14.33 4.12 - 44.68 35.43 - 99.32 16.1 2 1.25 0.16 - 12.86 4.32 - - - - - - - - - - - - -	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 - 100.92 16.2 2 1.36 - 14.52 5.34 - - (2,5)	6*2 1 1.92 - 5.27 12.28 - - 0.55 52.22 27.87 - 100 16.3 1 0.98 - - 15.71 4.22 - - - - - - - - - - - - -	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4 1 1.26 - - 11.94 7.84 -	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1 1 1.62 - - 16.66 3.61 - 0.26	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2 2 1.93 - - 11.20 8.79 - 0.47	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3 3 3.06 - 15.17 3.97 - 0.71	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99 24 1 2.07 - - 14.20 5.29 - - 0.53	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77 1.53 - 100 26 2 0.00 - - 3.12 12.50 - <td< td=""><td>18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 63.11 15.11 1.71 - 100.01 29 3 1.97 - 14.77 4.47 - 0.50 (-50)</td><td>18.4 1 1.09 - 18.08 1.98 - - 71.15 8.61 - 100.91 13*4 1 1.22 - 2.78 13.13 0.32 - 0.21</td><td>17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1 1.83 - 12.03 5.80 - 0.13 - - 1.55</td></td<>	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 63.11 15.11 1.71 - 100.01 29 3 1.97 - 14.77 4.47 - 0.50 (-50)	18.4 1 1.09 - 18.08 1.98 - - 71.15 8.61 - 100.91 13*4 1 1.22 - 2.78 13.13 0.32 - 0.21	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1 1.83 - 12.03 5.80 - 0.13 - - 1.55
$\frac{\text{Sample number (grains)}}{\text{Number of measurements}}$ $\overline{TiO_2}$ Al_2O_3 Cr_2O_3 FeO MnO MgO CaO Na_2O Sc_2O_3 Nb_2O_5 WO_3 Bi_2O_3 $\overline{Total amount}$ $\overline{Sample number (grains)}$ $\overline{TiO_2}$ Al_2O_3 $\overline{TiO_2}$ Al_2O_3 Cr_2O_3 FeO MnO MgO CaO Na_2O Sc_2O_3 \overline{FeO} MnO MgO CaO Na_2O Sc_2O_3 Nb_2O_5 $\overline{Sc_2O_3}$ \overline{FeO} MnO MgO CaO Na_2O Sc_2O_3 Nb_2O_5 $\overline{Sc_2O_3}$ Nb_2O_5 $\overline{Sc_2O_5}$ Sc_2O_5	20.3 2 0.78 - 14.33 4.12 - 44.68 35.43 - 99.32 16.1 2 1.25 0.16 - 12.86 4.32 - - 34.98 44.95	20.4 3 1.08 - 15.44 4.76 - - 70.01 9.63 - 100.92 16.2 2 1.36 - 14.52 5.34 - 14.52 5.34 - - 60.67 18.22	6*2 1 1.92 - 5.27 12.28 - - 0.55 52.22 27.87 - 100 16.3 1 0.98 - - 15.71 4.22 - - - - - - - - - - - - -	19.1 1 1.73 - 14.92 4.74 - 0.41 0.51 65.22 12.47 - 100 16.4 1 1.26 - - 11.94 7.84 - - 57.60 22.26	19.2 2 1.24 - 15.56 4.37 - 59.87 18.97 - 100.01 23.1 1 1.62 - - 16.66 3.61 - 0.26 71.78 6.07	19.3 2 3.75 - 13.85 4.94 - 0.74 59.04 16.96 0.73 - 100 23.2 2 1.93 - - 0.47 63.62 12.22	19.4 2 2.22 - 15.78 3.09 0.68 - 0.04 66.64 10.76 0.81 - 99.99 23.3 3 3.06 - 15.17 3.97 - 0.71 57.85 10.22	18.1 1 4.59 0.66 0.22 10.85 2 - 3.61 - 63.84 9.24 - 94.99 24 1 2.07 - - 14.20 5.29 - 0.53 66.76 9.23	18.2 1 1.25 - 16.43 3.17 - - 70.85 6.77 1.53 - 000 26 2 0.00 - - 3.12 12.50 - - 7.10 76 70	18.3 2 1.08 - 14.38 4.63 - 63.11 15.11 1.71 - 63.11 15.11 1.71 - 100.01 29 3 1.97 - 14.77 4.47 - 0.50 64.72 11.94	18.4 1 1.09 - 18.08 1.98 - - 71.15 8.61 - 100.91 13*4 1 1.22 - 2.78 13.13 0.32 - 0.21 24.36 52 (2)	17 4 1.31 - 14.96 3.90 - - 56.96 23.12 - 100.25 30.2 1 1.83 - 12.03 5.80 - 0.13 - 40.77 20.54
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Fig.3. Diagrams of the components content ratio in the Mayoko columbite group minerals: a - W и Ta/(Ta + Nb); b - Ca и Ta/(Ta + Nb); c, d- Sc и Ta/(Ta + Nb); e, f- Ti и Ta/(Ta + Nb); g- Si и Ta /(Ta + Nb); h- Mg и Ta/(Ta + Nb); h-



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Fig.4. Diagrams of the dependence of the components content in the Mayoko columbite minerals group: a - (Ti + Sc) and (Fe + Mn) + Ta + Nb); b - 3Ti and (Fe + Mn) + 2(Ta + Nb); c - Sc and Ti; d - W and (Ta + Nb)

The form of the Ti presence in the columbite group minerals can be predicted according to the following theory-driven schemes for the Ti isomorphism in the minerals structures [26, 31]:

$$M^{2+} + M^{5+} \longrightarrow M^{3+} + Ti^{4+}$$
 (euxenite type);
 $A^{2+} + 2B^{5+} \longrightarrow 3Ti^{4+}$ (rutile type).

The euxenite mechanism, when Ti substitutes Nb, is not always acceptable, since the ratio of A and B cations in the columbite structure are 1:2, while in the euxenite structure it is 1:1. But the presence of Sc in columbite makes it possible for Ti to enter precisely the Nb position (Fig.4, c) [26]. The correlation between the listed elements content in the columbite grains (Fig.4, a, b) suggests two equivalent scheme of Ti isomorphic substitution: euxenite and rutile.

The tungsten content in the studied mineral weakly correlates with the Nb and Ta content, so it can be assumed that the presence of W is associated with the finest tungsten-bearing mineral inclusion, such as founded micro inclusions of scheelite (Fig.4, d).

Probable ore igneous rocks source. As is known, tantalum-niobium mineralization with the columbite group minerals being the main ore minerals can be associated with pegmatites and raremetal granites [11, 12, 15, 17, 18]. The main geological and industrial types of tantalum and tantalum-niobium deposits are also associated with these rocks. An indicator of the genetic correlation of the columbite group minerals with certain rocks is the content of the main chemical components in these minerals composition [2, 5, 6, 26, 29].

The studies showed that the Ta_2O_5 content in the ferrocolumbite and manganocolumbite of the Mayoko district mainly varies from 14 to 40 % with an average value of 19 for the coarse grain fraction, and 21% for the fine grain fraction. At only 14 measurement points, the Ta_2O_5 content is



below 10, but more than 5 wt. %. According to this indicator, Mayoko ferrocolumbite and manganocolumbite cannot be assigned to accessory minerals of the rocks of subalkaline rare-metal granites formation and their metasomatites (albitites and greisenes), since these rocks are mainly characterized by ferro- or manganocolumbite with a lower content of tantalum pentoxide (Ta_2O_5) – up to 3 (according to K.A.Vlasov et al. [6, 7]) or less than 10 wt.% (according to S.M.Beskin [2]). In these metasomatites, the content of tantalum pentoxide very rarely reaches 28 wt.% in single crystals and ferrocolumbite crystals are often in intergrowth with the Fe and Ti-bearing minerals [28].

The belonging of the studied minerals to carbonatites and alkaline pegmatites is also excluded due to their significant difference from columbite in the tantalum pentoxide content. In addition, the columbite in carbonatites and alkaline pegmatites are usually in intergrowth with pyrochlore or hatchettolite [1], which is not observed for columbite in the study area.

Two possible types of rocks can be considered as the probable ore igneous rocks source of Mayoko columbite, in which ferrocolumbite and manganocolumbite have similar Ta_2O_5 content features: lithium fluoride rare-metal granites and their metasomatites; granite pegmatites.

The first type can be excluded from consideration because the columbite group minerals in such rocks are typical accessories with crystal sizes varying from 0.01 to 0.2, rarely reaching 0.5 mm [22, 25, 27]. Columbite group minerals form micro inclusions both in rock-forming minerals – quartz, muscovite, albite, lepidolite [22, 27], and in other accessory minerals – topaz and beryl [27]. As you can see, Mayoko columbite is characterized by completely different granulometry and mineralogical features.

Granite pegmatites are more likely can be the parent rock of Mayoko columbite. In some of these rocks columbite crystals (as in our case) reach significant sizes and are characterized by a similar chemical composition [3, 4, 21, 26, 29].

Granite pegmatites of a muscovite and even more so ceramic formation can be excluded from consideration, since tantalum niobates are not typical for them. Very rarely (single finds of B.M.Shmakin and other researchers) they are found in pegmatites of the muscovite formation [8, 19]. Moreover, in such pegmatites, these minerals are mainly represented by niobium mineral species [1].

Therefore, it can be concluded that pegmatites of the rare metal formation can be the main source of columbite of alluvial deposits in the Mayoko district, since it is precisely these formations that are characterized by the columbite group minerals with the same high content of tantalum pentoxide as in Mayoko columbite [3, 4, 20, 21, 29]. It is rare-element pegmatites that contain all mineral species of the ferrocolumbite – manganocolumbite – manganotantalite – ferrotanthalite system [1, 9, 15, 21, 29]. This situation occured in the Mayuko district: both ferro- and manganocolumbite and ferro- and manganotantalite minerals have been discovered here.

In this regard, one should turn to the pegmatitis petrogenetic classification developed by P.Černý [24]. As is known, Černý divides rare-element pegmatites into petrogenetic families: LCT, NYF, and mixed (LCT-NYF). The LCT pegmatites, depending on the composition of the predominant mineralization, are divided into beryl, complex, albite-spodumene, and albite types. P. Černýs diagram (Fig.5) shows the general trend in the composition of the columbite group minerals in the evolutionary series of beryllium and complex rare-element pegmatites formation. Comparing this diagram with the obtained data, we can conclude that the fine columbite fraction in the Mayoko district corresponds to the beryl and spodumene subtypes of pegmatites (Fig.5, a), and the coarse grained – to the spodumene and complex spodumene subtypes of these rocks (Fig.5, b).

Less definite is assigning the studied minerals to the albite-spodumene and albite types of rareelement pegmatites. It is known that columbite is characteristic of albite-spodumene pegmatites, while middle members of the columbite group series are characteristic of albite pegmatites [14-16]. At the same time, the composition of columbite in such pegmatites is still poorly studied, and therefore the option of assigning Mayoko columbite to such rocks is not considered in this case.





Fig. 5. The mineral composition of the Mayoko columbite group on the Ta/(Nb + Ta) and Mn/(Mn + Fe) diagrams in comparison with the mineral composition of this group in the evolutionary series of beryllium and complex rare-element pegmatites formation (arrows) according to P.Černý [23]: a – fine fraction, b – coarse fraction 1 – beryl type pegmatites, 2 – spodumene pegmatites, 3 – complex spodumene subtype pegmatites, 4 – complex lepidolite subtype pegmatites, 5 – complex petalite subtype pegmatites

Pegmatites of the NYF family are characterized by the following geochemical features of the columbite group minerals: increased content of Y, Ti, Sc, U, Th, Zr, F, and Nb > Ta [24, 26, 30]. In this family, there are REE pegmatites, which P.Černý attributed to rare-metal pegmatites type. By the trace elements content, the Mayoko columbite group minerals differ from the ones of the LCT pegmatites with a higher content of Ti (up to 5 wt. %), Sc (up to 1 wt. %) and the complete absence of Sn, which brings them closer to the pegmatites of the NYF family [26].

Exploration and mineralogical studies previously conducted in the Mayoko district did not reveal columbite mineralization in the P_1 and P_2 granite pegmatites. Therefore, these rocks cannot be considered as potential industrial sources of the columbite group minerals, since they do not have any mineralogical features of rare metal nature. According to our observations, secondary and accessory minerals, such as clevelandite, light-colored and polychrome tourmaline (elbaite), spodumene, lepidolite, and others, typical of rare-metal pegmatites, are not found in these rocks. Apparently, they should be attributed to barren pegmatites, the formation of which was associated either with the regional metamorphism and ultrametamorphism, or with granitoid magmatism in the rock mass represented in the Mayoko region by gneisses, schists, and amphibolites. Emergence of the rare-element pegmatites bodies, which were the columbite source in placers of this region, probably remain buried under the loose deposits in the Mayoko rainforests.

Conclusions

1. Columbite group minerals of the Mayoko alluvial deposits are characterized by two grainsized fractions that differ in the degree of roundness and size: fine, slightly rounded (0.2-1.5 mm); coarse, semi-rounded (1.6-15.0 mm). Among the grains of a fine fraction, crystals of flattened and short-columned habit are found. This suggests that columbite occured in alluvial deposits during the destruction of rocks that differ in compositional and structural characteristics.

2. According to the content of the main components, the Mayoko columbite group minerals mainly belong to the ferruginous mineral species of this group. Ferro- and manganocolumbite, ferro- and manganotantalite are distinguished. Ferro- and manganocolocolbit are characterized by an increased Ta_2O_5 content (6-51,62 %). The main trace elements are Sc, W, and Ti.

3. The chemical composition of the columbite group minerals is complicated by zoning, mineral inclusions, and veins of secondary alterations. Zonality is expressed in the alternation of micro-



zones, differing in the Nb and Ta content ratio. Mineral inclusions are represented by ilmenorutil, pyrochlore, microlite, scheelite, and kamarovite. Pyrochlore subgroup minerals form the veins during the alteration processes in the grains of both fractions. Coarse grains contain fissures filled with zircon.

4. According to the Nb and Ta content, the studied columbite group minerals correspond to ones from the rare-element granite pegmatites of the beryll and spodumene complex types (LCT – in accordance with the classification of P.Černý [23]). Given the increased content of Sc and Ti, many grains of the studied minerals correspond to rare-element mixed-type pegmatites (LCT-NYF).

5. The ore bodies of rare-element pegmatites, which are potential sources of Mayoko columbite group minerals, are probably hidden under loose deposits located near the alluvial placers where these minerals are found. The presence of two granulometric fractions in columbit loose deposits suggests that the probable ore body of the largest grains is the rare-element pegmatites, while the sourse of the columbite fine fraction can be fine-grained rocks, such as rare-element pegmatoid granites typical of pegmatite fields of many rare metal deposits.

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