CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 70-7

MINERALOGICAL INVESTIGATION OF NIOBIUM AND RARE-EARTH CARBONATITE SAMPLES FROM ST. HONORÉ, QUEBEC

by

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MINERAL SCIENCES DIVISION

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JANUARY 27, 1970

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SUMMARY OF RESULTS

A mineralogical investigation has been made of samples of diamond drill core from a carbonatite deposit near St. Honoré, Quebec, which were submitted by one of the officers of SOQUEM. The samples consist largely of dolomite, which is heavily indurated by hematite. The niobium minerals are pyrochlore and columbite; the rare-earth minerals are monazite and bastnaesite. Grain-size estimates indicate that over 60% of the niobium minerals occur in grains larger than 65 mesh.

Other minerals found in the samples include apatite, calcite, biotite, chlorite, feldspar, quartz, hematite, magnetite, pyrite, sphalerite, chalcopyrite, pyrrhotite, barite, zircon and fluorite, in approximate order of decreasing abundance.

Examination of mill products submitted by SOQUEM indicate that the niobium minerals are almost completely liberated from the other minerals at a grind of 78% minus 200 mesh.

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INTRODUCTION

Following correspondence between Mr. Marcel Vallee, geologist with SOQUEM, and Mr. H. M. Woodrooffe, Chief of the Mineral Processing Division of the Mines Branch, Mr. Vallee sent some samples containing niobium and rare earths, which were reported to have originated from a carbonatite deposit near St. Honoré, Quebec. Mr. Vallee requested that the mineralogy and "granulometry" of the samples be investigated. In subsequent discussions between Mr. D. E. Pickett and Mr. H. M. Woodrooffe, both of the Mineral Processing Division, and Dr. E. H. Nickel of the Mineral Sciences Division, it was agreed that the investigation would be carried out by the staff of the Mineralogy Section of the Mineral Sciences Division. The samples were received early in October, and a letter giving the preliminary results of the mineralogical investigation was sent to Mr. Vallee on November 6, 1969. The results of a grain-size analysis were reported by telephone on December 30, 1969.

Early in December, a shipment of mill products was received, with a request that the associations of the niobium minerals be determined.

SAMPLES

All the samples received were in connection with SOQUEM's St. Honoré project No. 782. The first shipment consisted of ten diamond-drill core samples and six samples of core crushed to minus $\frac{1}{4}$ inch; details provided by Mr. Vallee are given in Table 1. The second shipment consisted of five mill products, and some of the details on this shipment, also provided by Mr. Vallee, are given in Table 2.

TABLE 1

Samples in First Shipment Received for Mineralogical Investigation

Sample				Hole		Size of Sample
Type	No.	% Nb_2O_5	% R.E.	No.	Footage	
Core	-	0.96	-	707	144	2-in.-long split core
"	-	0.69	-	707	236.5	"
"	-	0.74	-	707	323	"
"	-	1.07	-	707	421.5	"
"	-	1.07	-	707	428	"
"	-	0.62	-	707	570	"
"	-	0.62	-	707	575	"
"	-	0.55	-	707	790.5	"
"	-	0.55	-	707	792.5	"
"	-	0.55	-	707	795	"
-$\frac{1}{4}$ inch	604-1-1-A	0.69	-	707	276 to 473	400 g
"	604-1-1-B	0.60	-	716	559 to 765	"
"	604-1-1-C	0.63	-	717	526 to 815	"
"	604-1-1-D	0.65	-	718	475 to 715	"
"	604-1-1	0.65	-	composite of A-B-C-D		200 g
"	605-1	-	3.4	704	122.8 to 378	400 g

TABLE 2

Reported Analyses of Mill Products

Sample	No.	Wt. (%)	Nb_2O_5 (%)	Nb_2O_5 Distr. (%)
Pyrite Conc.	604-1-231	1.2	3.5	6.0
Magnetite Conc.	604-1-233	1.4	0.28	0.6
Final Nb_2O_5 Rejects	604-1-236	56.8	0.09	7.3
Cleaner Rejects	604-1-238	38.6	0.38	20.8
Nb_2O_5 Conc.	604-1-237	2.0	22.7	65.3
		100.0		100.0

METHOD OF INVESTIGATION

Polished and polished-thin sections were prepared from each of the drill core samples, and these were examined under ore and petrographic microscopes to identify the minerals and to determine their grain sizes and textural relationships. Grain-size analyses of the niobium minerals were made by systematically traversing the polished sections under the ore microscope, counting the number of grains of niobium minerals falling within different size categories (+35, -35+65, -65+200, -200+325 and -325 mesh), and converting the values so obtained to approximate volume percentage ratios by multiplying the number of grains in each category by the cubed radius of the median size in that category.

The -$\frac{1}{4}$-inch samples were screened, and the fractions between 35 and 200 mesh were separated into sink and float products by means of heavy liquids. Polished sections were then prepared from some of the sink products for study under the ore microscope and by electron probe microanalysis. One of the samples (composite 604-1-1) was also separated magnetically to provide purer concentrates for study. The minerals were identified microscopically and, where necessary, they were submitted to Mr. E. J. Murray of the Crystal Structure Group for powder X-ray diffraction analysis. Electron probe microanalyses were made by Dr. D. C. Harris and Mr. D. R. Owens to provide compositional data on some of the minerals.

The mill products were evaluated qualitatively by microscopic examination of polished sections.

RESULTS OF INVESTIGATION

Mineralogy of Diamond Drill Core

The samples of diamond drill core consist chiefly of a dolomitic carbonate, which contains irregular disseminations and aggregations of the other minerals. The samples have a variegated appearance, varying from white, pink, brick red to almost black. Descriptions of the individual samples are as follows (the numbers represent the reported footage along diamond drill hole No. 707):

144: Coarse reddish grey carbonate, with some patches of biotite; minor pyrite; no appreciable pyrochlore or columbite observed.

236.5: Brecciated carbonate, brick red to white, with some soft grey chloritic material; some apatite; minor fluorite; abundant grains of disseminated pyrochlore and columbite.

323: Brick red to yellowish carbonate with a few small vuggy veinlets of pyrite, and scattered grains of pyrochlore and apatite.

421.5: Brick red carbonate with coarse grains of intergrown hematite and magnetite; minor quartz and apatite. No pyrochlore or columbite observed.

428: Variegated sample, consisting of white, brick red and, yellow carbonate, with streaks of hematite; disseminated apatite, pyrochlore and columbite.

570: A dark rock, consisting largely of chlorite, with white carbonate, highly altered feldspar, and mica; fairly abundant pyrite; disseminated pyrochlore and zircon; minor sphalerite.

575: Largely pink carbonate with irregular patches of bright orange apatite. Disseminated crystals of pyrochlore and/or columbite.

790.5: Variegated from brick red through pink to white. Mostly carbonate, with some irregular patches of grey quartz; some open weathered cavities. Disseminated apatite and pyrochlore crystals.

792.5: White carbonate, with disseminated grains of chlorite, mica apatite and pyrochlore; minor sulphides.

795: White carbonate with abundant apatite. Disseminated pyrochlore crystals; minor sulphides in vuggy patches.

Monazite and bastnaesite also appear to be present in most of the above samples, but since they are difficult to distinguish unequivocally from some of the other minerals, it was considered safer not to include them in the individual listings, above.

The crushed samples of the 604 series appear to have similar compositions to the solid drill cores from Hole No. 707. Estimates of the mineral proportions, based on heavy liquid separations, indicate that dolomite generally comprises about 80% of the samples, and apatite, about 10%. Sample 1-1-C appears to have somewhat less dolomite and more apatite. All the samples in this series contain pyrochlore and columbite, from grey to black in colour. In addition, some barite was found in all the heavy concentrates; it may also be present in the 707 series, but may have been overlooked because of its similarity to the other minerals.

The crushed sample No. 605-1 has a different appearance from the other samples, having a pronounced grey colour in contrast to the reddish hues of the samples in the 604 series. The dolomite appears to have a higher specific gravity than normal, which may be due, at least in part, to intergrowths with the rare-earth minerals. Both monazite and bastnaesite were found in the heavy concentrate of sample 605-1, and these minerals may also be responsible for the anomalously high specific gravity of the dolomite.

Detailed Mineralogical Descriptions

Carbonate Minerals: The principal carbonate mineral, and the predominant mineral in all the samples, is dolomite ($CaMg(CO_3)_2$). Most of it is heavily indurated by tiny particles of hematite, which give the dolomite a brick red appearance. In a few samples, the dolomite has remained white (707-792.5 and 707-795), or is coloured a light pink (707-575). A relatively minor amount of calcite was also found, and where it is associated with the stained dolomite, it can generally be distinguished by its fresh, white appearance. The rare-earth carbonate, bastnaesite, is described in the section "Rare-earth Minerals".

Niobium (Columbium) Minerals: Two niobium minerals were found: pyrochlore $(Ca, Na)_2(Nb, Ta)_2O_6(O, OH, F)$ and columbite $(Fe, Mn)(Nb, Ta)_2O_6$. The pyrochlore has two principal modes of occurrence. In those samples that are relatively low in iron, characterized by the presence of white, or lightly coloured dolomite, the pyrochlore is grey in colour with a resinous lustre, and frequently occurs as well-developed octahedral crystals (Figure 1). Some of the pyrochlore grains contain substantial numbers of inclusions (Figure 1); others appear to be free of them. Electron probe microanalyses have shown that the pyrochlore has a low titanium content (an element that frequently replaces niobium) and that it does not contain an appreciable amount of rare earths.

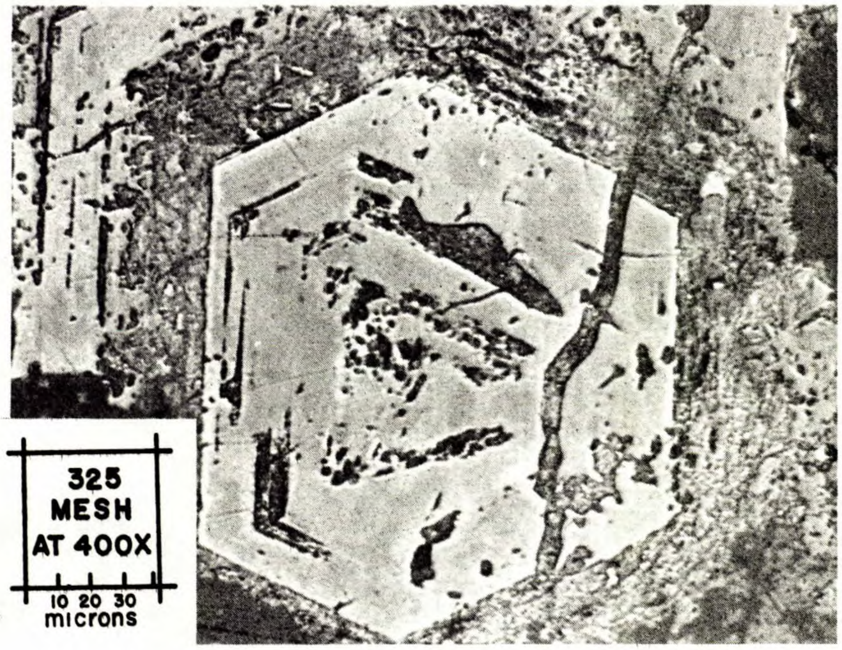

Figure 1. Photomicrograph of a polished section of a euhedral pyrochlore crystal from composite sample 604-1-1. The pyrochlore crystal contains oriented inclusions of gangue, and is cut by a veinlet of gangue material.

In the samples in which the dolomite is indurated by hematite, however, the pyrochlore is intimately intergrown with the columbite -- apparently the result of the partial decomposition of the pyrochlore. In some cases these "polyphase" grains consist largely of pyrochlore, with columbite surrounding the pyrochlore and filling fractures in it (Figure 2); in others, the grains consist largely of columbite with only small remnants of pyrochlore; many variations between these extremes are observed. The "polyphase" grains are black in colour, with a dark brown streak characteristic of columbite. Some of them occur as octahedral crystals, but more

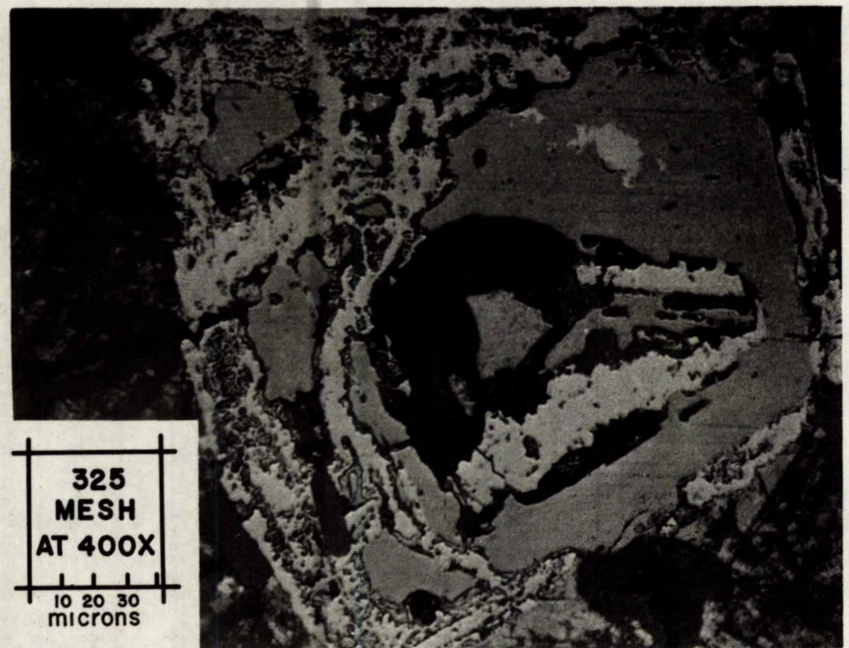

Figure 2. Polished section of a pyrochlore grain (light grey), bordered and veined by columbite (white). Gangue minerals are black.

often the grains are irregular in outline. Electron probe microanalyses of the columbite have shown that it is very iron rich -- a variety sometimes referred to as ferrocolumbite.

In one sample (236.5), the columbite is highly fragmented, and occurs embedded in a dark matrix. The nature of this matrix material could not be established because it appears to be amorphous to X-ray diffraction, and electron probe microanalysis indicates that no heavy elements are present. The most logical explanation for these observations is that the material consists of carbonaceous material, and it will be referred to as such here. The carbonaceous material is directly associated with the columbite, and varies from relatively minor amounts enclosing the the columbite to major amounts in which the columbite occurs as scattered grains (Figure 3).

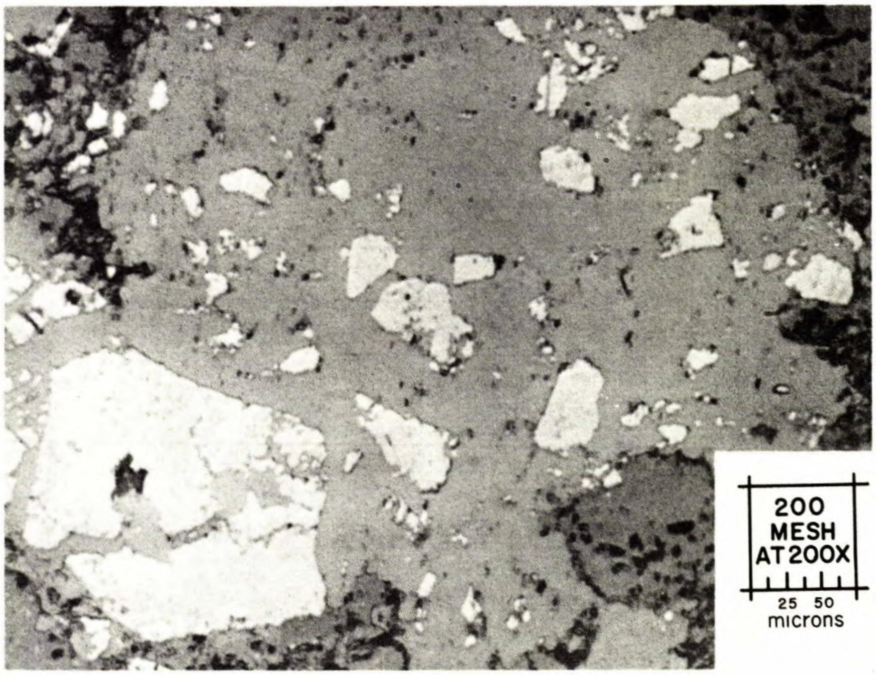

Figure 3. Polished section of sample 707-236.5, showing part of a large grain of carbonaceous material (light grey) enclosing irregular fragments of columbite (white). A few gangue minerals (medium grey) can be seen around the edges of the carbonaceous grain.

The pyrochlore and columbite occur over a wide range of grain sizes, but the great majority is coarser than 65 mesh, as illustrated by the results of a grain count made on seven polished sections (Table 3). In this analysis, no attempt was made to distinguish between pyrochlore and columbite. Also, because of some uncertainties in mineral identification, the results should be interpreted as estimates, rather than strictly quantitatively.

No estimates were made for samples 144, 323 and 421.5, since insufficient pyrochlore was observed to permit grain counts to be made.

TABLE 3

Estimated Grain-Size Distribution of Niobium Minerals in Samples from DDH. 707

Footage	+35 Mesh	-35+65 Mesh	-65+200 Mesh	-200+325 Mesh	-325 Mesh
236.5	-	62.3	29.2	7.0	1.5
428	45.0	30.7	21.3	2.8	0.2
570	55.7	33.2	9.5	1.5	0.1
575	87.4	11.2	1.4	-	-
790.5	62.5	31.9	4.5	1.0	0.1
792.5	-	80.0	17.6	1.6	-
795	42.4	51.4	5.6	0.6	-

(Values in table given as volume percentages).

Rare-Earth Minerals: Two rare-earth minerals were identified: monazite ($CePO_4$) and bastnaesite ($CeFCO_3$). These two minerals appear to be particularly abundant in sample 605-1, from which large discrete grains of these minerals could be concentrated by heavy liquids. Rare-earth minerals were also detected in most of the other samples by electron probe microanalysis, where the rare-earth mineral, or minerals, occurs in a finely dispersed form. In general, the dispersed grains were too small to be positively identified, but one cluster of brownish prismatic grains in sample 323 (Figure 4) was sufficiently large for X-ray diffraction analysis, which showed that the rare-earth mineral was bastnaesite.

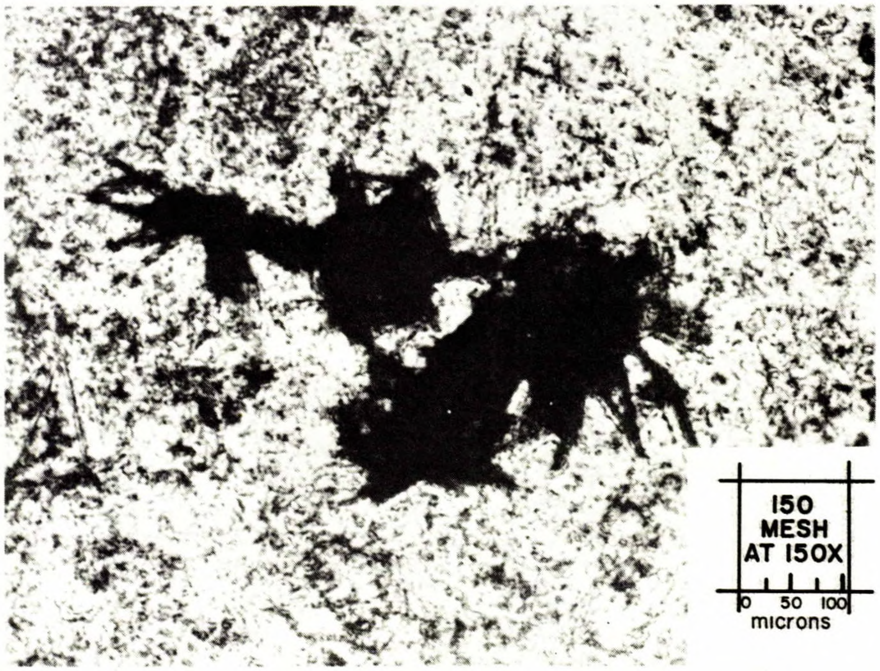

Figure 4. Photomicrograph of a thin section of sample 707-323 showing a cluster of bastnaesite grains (black) in dolomite (mottled white).

The larger grains of monazite and bastnaesite are dark brown to black, and are therefore very difficult to distinguish from the other dark minerals in the ore. In the polished section, the bastnaesite has the same general appearance as dolomite, whereas the monazite grains are quite similar to pyrochlore (Figure 5). In thin section they are largely, or completely, opaque.

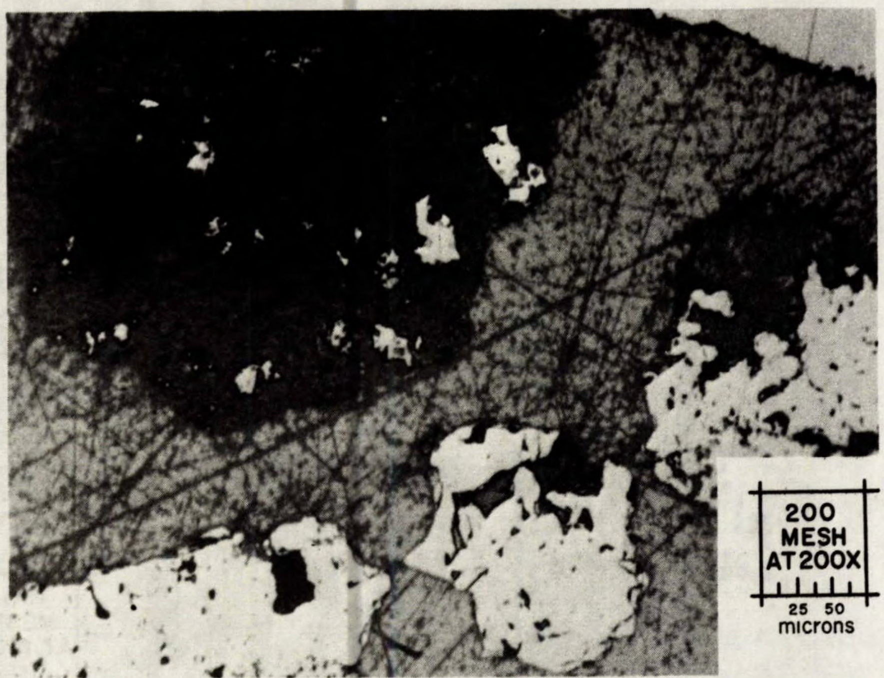

Figure 5. Polished section of sample 605-1, showing a large grain of bastnaesite (dark grey), and several grains of monazite (white). The intervening light grey material is the plastic mounting medium.

Sulphide Minerals: Sulphide minerals are sparsely disseminated in most of the samples examined. The principal sulphide appears to be pyrite, but minor amounts of sphalerite, chalcopyrite and pyrrhotite were also noted.

Iron Oxides: Hematite is very prevalent in most of the samples; it occurs predominantly as finely dispersed grains in, and thin intergranular films around, some of the other minerals, notably dolomite. This finely dispersed hematite gives much of the rock a brick red colour, and unfortunately tends to mask many of the mineralogical features. The hematite also occurs in larger grains, up to several millimetres in diameter, and in these cases it is frequently associated with magnetite, which appears to have been partly replaced by the hematite.

Other Minerals: Silicates are not very abundant in the samples examined, and appear to be represented chiefly by chlorite and biotite, generally in the form of disseminated grains. Some of the chlorite occurs as relatively large grains; some, however, occurs as soft, polycrystalline masses. The biotite is of a dark pleochroic variety. Some coarse, highly altered pink feldspar was observed in one of the samples; it is rather similar to the dolomite in outward appearance. Small amounts of zircon were found in a few of the samples; it is clear and transparent, with a light pink hue. Quartz was observed in one of the sections, as stained, fine-grained aggregates.

Apatite is quite abundant in all the samples examined. It is generally found as disseminated crystals throughout the dolomite, but it also occurs as aggregates of crystals. It varies from colourless to brick red; the latter colour can be attributed to intergranular films of hematite. Minor amounts of barite were detected in the heavy concentrates obtained from the crushed samples. It is clear and colourless. Fluorite was detected in only one sample.

Mineralogical Evaluation of Mill Products

Pyrite Concentrate (604-1-231): This sample consists predominantly of pyrite, but also contains other minerals such as dolomite, hematite and pyrochlore. The pyrochlore is present chiefly as free grains, and there is no appreciable association with the pyrite. In general, the pyrite grains are also substantially liberated from the gangue minerals.

Magnetite Concentrate (604-1-233): This sample consists largely of hematite and magnetite, intimately intergrown. The intergrowths have the appearance of magnetite partially oxidized to hematite. A few grains of gangue are present, but gangue minerals are not significantly intergrown with the hematite or magnetite.

Final Nb_2O_5 Rejects (604-1-236): This product consists largely of dolomite, but with a substantial amount of apatite. Several large grains (approx. 80 microns in diameter) of pyrochlore were observed in the polished section made from this product. There does not appear to be an appreciable amount of fine-grained pyrochlore in the gangue.

Cleaner Rejects (604-1-238): The niobium content in this sample also seems to be largely accounted for by a few relatively coarse grains of free pyrochlore. The sample consists largely of dolomite, with some apatite.

Nb_2O_5 Concentrate (604-1-237): The principal constituents in this sample are pyrochlore and columbite, generally intergrown. Only a small proportion of the grains contain adhering or included gangue minerals. The principal gangue minerals in the sample are dolomite and biotite, but small amounts of the other gangue minerals are also in evidence. The great majority of the gangue minerals do not have adhering or included niobium minerals.

Some monazite and/or bastnaesite are undoubtedly present in the mill products, and evidence for the presence of monazite in both the pyrochlore concentrate and the final reject was found in X-ray diffraction patterns. Because of the difficulty of distinguishing monazite and bastnaesite from some of the other minerals, however, no conclusions can safely be drawn on the distribution of these minerals among the mill products. It is quite possible, in fact, that some of the grains ascribed to pyrochlore in this investigation may actually be monazite, and that some of the bastnaesite may have been mistaken for dolomite.

CONCLUSIONS

1. The niobium-bearing minerals in the ore are pyrochlore and columbite, and they are frequently intergrown with each other. They occur as disseminated grains, mostly larger than 65 mesh.
2. The rare-earth minerals are monazite and bastnaesite. Because of the difficulty of distinguishing them from other minerals in the ore, no general estimate of their grain sizes can be made.
3. Examination of the mill products shows that liberation of the niobium minerals is essentially complete at the grind performed (78% minus 200 mesh).

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