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REPORT 79-1

TANTALUM AND NIOBIUM ORE DRESSING INVESTIGATIONS AT CANMET

D. RAICEVIC AND H.L. NOBLITT

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TANTALUM AND NIOBIUM ORE DRESSING INVESTIGATIONS AT CANMET

by

D. Raicevic* and H.L. Noblitt**

ABSTRACT

Tantalum and niobium have become important metals with a variety of metallurgical uses. They are in short supply on a world basis and as demand is certain to increase, it is in Canada's interest to search for and develop additional resources. The purpose of the present report is to encourage this. It describes the properties, uses and resources, both domestic and global, and gives production and consumption statistics for both metals.

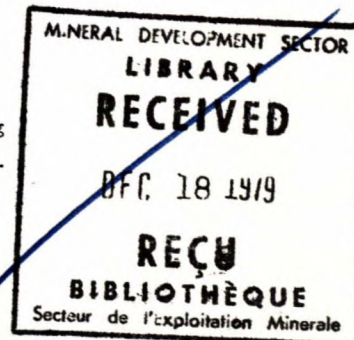
Tantalum is being mined in Canada only in low-grade pegmatite ores containing less than 0.2% Ta_2O_5 at Bernic Lake, Manitoba, and is recovered by gravity concentration followed by flotation of sulphide minerals from the tantalum concentrate when a larger than normal amount of these minerals occur in the concentrate. The tantalum concentrate produced has 50% Ta_2O_5 grade with 65 to 75% tantalum recovery depending on the degree of dissemination of the tantalum-bearing minerals (tantallite or wodginite, microlite and tapiolite) in the ore. The other known deposits have so far not been thoroughly explored.

As for niobium, present production is by flotation from carbonatite ores containing 0.76% Nb_2O_5 at St-Honoré, Quebec. A deposit grading 0.5% Nb_2O_5 was worked at Oka, Quebec between 1961 and 1976, where extensive reserves of a lower grade still exist on the original and neighbouring properties and production may resume depending on economic conditions. Another major deposit where the grade is 0.52% Nb_2O_5 awaits development near Moosonee, Ontario, but improved processes with recoveries greater than 70% as at present will likely be required before new plants are built.

Information on the mineralogy of the ores and their amenability to beneficiation is intended to facilitate the evaluation of potential resources and direct future ore dressing investigations to achieve better recovery at lower cost.

The report has two appendices consisting of brief reviews of ore dressing investigations related to the mineralogy and concentration of Canadian ores conducted at CANMET; it also covers research on foreign niobium ores considered to be of interest in treating domestic ores. The summaries consist of brief descriptions of the mineralogy and analyses of the ore samples tested, state the purpose of each investigation, the methods of concentration, results obtained, and conclusions. They also offer recommendations for future action.

*Research Scientist and **Research Scientist (on contract), Ore Processing Laboratory, Mineral Sciences Laboratories, CANMET, Energy, Mines and Resources Canada, Ottawa.



ETUDES SUR L'ENRICHISSEMENT DES MINERAIS DE
TANTALE ET DE NIOBIUM AU CANMET

par

D. Raicevic* et H.L. Noblitt**

RESUME

Le tantale et le niobium sont devenus des métaux importants ayant des usages métallurgiques variés. Les sources sont peu abondantes sur les marchés mondiaux et comme la demande augmentera certainement dans les prochaines années, le Canada ferait bon de rechercher et exploiter des sources additionnelles. Le but du présent rapport est d'encourager cette orientation. On décrit les propriétés, les usages et les sources soit domestiques ou mondiales et fournit les statistiques de production et de consommation des deux métaux.

On exploite, au Canada, seulement le tantale contenu dans les minerais de pegmatite à basse teneur situé à Bernic Lake (Manitoba). La concentration du Ta_2O_5 dans le minerai est de 0.2%, et il est récupéré par une concentration par la gravité suivie par la flottation des minéraux de sulfures du concentré de tantale au moment où ces minéraux sont en plus grande quantité que normale dans le concentré. Le concentré de tantale ainsi obtenu est concentré à 50% Ta_2O_5 avec une récupération de tantale de 65 à 75% selon le degré de dissémination des minéraux porteurs de tantale (tantalite ou wodginite, microlite et tapiolite) dans le minerai. Jusqu'à présent les autres gisements connus n'ont pas encore été explorés.

Dans le cas du niobium, la production actuelle se fait par flottation du minerai de carbonatite contenant 0.76% Nb_2O_5 à St-Honoré (Québec). Un gisement de 0.5% Nb_2O_5 a été exploité à Oka (Québec) entre 1961 et 1976 où des gisements abondants se trouvent encore sur la propriété d'origine et les environs et la production pourrait reprendre selon les conditions économiques. Un autre gisement important dont le teneur de Nb_2O_5 est de 0.52% attend d'être exploité à Moosonee (Ontario). Pour le moment, de nouveaux procédés capables de donner une récupération de plus de 70% devront être mis au point avant que de telles usines soient construites.

L'information sur la minéralogie des minerais et leur possibilité d'enrichissement a pour but de faciliter l'évaluation de sources virtuelles et de donner une orientation aux études sur le traitement du minerai futures afin d'atteindre de meilleures récupérations et à un prix moindre.

Le rapport comporte deux annexes composées de brèves descriptions d'études sur le traitement de ces minerais effectuées à CANMET; on y retrouve aussi les recherches effectuées à l'étranger sur les minerais de niobium susceptibles d'être employés dans le traitement du minerai canadien. Ces résumés décrivent brièvement la minéralogie et les analyses des échantillons de minerai et expose le but de chaque étude, les méthodes de concentration, les résultats obtenus et les conclusions. On donne aussi des recommandations d'usages futures.

*Chercheur scientifique et **chercheur scientifique (sous contrat), Laboratoire de traitement des minerais, Laboratoire des sciences minérales, CANMET, Énergie, Mines et Ressources Canada, Ottawa.

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INTRODUCTION

Tantalum and niobium, or columbium, are becoming important metals in industrialized countries because of their unique properties.

Although both metals were discovered early in the nineteenth century, it was not until the 1930's that their use in metallurgy became widespread. At present, Canada ranks second to Brazil in the production of niobium and tantalum concentrates in the non-Communist world. Most of the Canadian production is exported to the United States where it is processed. Tantalum and niobium are extracted in various forms, principally as metal powders and ferroalloys. Most products containing these metals that are required by Canada are imported from the United States, although Masterloy Products Ltd. of Ottawa produces some ferroniobium on a commercial scale. The objectives of this report are to present a brief description of resources and production statistics of these two metals both domestic and foreign and to summarize ore dressing research carried out at CANMET. In the case of niobium, research activities conducted elsewhere

are also included. The tantalum sections were compiled by D. Raicevic and those on niobium by H.L. Noblitt.

A summary of each investigation contains the following information:

- number and title of investigation,
- mineralogy and analysis of ore samples tested,
- purpose of investigation,
- methods of concentration, and
- results and conclusions.

Information on mineralogy of the deposits and their amenability to beneficiation will be helpful in evaluating resources and will also serve as a guide for future ore dressing investigations, indicating how new or improved beneficiation methods would be required to help bring promising deposits into production.

Most of the work on these two metals was originally reported on using Imperial units and this system has been retained in the present compilation rather than converting entirely to the International System of Units (SI).

Tantalum uses

Tantalum uses are related to its chemical and physical properties, the most important of which are:

1. ability to form an oxide film with excellent dielectric properties, transmitting alternating current in one direction only,
2. resistance to chemical attack at moderate temperature,
3. high melting point,
4. strength and ductility, and
5. ability to form stable carbides.

The principal uses are in electronic components, chemical processing equipment, cutting tools as tantalum carbide, and dies. The largest single usage of tantalum is in the electronic field, principally for capacitors, because it has the highest dielectric constant of any known metal oxide. Tantalum is also used in rectifier tubes, in signal and alarm devices as well as in vacuum and electronic tubes.

Tantalum finds use in corrosion-resistant applications because of its chemical inertness. The oxide film which forms on the surface of tantalum metal is very stable and seems not only to retard further oxidation of the metal, but is also soluble in very few corrosive media. Because the film remains relatively thin it does not impede heat transfer. These properties, in addition to its ability to maintain its strength and corrosion resistance at moderate temperatures, cause it to have many uses in corrosion-resistant applications, in particular as a lining of vessels used in the production of hydrochloric acid, hydrogen peroxide, bromine and several high purity chemicals, and in the recovery of sulphuric acid. Its ability to transmit heat is utilized in heat exchangers, condensers, coils and other such parts. In addition to these uses, these same properties account for its use in surgical implants, nuclear reactors and laboratory equipment. Niobium can substitute for tantalum; however, tantalum usually has a much longer service life.

Tantalum is also used in some superalloys employed in aerospace structures, jet engines and

gas turbines where strength at elevated temperatures and corrosion resistance is required.

About 70% of tantalum usage is as metal powder, principally in the manufacture of electronic components, although minor amounts are used as alloying agents. Tantalum carbide accounts for about 15 to 20% of the tantalum used and tantalum mill products for another 10%. Less than 5% is used in ferroalloys and chemicals.

Approximately 10 to 12% of world demand is met by recycled material. This is recovered from old capacitors, tantalum metal parts and turnings, and cutting tools and dies. It is expected that the percentage of tantalum demand supplied by recycled material will increase and eventually stabilize at around 20%.

Niobium uses

The major niobium uses are as an addition in various types of steels and to prepare niobium carbide.

Ferritic type stainless steel

What was initially a low-carbon 12% chromium steel has been improved by additions of molybdenum, vanadium and niobium to produce a stainless steel with improved resistance to deformation in the 500 to 600°C range. A 0.40% niobium content extends the time for 0.1% deformation from 400 to about 500 hours at 600°C.

Austenitic type stainless steels

When an 18-8% chromium-nickel steel is quenched at 1000 to 1100°C, carbon is largely retained in solid solution. If such a steel is subsequently heated to between 500 and 900°C, chromium carbide, containing as much as 90% chromium is precipitated at the grain boundaries. Those regions of the grains adjacent to the boundaries are largely depleted of chromium and their resistance to corrosion is greatly reduced leading to possible failure. Adding niobium to the melt before quenching ties up the carbon as a stable carbide which is resistant to corrosion. Titanium has been used in this application as well. At 650°C titanium-stabilized

steel ruptured after 10 000 hours; at the same temperature, niobium-stabilized material lasted 22 000 hours.

High strength low alloy steels (HSLA)

At present most of the niobium produced as ferroniobium is used at the rate of 0.035% to 0.1% in structural steels to control grain size and so increase strength by up to 50%. They are used in bridges, towers, columns in high-rise buildings, pipelines, offshore drilling platforms and similar applications.

HSLA steels are used in the transportation industry in ship structures, motor vehicles, railroad equipment, pipelines, and aircraft engines.

Niobium carbide

Niobium carbide, one of the hardest substances known, is used in machine-cutting tools and in teeth for power shovels.

Minor amounts of niobium are used in permanent magnet alloys, electric components, electrical resistance alloys, flame plating alloy coatings and metal-to-glass seal materials.

World production of tantalum and niobium concentrates

Approximately 96% of the non-Communist world production of columbium and tantalum combined comes from Brazil and Canada - 88% from Brazil and 8% from Canada. The remainder is from the columbo-tantalite concentrates of Nigeria, Zaire, Mozambique, Australia and Thailand. Approximate figures for world production of niobium concentrate from 1973 to 1975 are recorded in Table 1.

Niobium concentrate prices during 1976 increased chiefly due to increases in mining and ore treatment costs, ranging between \$3.50/kg (\$1.60/lb) at the beginning of the year and \$4.20/kg (\$1.90/lb) at the end based on the Nb_2O_5 or $Cb_2O_5^*$ contained in the concentrate.

Of the 1.22 million pounds of non-Communist world production during 1977, nearly one quarter of tantalum concentrates came from Tantalum Mining Corporation of Canada Ltd. (TANCO).

These prices refer to concentrates having a $Nb_2O_5:Ta_2O_5$ ratio of 10:1 (1).

* Cb_2O_5 when name columbium is used instead of niobium

Table 1 - World production of niobium concentrates, 1973-1975 (5)

Country	1973		1974		1975	
	Nb	Nb-Ta	Nb	Nb-Ta	Nb	Nb-Ta
(000 kg gross*)						
Brazil						
Pyrochlore	19 426	--	17 878	--	18 000	--
Columbite-tantalite	169	--	92	--	90	--
Canada						
Pyrochlore	2 885	--	3 848	--	3 372	--
Nigeria	1 240	--	1 193	--	990	--
Zaire	--	46	--	46	--	73
Malaysia	--	92	--	83	--	75
Thailand	8	--	31	--	7	--
Rwanda	33	--	37	--	45	--
Australia	--	200	--	128	--	120
Other countries	1	44	1	43	1	43
Total	23 762	382	23 080	301	22 505	311

*Nb or Nb-Ta contained

The world production of tantalum concentrates from non-Communist countries between 1975 and 1978 is recorded in Table 2.

During 1977, the Canadian price of tantalum concentrate of minimum 50% Ta_2O_5 grade was \$24.00/lb or \$52.90/kg of contained Ta_2O_5 in the concentrate.

Canadian niobium and tantalum requirements

Although Canada produces and exports niobium and tantalum concentrates, most Canadian requirements of these two metals are imported, chiefly from the United States. The Canadian consumption in various forms between 1967 and 1975 inclusive is recorded in Table 3.

Canadian production of tantalum concentrates

A small amount of tantalum concentrate was produced during the Second World War from deposits in the Yellowknife-Beaulieu River area, District of Mackenzie, N.W.T. However, it was not until 1969 that Canada went into large-scale production from the Bernic Lake pegmatite deposit in Manitoba of TANCO. The process consisted of gravity concentration by tabling and low and high intensity magnetic separation (2) Fig. 1, 2, on pages 19 and 21.

TANCO's production is recorded in Table 4.

Chemical analysis of a typical TANCO concentrate is given in Table 5.

"Known reserves of tantalum are small and there is a good possibility that a shortage could again occur in mid-to-late 1980's if new mines are not brought into production in the next five or six years" (1,3).

Canadian production of niobium concentrates

Niobium mineral production in Canada has been limited to no more than a dozen producers. Several of these operated small mills in the Great Slave Lake area producing a few tons of tantalite-columbite concentrates but there was no sustained production.

Canada's first producer of pyrochlore concentrates was St. Lawrence Columbian and Metals Corp., Oka, Quebec, which began operation

in 1961 and continued until early 1976. During that time it was the only producer in North America. When the plant was closed after a prolonged strike at St-Honoré, Quebec, a new producer, Niobec, began operations. Niobec has since provided some competition for the world's largest producer, C.B.M.M., at Araxa, Minas Geraes, Brazil.

Much credit is due to the management and directorate of St. Lawrence Columbian and Metals Corp. Ltd., for the courage to begin production of a new niobium-bearing concentrate to which ferroniobium manufacturers were unaccustomed. From the start of milling to Sept. 30, 1973, the plant treated 5 052 522 tons of ore and produced approximately 33 360 000 lb of contained Nb_2O_5 . Estimated total production until shutdown was about 39 000 000 lb of Nb_2O_5 from approximately 6 000 000 tons of ore.

Official production for Niobec is not available. However, using the metallurgical balance from Robert's paper, "The Niobec Mill" (4) production at 330 days a year should be about 6 000 000 lb of contained Nb_2O_5 . Since sales contracts are probably long-term, this would be consistent for 1977 on.

Reported production of niobium in pyrochlore concentrates in kg of contained Nb_2O_5 from 1961 to 1975 is shown in Table 6.

Specifications of Canadian pyrochlore concentrate are recorded in Table 7.

Characteristics of Canadian tantalum and niobium ores

Neither tantalum nor niobium are found in the free metallic state but usually occur together in a fairly large number of minerals. Because of similarity of atomic structures of these two metals, identical atomic radii and the same valence, mineralogical differentiation is often difficult.

The principal source of tantalum is the columbite-tantalite series of minerals - isomorphous iron, manganese, niobium and tantalum oxides. The general formula is $(Fe, Mn)(Nb, Ta)_2O_6$. The mineral is called tantalite

Table 2 - World production of tantalum concentrates, 1975-1977 (5)

	(000 pounds contained in Ta ₂ O ₅)			
	1975	1976	1977	1978*
Concentrates				
Australia	85	100	120	100-120
Brazil	195	190	120	100-120
Canada	400	260	325	325-330
Malaysia and Thailand	190	190	125	150
Mozambique	40	50	100	60-100
Spain-Portugal	30	30	60	60
Zaire	100	100	100	80-100
Other Africa	30	60	90	110
Other sources	<u>70</u>	<u>180</u>	<u>180</u>	<u>100</u>
Subtotals	1 140	1 160	1 220	1 085-1 190
Tin slags				
Thailand	420	480	450	500
Other	<u>695</u>	<u>900</u>	<u>1 000</u>	<u>985</u>
Subtotals	1 115	1 380	1 450	1 485
Totals, all sources	<u>2 255</u>	<u>2 540</u>	<u>2 670</u>	<u>2 570-2 675</u>

*Projected

Table 3 - Imports and consumption of niobium and tantalum in Canada 1967-1975 (1)

Year	Imports from United States - kg				Consumption
	Niobium and alloys, wrought	Tantalum and alloys, wrought	Tantalum and alloys, unwrought	Tantalum and alloys, powder	ferro-niobium and ferro- tantalum- niobium Nb and Nb-Ta content
1967	84	565	15 837	524	35 380
1968	170	894	1 557	830	130 635
1969	534	849	1 998	3 396	110 677
1970	--	387	848	1 125	132 449
1971	2 296	674	6 458	1 406	176 901
1972	741	526	1 440	656	263 084
1973	1 196	2 466	--	7 497	205 024
1974	1 992	2 330	--	5 662	231 332
1975	--	--	--	--	215 910

Table 4 - Production of Ta_2O_5 in concentrate by Tantalum Mining Corporation of Canada Limited, 1969-1976 (1)

Year	Ta_2O_5 contained in concentrate (kg)
1969	74 521
1970	192 367
1971	161 496
1972	147 911
1973	24 954
1974	121 777
1975	181 009
1976	127 813
1977	200 000*

*Projected

Table 5 - Chemical analysis of TANCO concentrate* (%)

Ta_2O_5	48 - 53
Nb_2O_5	2.5 - 3
SnO_2	8.5 - 9
MnO_2	7.5 - 8
TiO_2	1.5 - 2
P_2O_5	0.5 - 0.6
SiO_2	9 - 10
Li_2O	0.1 - 0.2

*Personal communication

if the Ta_2O_5 content exceeds the Nb_2O_5 content and columbite if the reverse is true. The only other major commercial source of tantalum is the microlite-pyrochlore series which has the general formula $Ca_2(Nb,Ta)_2O_6(OH,F)$. Similarly, this mineral is called microlite if the tantalum content exceeds that of niobium and pyrochlore if the reverse is true.

Columbite-tantalite, commonly associated with one or more of spodumene, lepidolite, beryl, cassiterite and pollucite appears in certain complex pegmatites. Wodginite, a tin-bearing

Table 6 - Production of Nb_2O_5 in concentrates by St. Lawrence Columbium and Metals Corp., Oka, Quebec, 1961-1975 (1)

Year	Nb_2O_5 (kg)	Year	Nb_2O_5 (kg)
1961	59 194	1969	1 548 805
1962	428 740	1979	2 129 293
1963	685 703	1971	1 058 089
1964	967 295	1972	1 757 138
1965	1 058 680	1973	1 441 030
1966	1 196 587	1974	1 920 101
1967	979 568	1975	1 684 659
1968	989 433		

Table 7 - Specifications of Canadian pyrochlore concentrate (1965) (6)

	Type SA-64%	Type SB-64%	Type SD-64%
Nb_2O_5	50.0 (min)	50.0 - 56.0	52 - 58.0
Ta_2O_5	0.10 - 1.0	0.10 - 1.0	0.10 - 1.0
SiO_2 (max)	4.0	4.0	1.0
SnO_2 (max)	0.02	0.02	0.02
TiO_2	4.0 - 8.0	4.0 - 7.0	4.0 - 6.0
P (max)	0.25	0.06	0.06
S (max)	0.4	0.1	0.1

variety of tantalite, forms a commercial deposit at Bernic Lake, Manitoba and occurs in other pegmatites in West Australia (7,8). Fergusonite, samarskite, eschynite and pyrochlore have been reported in mineral occurrences from pegmatites, granites, porphyries and aplites in several areas of Canada.

Important quantities of tantalite and columbite are contained in cassiterite (tin) deposits in Thailand, Zaire and Portugal.

The major niobium producer of the non-Communist world is Brazil from its operation in

the Araxà region where pyrochlore is the major niobium-bearing mineral.

"Eluvia" or soils produced in situ from deeply weathered bedrock, widespread in the tropical areas of the world, also contain columbite-tantalite or pyrochlore-bearing crystalline rocks (8).

Sands and gravels (placers) are also known to contain tantalum- and niobium-bearing minerals associated with many others such as ilmenite, rutile, zircon, cassiterite, monazite, etc.

Carbonatites, typically occurring in Precambrian continental shield, are also known to contain a fair amount of niobium and a wide variety of other minerals. There are more than 30 carbonatite deposits known in Ontario, as well as several in Quebec, Labrador, British Columbia and the Northwest Territories. The principal Canadian columbium mineral deposits in carbonatite complexes are:

- in Quebec: St. Lawrence Columbium and Metals Corporation mine, near Oka; the properties of Columbium Mining Products Ltd., Main Oka Mining Corporation and Columbium Limited, all in the Oka region; the Niobec Inc. mine at St-Honoré, near Chicoutimi.
- in Ontario: deposits at James Bay, at Manitou Island near North Bay and at Lackner Lake and Nemegosenda Lake, near Chapleau.

Granites and granite-pegmatites have for many years been known to contain niobium and tantalum but the evaluation and exploitation of these sources started at the beginning of the sixties.

The present important commercial minerals of tantalum and niobium in Canada are tantalite (wodginite) or stanniferous tantalite with a general chemical formula $(\text{TaCbSn}_{2x})_2(\text{MnFeSn}_x)_6$ and pyrochlore with approximate formula $\text{Cb}_2\text{O}_5\text{CaONaF}$. The composition of Brazilian pyrochlore is $\text{Cb}_2\text{O}_5\text{BaO}$.

Presently 267 deposits containing these metals are known in Canada and of these, 15 have published reserves of niobium and 3 have estimates for tantalum. About 180 of the deposits

are pegmatite, 64 are alkaline syenite-carbonatite complex, 3 are placers and 17 are an undefined type. Approximate locations of the most significant Canadian deposits with tantalum and niobium occurrences are shown on the map in Fig. 1.

The most important tantalum deposit in Canada, and actually the only one in operation on the North American continent, is located beneath Bernic Lake, Manitoba.

The Bernic Lake deposit is a complex, zoned pegmatite and contains a wide variety of minerals, some of which are quite unusual. The most abundant are quartz, feldspar, spodumene (LiAl_2O_6) and pollucite $[(\text{Cs}, \text{Na})(\text{Si}, \text{Al})_3\text{O}_6 \times \text{T}_2\text{O}]$ and in smaller but significant amounts are beryl ($\text{Be}_3\text{Al}_2\text{Si}_6\text{O}_{18}$) and amblygonite $[\text{LiAlF}(\text{PO}_4)]$.

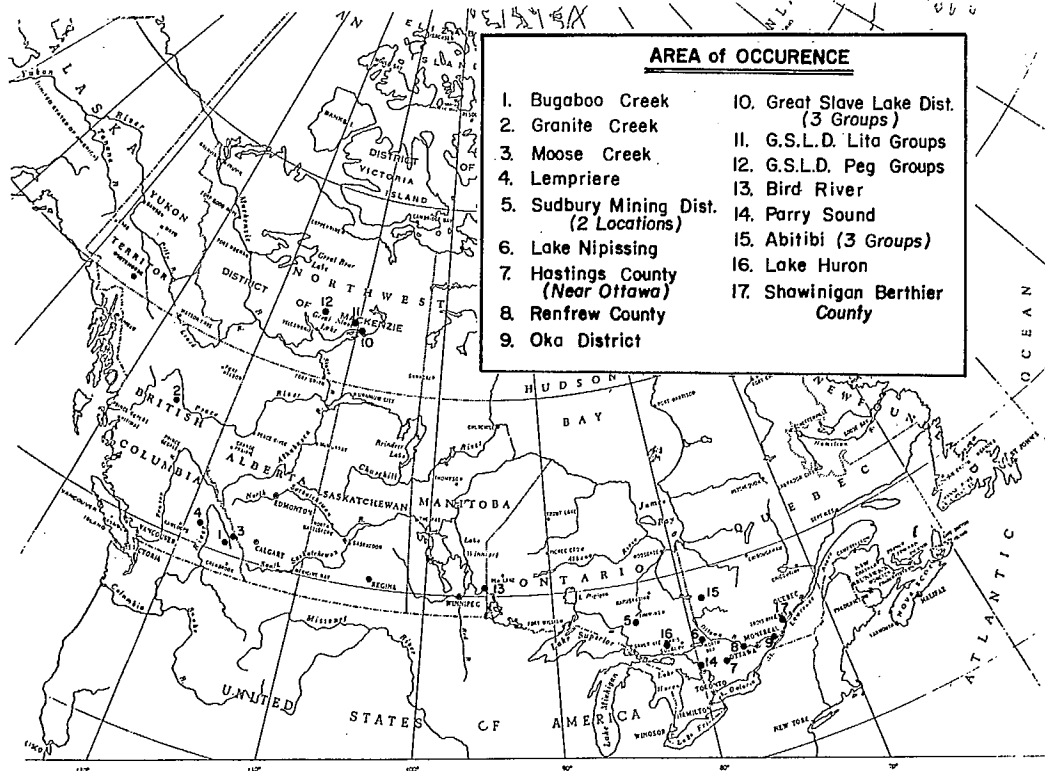
Tantalum in this deposit is contained in two types of tantalite, regular tantalite - $(\text{Ta}, \text{Nb})_2(\text{Mn}, \text{Fe})_6\text{O}_6$ - and stanniferous tantalite - $(\text{Ta}, \text{Nb}, \text{Sn}_{2x})_2(\text{MnFeSn}_x)_6$ - the latter called wodginite in new terminology. Two additional tantalum-bearing minerals, microlite - $(\text{Ta}, \text{Nb})_2(\text{Ca}, \text{Na})_2\text{O}_6(\text{OH}, \text{F}, \text{O})$ - and tapiolite - $(\text{Ta}, \text{Nb})_2(\text{Mn}, \text{Fe})_6\text{O}_6$ - are also present in this deposit. The stanniferous tantalite is by far the most common while only small quantities of other tantalum-bearing minerals are present. These are albite, lithiophilite, apatite, rhodochrosite, magnetite, ilmenite, scheelite, zircon, stannite, tourmaline, calcite, clay minerals, etc. The grains of the tantalite vary from highly irregular to fairly regular axe-shaped crystals and contain many silicate inclusions, chiefly muscovite.

The crystals of tantalites range in diameter from less than 1 mm to about 1 cm. Colour ranges from reddish brown to black.

The chemical composition of the stanniferous tantalite shows that it contains 70% tantalum pentoxide and 13.2% tin oxide, which is an unusually high amount of tin.

The other two tantalum-bearing minerals, tapiolite and microlite, are very similar to tantalite.

Tapiolite has the same colour and chemical formula as tantalite, but differs crystal-



Canadian deposits with tantalum and niobium occurrences (9). (Detailed map - "Niobium and Tantalum in Canada", MAP 1354A - was published in 1973 by the Geological Survey of Canada, Energy, Mines and Resources Canada)

lographically. It also occurs in irregular grains which, like tantalite, have many siliceous inclusions.

Microilite is generally irregular in shape, although octahedral crystals have been found. This mineral is creamy white in colour and contains 71.5% Ta_2O_5 (7,10).

For niobium the alkaline rocks have long held a peculiar fascination for both professional and amateur geologists, out of all proportion to their volumetric importance. Explanation of this unique suite of rare minerals, often crystallized in spectacularly large and beautiful forms and of their amazing diversity of chemical compositions, strains or exceeds the bounds of present petrogenetic theories. Economically, they are the

major source of niobium, diamonds and nepheline and a significant source of iron, copper, molybdenum, rare-earths, zirconium, uranium, mica (vermiculite), barite and apatite. They also represent a major potential source of aluminum, so far untapped except in the U.S.S.R.

Although the unusual appearance and mineralogy of alkaline rocks excepting nepheline gneisses assure their recognition and although many Canadian occurrences have been studied in some detail, basically little is known about them. For example, how is the distribution of alkaline rocks related to other rock-types or to major geologic structures? How are the various types of alkaline rocks genetically related to one another? How do the trace element content

and economic potential vary with mineralogy and structure? Because much detail is known about small areas and relatively little about more general problems, a compilation of data should help to clarify some of the larger problems, if only to define them more precisely. The present work aims to provide such a compilation. Hopefully, it will also provide part of the data needed to relate the origin of alkaline rocks to larger concepts of geologic theory such as plate tectonics and the origin of igneous rocks in general (11).

All primary niobium mineralization in Canada is found in intrusive rocks. Niobium is usually accompanied by tantalum to a greater or a lesser degree. The Ta/Nb ratio is much greater than in pegmatites in acid granites; and less than in alkaline rocks such as syenites. In ultra-alkaline calcite assemblages of carbonatites the ratio approaches 0.02 for pyrochlore and zero for niobian perovskite (dysanalite).

All niobium minerals of economic interest in Canada are resistant to decomposition by water and atmospheric weathering. Thus they are to be found in residual cappings of carbonatite deposits or in placers derived from them.

All tend to be brittle and may be slided in a stream bed through grinding by tougher stones. This brittleness is partly due to radioactive damage as most of them contain some uranium or thorium or both.

The habit of their crystal growth varies. Columbite and tantalite are orthorhombic and tend to grow into relatively long, thin crystals or into thin plates between the sheets of mica or sheared feldspars. These break readily when their enclosing quartz, mica or feldspar is ground. Pyrochlore is cubic and grows into octahedra and cubes. These forms break free more readily from softer silicates and very readily from calcite.

The coefficients of thermal expansion of pyrochlore and of niobian perovskite are different from that of the titaniferous magnetite with which they are usually found in carbonatites. Thus, a dried magnetic concentrate needs only to

be heated in flame, cooled and followed by dry magnetic separation to reduce the Nb_2O_5 content of the magnetic concentrate to well below 0.1%, if the material is minus 149 μm (100 mesh) in size.

It seems self-evident that the future world supply of niobium must come from carbonatite ores. This means pyrochlore. Carbonatites are visualized as masses of more or less impure limestone that have been rendered plastic by heat and pressure and squeezed through an orifice in the earth's crust in much the same fashion that volcanoes are formed. Their subsequent partial cooling and contraction permits the injection of basic magma which carries with it some or all of the following elements: sodium, niobium, titanium, phosphorus, tantalum, rare earths, iron, sulphur, thorium, fluorine and uranium. Since Nb_2O_5 melts at about 1520°C and the magma may be at a lower temperature than this, the former is probably present as a phosphate or as a phosphate complex. Phosphate, in the form of apatite, is found in all carbonatites some of which carry as much as 15% in pyrochlore-rich sections. It is probable that titanium is introduced in the same way.

Contact between the impure limestone or dolomite and the molten alkaline intrusive induces reactions between their constituents: phosphate and fluorine ions combine with calcium oxide from calcite to produce apatite. As the concentration of phosphate is reduced, tantalum, niobium and titanium, then without a carrier, combine with other available ions. Thus the first niobium-bearing minerals to be formed are grains of pyrochlore that are relatively high in tantalum, niobium, calcium and uranium, but low in titanium. Iron and excess titanium produce magnetite and ilmenite. These crystallize as titaniferous magnetite. Mobile ions continue to migrate into the carbonate and to precipitate apatite as well as grains of pyrochlore with less tantalum and niobium but with more titanium, sodium, iron and silica. Titanium seems to be more mobile and migrates farther. As the distance from the intrusive increases the number,

niobium content and size of pyrochlore grains decrease. Eventually, if mineralization penetrates far enough into the carbonate, the crystal system changes from octahedral to simple cubic and the grains formed are niobium-bearing perovskite.

If mineralization progresses further yet, it is probable that it would be confined to those crystals that became steadily smaller, fewer and lower in niobium content. As a result these crystals would disappear. This is the pattern of deposition in the Oka, Quebec, property of Columbian Mining Products Ltd., where dikes and sheets of intrusive are relatively narrow and are separated by up to 30 feet. On the other hand, if the dikes are thicker and closer together, the grade in Nb_2O_5 in the pyrochlore may remain high enough to prevent formation of perovskite by meeting an advancing face of mineralization from an adjacent dike. This is the case in the No. 2 pit of St. Lawrence Columbian and Metals. High grade ore made up of high grade grains of pyrochlore in curving sheets of altered carbonate lies between relatively thick sheets of intrusive waste, for the intrusive itself carries only very low niobium.

At the same time these changes are taking place, the impure calcite is being metamorphosed in places to diopside, monticellite, garnet, zeolites and melilite, with biotite, as well as accessory phlogopite, sphene, barite, fluorite, sericite, arfvedsonite and vesuvianite. The intrusive supplies aegirineaugite and nepheline.

In other parts of the Oka carbonatite and in portions of other carbonatites, notably in the part of the Newman Island portion of the Lake Nipissing occurrence mined for test work, the host rock for the pyrochlore ore is much more siliceous and pyroxenes are present. This makes an ore that is harder to drill and to grind but is more competent for support. It may also have a much higher uranium content.

Many of the pyrochlore grains have inclusions of calcite, apatite, magnetite and sulphide that affect the gravity concentration of these ores, as well as final Nb_2O_5 content and the concentration of penalizable impurities.

For any section in any deposit being mined, the grade of concentrate produced cannot exceed that occurring in the purest mineral, or minerals, found in the ore. Ore dressing can concentrate the desirable minerals by removing them from undesired minerals or by the reverse process. Improvement in grade above that in the minerals present can only be done by destroying the crystal form by chemical or other means and discarding the diluting material. Three such tests are included in this report.

According to Strukturbericht (12), koppelite, a type of pyrochlore, has a unit cell edge, (a) , of 10.37 Å. According to Nickel (13) the pyrochlore of Oka from Columbian Mining Products Limited has $a = 10.37$ Å to 10.38 Å. That from the Multi-Minerals Limited deposit at Nemegos, Ontario (carbonate type), was 10.37 Å. That from the No. 8 orebody from the same carbonatite was 10.35 Å. These cell edges are for ignited samples, so treated to remove the distortion caused by radioactive damage.

The R elements, (Ca, Ce, Na and K), appear to be in contact along a diagonal of a cell face. The distance between their centres is then, $a/2/4 = 10.37/2/4 = 3.6663$ Å. That between the centres of alternate large ion sites is then, $2 \times 3.663\text{Å} = 7.33$ Å. This mineral should be floatable with octadecyl succinidiamine which has a centre to centre, N to N distance of 7.272 Å; $7.334 \text{ Å} - 7.272 \text{ Å} = 0.062 \text{ Å}$. Also, columbite, with a c dimension of 5.730 Å should float well with octadecyl propane-diamine which has a centre to centre, N to N, distance of 5.684 Å; $5.730 \text{ Å} - 5.684 \text{ Å} = 0.046 \text{ Å}$. Both are within the 0.1 Å limit set for good attachment of a diamine to a mineral surface. It is noteworthy that the reagent used to float pyrochlore and columbite at St-Honoré, Quebec, is Duomac T, the acetate salt of tallow propanediamine. Also, from the report on the mineralogy of the St-Honoré deposit it appears that much, perhaps most, of the pyrochlore has attached columbite, which explains why Duomac T is effective in producing a flotation concentrate of suitable grade.

Amenability to concentration of tantalum and niobium ores

Little information has been published on the concentration of tantalum-bearing ores. Major work at CANMET on Canadian tantalum-bearing ores started in 1967 (2). On the basis of this work Tantalum Mining Corp. of Canada was formed and production of tantalum concentrate began in early 1969 (2,10). As indicated earlier, tantalum is often found associated with niobium which is concentrated by gravity or flotation, or a combination of both. In cases where the tantalum minerals occur alone such as in pegmatite deposits at Bernic Lake or in alluvial deposits, they are usually concentrated by gravity alone or in combination with low and high intensity magnetic separation (2,7).

To concentrate grains of pyrochlore or other niobium-containing minerals, liberation from associated gangue is a necessary prelude. All of the niobium minerals are brittle. In many carbonatite ores the grains of niobium-containing minerals vary in size from the smallest "seed" crystals to 0.5 cm. As long as the niobium mineral is completely enclosed in gangue it has the surface properties of the gangue. However, if it is freed, wholly or partially, its exposed surface can exert its own distinctive properties. Fine crystals of a mineral less than 10 μm in diameter are more difficult to concentrate by flotation. While they may attach to bubbles, the thinness of the coating of a fully-armored one-grain thick bubble leads to easy coalescence of bubbles and a coarse-grained, poorly mineralized froth. With fine bubbles the number of grains of mineral in a cubic metre of froth may be 50 times as great as in the same volume of froth made up of large bubbles. The first decision is how fine the ore is to be ground, how high a "seed" crystal loss can be accepted and what grinding cost can the grade of feed justify.

In the St. Lawrence Columbian and Metals No. 1 plant, pyrochlore was separated from the gangue by close sizing on screens and tabling (14). Losses were understandably high since all slimed pyrochlore was lost. In plant No. 2 de-

sliming ahead of pyrochlore flotation led to a high slimed pyrochlore loss (15). Similarly, in the third plant, operation desliming was practiced with consequent loss of pyrochlore in slimes (6). When the new plant was being designed for the Niobec mill, the decision was made to deslime before floating off carbonates. It is interesting to note that no metallurgical balance is given for niobium in any of the St. Lawrence articles and that the Niobec mill paper notes a 9.39% loss of niobium in the slimes, 14.38% loss in cleaner tailings and a 70.24% recovery in the final concentrate. This last figure is a good average for the operation of the St. Lawrence mill as well.

St. Lawrence Columbian and Metals had one problem that might not be shared to the same extent by other potential producers in Canada. In certain parts of the deposit hydrothermal alteration has partially or totally decomposed the micas. Those that are partially decomposed share with montmorillonite the property of absorbing diamines between their constituents sheets. On the property controlled by Columbian Mining Products Limited, one of the drill holes encountered a considerable thickness of vermiculite and this hydrated mica is found sporadically in or near the intrusive dikes where alteration has taken place. It has been shown that preliminary flotation in water with a short-chain monoamine produced a concentrate which contained about 1% of the weight of the feed as well as 1% of the niobium but that reduced such partly decomposed micas to a low level in pyrochlore flotation and reduced the cost of reagents. This was effective in the one case in which St. Lawrence ore was treated by the Columbian Mining Products process, as long as the usual high carbonate ore was being fed. When more refractory ore was treated, frothing went out of control and was only corrected by blocking intersheet openings in the mica with guar gum as is done in the New Mexico plants floating potassium chloride in the presence of montmorillonite.

As mentioned in the paper in the Proceedings of the Sixth International Mineral Process-

ing Congress the high recovery of pyrochlore in the first pilot run could be attributed to the use of only one fixed reagent mixture to float pyrochlore and one modifying agent, ammonium bicarbonate (16). In this way any middlings or cleaner tailings could be recirculated to the head of pyrochlore flotation without complications. It would appear that primary cleaned concentrate might advantageously be acid-treated to remove very fine calcite and so avoid settling problems in concentrate thickeners.

Another advantage of the diamine reagent approach lies in the ease with which the collector can be removed from concentrates with chlorine followed by washing with water. This has been demonstrated for many years with monoamines by the Lithium Corp. of America in the Carolinas and by the lithium producer in the Black Hills of South Dakota.

It was demonstrated in Pilot run No. 2 on Columbian Mining Products ore that a low air supply to the rougher cells of primary flotation gave a high recovery as well as a high grade concentrate that was low in apatite (16). It follows that, if the tailings from this flotation step, containing about 0.02% Nb_2O_5 , were re-floated with a higher air supply, the concentrate should be almost entirely apatite. Certainly this was the result of using high air in rougher primary flotation where the concentrate grade increased from 3.25% to 15% Nb_2O_5 on reducing the air supply. With this procedure the apatite concentrate could easily be de-collectorized with chlorine as above, followed by desliming if practicable and then re-floated with fatty acid. The concentrate should be acceptable for the making of phosphorus products and fertilizers. It may be noted that Niobec, with close to 10% apatite in its ore and a throughput of 1740 metric tpd (1950 TPD), is discarding approximately 156 metric tpd (175 TPD) of apatite into its tailing dump as well as 4 tons of pyrochlore and columbite.

Unfortunately for pyrochlore concentrate producers, the thermite reduction of pyrochlore ores is a vigorous process. Temperatures

approaching 2200°C cause strong currents of heated air and other gases to emerge from the reaction vessel with sufficient velocity to carry away some of the unreacted fine-grained concentrate. It would be expected that the finer the concentrate the higher will be the dust losses. Hence, a coarser concentrate can be more valuable than one that is fine unless the ferro-niobium producer is also the producer of concentrates. In Brazil the latter is the case. In Canada, both at St. Lawrence Columbian and Metals and now at Niobec, the fines produced in grinding are thrown away by desliming with the result that a relatively coarse, granular product is marketed in an effort to maintain sales.

SUMMARY OF RESEARCH AND DEVELOPMENT

Tantalum

The major tantalum-bearing mineral in Canadian deposits is tantalite. Canadian tantalite contains an unusually high amount of chemically combined tin and, as a result, is often called stanniferous tantalite. Microlite and tapiolite are also present in the deposits but in much smaller amounts than tantalite. Small amounts of tantalum are also associated with niobium-bearing minerals.

A gravity-magnetic separation process was successful in concentration of tantalum minerals due to their high specific gravity. The process, developed at CANMET is in operation at Tantalum Mining Corporation of Canada Ltd. (2).

Niobium

Columbite and pyrochlore are major niobium-bearing minerals. While there may be as much niobium in Canada in the form of columbite as there is in that of pyrochlore, it is so well dispersed in areas of the Canadian Shield and in other rocks of pre-palaeozoic age that there is no more than one example of commercial exploitation at this time.

Early work on the concentration of niobium-bearing minerals followed a familiar pattern. As most of these minerals have specific

gravities greater than that of quartz and feldspar, attempts were made to take advantage of this property. Tabling, jigging and heavy media separation were tried and abandoned because of excessive production of fines in grinding (14, 17, 18, 19, 20, 21).

Flotation with fatty acids and soaps was used, and still is, in the treatment of pyrochlore-bearing ores, not for the flotation of niobium minerals but for the removal of calcite. In some cases 60% of the feed is removed in this way compared with the 1 to 1.5% of the feed made up of the valuable minerals (6, 18, 19, 21, 22, 23).

Flotation with primary monoamines has not been employed successfully in Canada. It has been used in U.S.S.R. where the favourite reagent is called ANP-14 (24,25).

Hydroxamate has been used there too as a means of floating niobium-bearing minerals, notably pyrochlore (23,26).

Sodium alkyl sulphate in a solution having a pH of 2 has been recommended as a collector for pyrochlore, the high acidity preventing activation of other minerals by calcium ion (27,28).

The use of fluoride ion in the flotation of pyrochlore with diamines was practised at St. Lawrence and is being used by Niobec. Fluoride ion, HF, (6, 14, 15, 24), fluorosilicic acid (29), and magnesium fluosilicate (18) have been used with apparent success.

Sodium silicate depresses pyrochlore in the flotation of calcite and other minerals (21, 27, 31).

Sodium hydroxide is used to depress pyro-

chlore in "reverse" flotation (6), in flotation of calcite from pyrochlore ore (18), and in pH control with IM-50 reagent, hydroxamate (23).

Exotic collectors such as "catechol" and 8-quinolinol have been proposed as collectors in research work in Canada but neither has been used in production (18, 30).

Diamines as pyrochlore collectors were introduced in Tanganyika in 1955 (31). They were first used in Canada in 1956 in work on Columbian Mining Products' ore. Both Canadian producers have employed diamines for pyrochlore collectors and it is thought that a diamine is being used to concentrate the pyrochlore of Araxa in Brazil. Diamines give much greater selectivity than single-attachment reagents. Ammonium bicarbonate has been used in diamine flotation of pyrochlore to depress mica and silicates (16, 32).

When the supplies of more easily treated ores at St. Lawrence, Niobec and South Bluff Creek (Argor) are exhausted, it is probable that the lower grade deposits of the Oka area will be utilized. Usefulness of the all-alkaline concentration, developed by Columbian Mining Products with its higher recovery will then look more attractive to developers (16).

Extractive tests

The Oka pyrochlore concentrates are suitable feed for the production of pure niobium pentoxide and niobium metal only if they contain no niocalite. The perovskite concentrate produced by electrostatic separation from a pyrochlore which does not contain Ta_2O_5 , the pentachloride-to-metal route should yield reactor grade metal.

CONCLUSIONS

Tantalum

Tantalum is not found in the free metallic state in nature but, in most cases is associated with a large number of other minerals, mainly with niobium. All known Canadian deposits have a low tantalum content of less than 0.1% Ta_2O_5 but most have not been thoroughly explored. The only sizeable deposit is that of TANCO at Bernic Lake, Manitoba. The major tantalum-bearing mineral in this pegmatite deposit is tantalite often called stanniferous tantalite. By new terminology this tantalite is also called wodginite. Minor amounts of microlite and tapiolite are also present.

TANCO is also the only producer of tantalum concentrate on the North American continent. The concentrate is produced by gravity concentration, rejecting sulphides from the concentrate by flotation later when required. The concentrate is being sold to the U.S.A. a year or more ahead of production, indicating a high demand.

Niobium

Canada's production of niobium can soon be expected to come from the St-Honoré and Oka areas of Quebec. Other deposits may have to defer development until these two are more fully exploited. The deposit most likely to be developed next is that at South Bluff Creek south of

Moosonee, Ontario, where exploration has outlined 62.5×10^6 tons with a grade of 0.52% Nb_2O_5 .

In the Oka area, St. Lawrence Columbian and Metals mined between 5 and 6 million tons of ore with an average grade of about 0.5% Nb_2O_5 . The property is by no means exhausted but the surface plant was sold in 1976. The property of Columbian Mining Products with a reported 110 000 000 tons of 0.25% Nb_2O_5 ore amenable to concentration by flotation might be brought into production if economic conditions improve. There may be several hundred million tonnes of ore of various grades available in the whole Oka area.

At St-Honoré, Quebec, the Niobec deposit has an estimated 40 million tons grading 0.76% Nb_2O_5 . Of this 8 500 000 tons averaging 0.72% Nb_2O_5 are foreseen as feed to the present mill. Its estimated treatment rate is about 650 000 tons a year, so ore is available until 1989 at this site unless the milling rate is increased. Canada should be producing pyrochlore concentrates from Niobec and either St. Lawrence or Argor Corp. near Moosonee, until after the year 2000.

In any event new plants using concentrating processes recovering less than 70% of the niobium in the ore are unlikely to be built in the future.

RECOMMENDATIONS

Tantalum

Because known reserves of tantalum at Bernic Lake are limited and both demand and prices are rising, (1, 3, 5, 11), it is recommended that the following steps be taken:

1. establish true reserves and grades of Canadian deposits and determine their potentials;
2. establish the mineralogical association of the tantalum-bearing minerals with other minerals, in particular with niobium minerals as they are often closely associated;
3. conduct concentration investigations of representative samples from each promising deposit and establish their amenability to concentration with the objective of encouraging new producers.

Niobium

1. It is recommended that test work be done to concentrate pyrochlore and columbite from slimes, cleaner tailings and other products presently being wasted by plants producing niobium. Such concentrates might be used in the production of ferroniobium if some type of agglomeration could be found to reduce

dust losses.

2. As all niobium minerals being concentrated in Canada today contain uranium and thorium, the loss of these minerals to tailings means not only a loss of potential byproducts but also a potential environmental problem. The simplest approach would be to increase recovery of the niobium minerals which would consequently increase uranium and thorium recovery and thus reduce the environmental problems.
3. Because of the uranium and thorium content in the niobium concentrates and as these can be expected to report in the slag from ferroniobium production, the retreatment of slag to prevent contamination of the environment should be considered.
4. In the Union of South Africa new mineral dressing plants must provide methods for the recovery of metallic or industrial minerals that may be used as raw material for domestic production of fertilizer. Such a provision might be applied with advantage in Canada to help supply phosphate needs and reduce a trade deficit as most pyrochlore ores contain from 5 to 10% apatite.

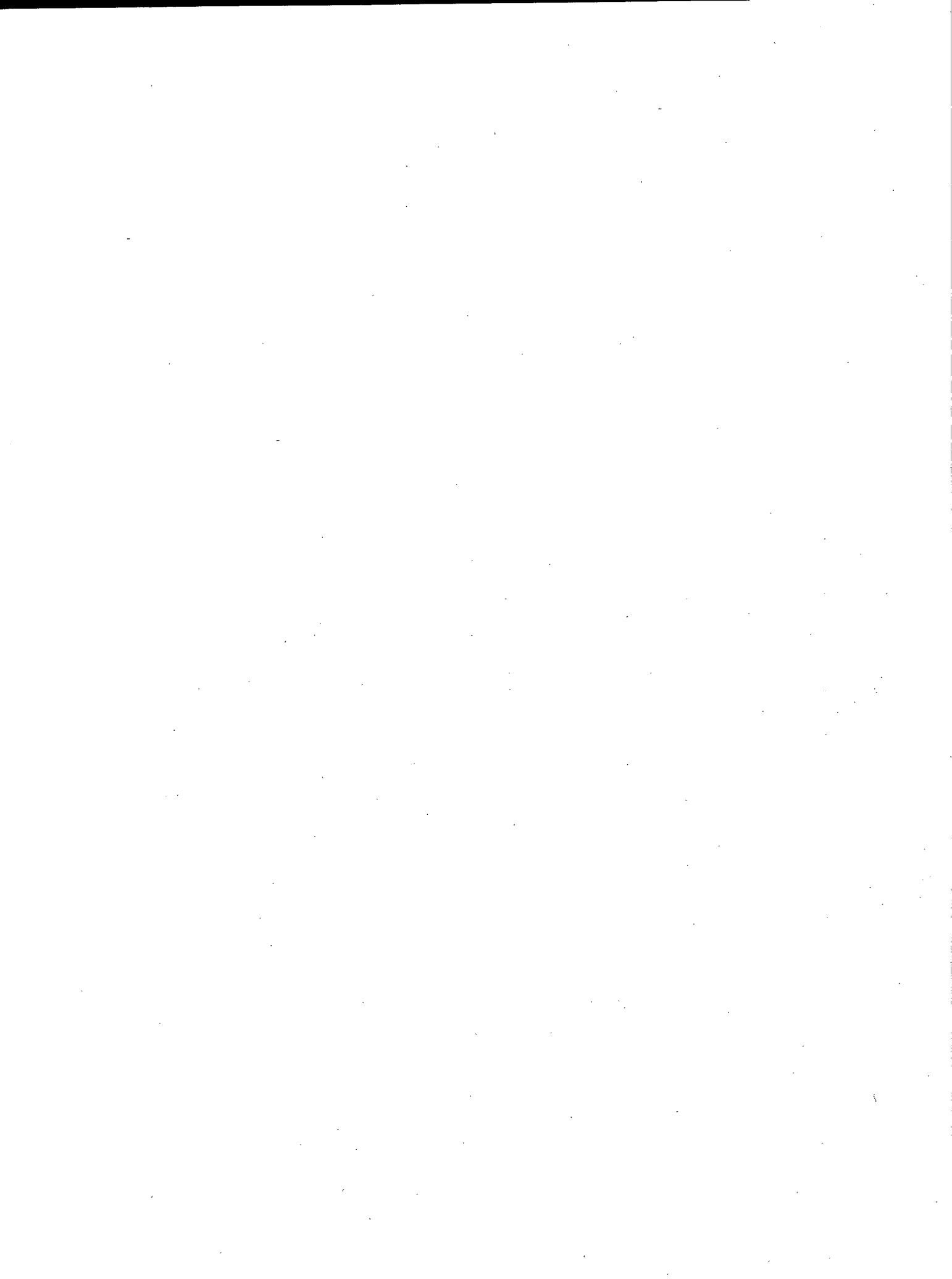
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APPENDIX A
RESEARCH AND DEVELOPMENT
ON
CANADIAN TANTALUM ORES FROM MANITOBA



Mineral Proc. Div test report MPT 61-25,
March 3, 1961

GRAVITY CONCENTRATION OF TANTALUM-BEARING
ORE FROM CHEMALLOW MINERALS LIMITED,
BERNIC LAKE, MANITOBA

by

W.S. Jenkins

Ore sample tested: About 95 lb of tantalum-bearing ore marked No. 281, representing 150 samples from one zone.

Mineralogy: A mineralogical study was conducted on a gravity concentrate produced by tabling and superpanning and not on the total ore sample (Internal Report MS-60-88, by E.H. Nickel, September 20, 1960).

The heavy concentrate was separated into its component minerals by means of heavy liquid and magnetic separations. The minerals were identified by X-ray diffraction analysis and by microscopic study.

The estimated composition of the sample, based on weights of the separated mineral fractions, is shown in the table below.

Mineral composition of the heavy concentrate

Mineral	Generalized chem. formula	Colour	Spec. grav.	Wt %
Tantalite	(Fe,Mn) (Ta,Nb) ₂ O ₆	black,brown	7.9	62
Magnetite	Fe ₃ O ₄	black	5.2	14
Microlite	(Na,Ca) ₂ Ta ₂ O ₇	white,tan	6.0	10
Cassiterite	SnO ₂	dark brown	7.0	5
Amblygonite	LiAlF(PO ₄)	white	3.1	3
Scheelite	CaWO ₄	white	6.1	2
Ilmenite	FeTiO ₃	black	4.7	1
Lithiophilite	Li(Mn,Fe)PO ₄	pink	3.5	1
Zircon	ZrSiO ₄	white	4.7	1
Others (stannite, quartz, apatite)				1

The spectrographic determination of the tin content in the concentrate was 13.6% SnO₂ or 10.7% Sn and niobium - Nb - content was about 3%.

A notable feature of the tantalite and microlite analyses was the high tin content of both. Evidence obtained from mineralogical work on other samples from this property indicated that the tin shown in the tantalite analysis is in chemical combination with the tantalite. This may also be true for the microlite. Another noteworthy feature is the low niobium content of both the tantalite and microlite, which indicates that these minerals are very close to the tantalum end-members of their respective mineral groups.

It was concluded from this mineralogical investigation that the heavy concentrate contained a relatively large number of mineral species. Most of the tantalum occurred in the form of tantalite, with some as microlite. The tin was distributed chiefly between tantalite, cassiterite and microlite.

Analysis: The sample assayed 0.57% Ta₂O₅.

Methods of concentration: Gravity concentration applying tabling and superpanning of the ore ground to minus 65 mesh.

Summary of results and conclusions: The summary of the results are recorded in the table below.

Superpanner concentration of table concentrate

Product	W, %		Analysis % Ta ₂ O ₅	Distribution %	
	In test	In orig feed		In test Ta ₂ O ₅	In orig feed Ta ₂ O ₅
Superpanner feed	100.00	7.68	2.964	100.0	39.2
Conc.	6.03	0.46	41.25	83.4	32.7
Tailing	93.97	7.22	0.525	16.6	6.5

The following conclusion was drawn. "The results indicated that 32.7% of the Ta₂O₅ in the ore could be recovered in a gravity concentrate, which assayed 41.25% Ta₂O₅ at a ratio of concentration of 217:1."

Mines Branch Investigation Report IR 67-52,
June 1969

CONCENTRATION OF TANTALUM FROM THE
BERNIC LAKE PEGMATITE DEPOSIT, MANITOBA

by

D. Raicevic

Ore samples tested: Three ore samples were tested - the first two of about 300 lb each, were taken from two underground locations and the third, weighing about 650 lb was described as being truly representative of the orebody.

Mineralogy: As described in "Mineralogy of Bernic Lake Pegmatite, Southeastern Manitoba", by E.H. Nickel (7).

Analysis: The first two samples contained 0.38-0.39% and 0.35-0.36% Ta_2O_5 respectively, and the third contained 0.2% Ta_2O_5 .

Purpose of investigation: To investigate the possibility of producing a commercial-grade of 45 to 50% Ta_2O_5 concentrate.

Methods of concentration applied: Gravity concentration and low and high intensity magnetic separation.

Summary of results and conclusions: A summary of the laboratory results is recorded in the table below.

Summary of three ore samples

Ore sample	Rghr grind, mesh	Rghr grind, mesh	Feed % Ta_2O_5	Final tantalum conc			Tailings and Fe conc			
				% Wt	% Ta_2O_5		% Wt	% Ta_2O_5		
					R/C	Assay		Distn	Assay	Distn
No. 1	20	65	0.394	0.68	147:1	44.0	75.9	99.32	0.096	24.1
No. 2	28	65	0.361	0.51	196:1	50.0	70.5	99.49	0.107	29.5
Representative	28	65	0.206	0.32	313:1	47.93	74.4	99.68	0.053	25.6

The above results showed that commercial-grade tantalum concentrates were produced from these ore samples.

Mines Branch Investigation Report IR 67-82,
November 1967

PILOT-PLANT INVESTIGATION OF TANTALUM
CONCENTRATION OF THE BERNIC LAKE ORE,
CHEMALLOY MINERALS LTD.,
LAC DU BONNET, MANITOBA

by

D. Raicevic

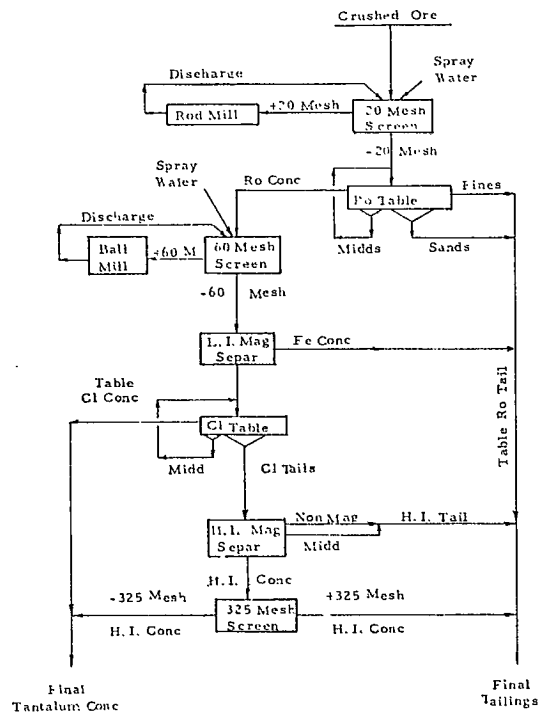
Ore sample tested: About 50 of the 75 tons of ore received was used for this pilot-plant testing.

Mineralogy: As described in "Mineralogy of Bernic Lake Pegmatite, Southeastern Manitoba", by E.H. Nickel (7).

Analysis: The average analysis of this sample was 0.31% Ta_2O_5 , 0.03% SnO_2 .

Purpose of investigation: The purpose of the pilot-plant investigation was to verify the laboratory process developed for concentration of this ore and to produce about 250 lb of the commercial-grade tantalum concentrate to be used for further treatment. Concentration of other minerals from this ore was not requested.

Methods of concentration applied: Sand- and slime-deck tabling and low and high intensity magnetic separation. The pilot-plant flowsheet of the process developed and used as a basis for the mill design of Tantalum Mining Corporation of Canada Limited, in Bernic Lake, Manitoba, is presented in the figure below.



Pilot plant flowsheet

Summary of results and conclusions: Tabling of this ore produced tantalum concentrates of 48.5 to 56.0% Ta₂O₅ with 78.8 to 77.5% recovery respectively.

Additional recovery of 1.3 to 2.9% Ta₂O₅ was obtained in scavenger concentrates with a grade of 42.4 to 17.5% Ta₂O₅ respectively by high intensity magnetic treatment and sizing.

These two operations, combined, produced tantalum concentrates of 47.4 to 55.7% Ta₂O₅ grade with overall recovery of 80.5 to 78.8% respectively.

The pilot-plant operation produced about 275 lb of concentrate for further commercial treatment. This concentrate had the following analysis:

Analysis of the 275-lb pilot-plant tantalum concentrate (10), %

Ta ₂ O ₅	50.2
Cb ₂ O ₅	2.88
SnO ₂	8.87
MnO ₂	7.64
TiO ₂	1.63
P ₂ O ₅	0.58
SiO ₂	9.30
Li ₂ O	0.18

Mineral Processing Division Internal Report 66-67
October 15, 1967

MEETING CONCERNING MILL DESIGN FOR
CHEMALLOY MINERALS LTD.

by

D. Raicevic

This report discusses major points and conclusions of the meeting regarding the mill design for the tantalum concentration of the ores from the Bernic Lake pegmatite deposit. It was decided to adopt the flowsheet shown previously.

Paper published in CIM Bulletin, December 1968

CONCENTRATION OF TANTALUM FROM THE
BERNIC LAKE PEGMATITE DEPOSIT, MANITOBA

by

D. Raicevic

This paper gives the pertinent mineralogy of the Bernic Lake deposit and highlights of the laboratory and pilot-plant investigations. Only an abstract described in reports IR 69-52 and IR 67-82 of the paper will be recorded here as the paper can be found in the CIM Bulletin of December 1968.

"Interest has recently been revived in the recovery of tantalum from the Bernic Lake pegmatite deposit. A gravity concentration process was developed to give satisfactory recovery and concentrate grade. The application of high-intensity magnetic separation was also studied. A pilot-plant investigation that confirmed the laboratory testing and provided a commercial-grade concentrate is described."

Mines Branch Investigation Report IR 69-90,
December 1969

STUDY OF METHODS FOR IMPROVING TANTALUM
RECOVERY AT TANTALUM MINING CORPORATION
OF CANADA LIMITED, BERNIC LAKE, MANITOBA

by

D. Raicevic

Ore samples tested: Three samples from TANCO mill were investigated:

Sample 1: fines from the cleaner tailings,
Sample 2: total cleaner tailings, and
Sample 3: plus 35-mesh fraction of tantalum rougher concentrate.

Mineralogy: Not determined.

Analysis: Sample 1. (minus 200 mesh fraction) assayed 8.94% Ta_2O_5 , minus 200 mesh from Sample 2 assayed 9.5% Ta_2O_5 ; Sample 3 assayed 4.5% Ta_2O_5 .

Purpose of investigation: (a) to recover the maximum of the finely-ground tantalum-bearing minerals being wasted, (b) upgrading the coarse portion of the rougher concentrate before secondary grinding. The concentrates from (a) and (b) should have tantalum grades close to 50% Ta_2O_5 .

Methods of concentration: Slime-deck tabling and high intensity magnetite separation of Sample 1 and 2, and sand-deck tabling of Sample 3.

Summary of results and conclusions: (1) Slime-deck tabling of the fines from cleaner-table tailings (Sample 1 and 2) being wasted, produced tantalum concentrates with grades of 56.0 to 61.7% Ta_2O_5 containing 4 to 5% of the Ta_2O_5 . (2) Tabling the coarse portion of the rougher concentrate (Sample 3) without the secondary grind, thus reducing formation of tantalite fines from this portion of the rougher concentrate, produced a coarse cleaner concentrate that contained 43.5% Ta_2O_5 . The calculate tantalum concentrate from (1) and (2) would have a tantalum grade of about 50% Ta_2O_5 .

Mines Branch Investigation Report IR 73-11,
February 1973

RECOVERY OF TANTALUM-BEARING MINERALS FROM
FINELY DISSEMINATED ORES IN A PEGMATITE
DEPOSIT AT BERNIC LAKE, MANITOBA

by

D. Raicevic

Ore samples tested: Three low-grade refractory ores were used.

Mineralogy: Extensive mineralogical investigations were conducted by D.C. Harris, on the various tantalum concentrates from the samples investigated (IR 72-39 and IR 72-54). A pertinent mineralogical summary from these reports is recorded here as these ores differed considerably from the normal tantalum ore of Bernic Lake pegmatite deposit.

Tantalite, wodginite and microlite were the principal tantalum-bearing minerals in the various concentrates. Due to their similar optical properties under reflected light, these minerals could not be distinguished from each other. Microlite was rarely detected.

Wodginite is a distinct mineral that can be considered as a high-tin tantalite previously called stanniferous tantalite.

Cassiterite was fairly abundant in the concentrates but, because it is of a colour similar to tantalite-wodginite, its relative proportion could not be determined. About half of the cassiterite grains contained up to 4.4% Ta. Tin also occurred in significant amounts in the wodginite-tantalite grains.

Several sulphide minerals were identified and, based on grain counting, constituted between 20 and 22% by volume of the concentrates. The principal sulphides were arsenopyrite and pyrite with minor to trace amounts of pyrrhotite, chalcopyrite, galena and native bismuth intergrown with an unidentified Ag-Pb-Bi sulphosalt, niccolite, native silver and stannite.

Magnetite and ilmenite were also found in several concentrates obtained from these ore samples.

Analysis: The average assays of the three ore samples investigated was as follows:

Date of sampling	% Ta_2O_5
November 18, 1971	0.14% Ta_2O_5
November 25, 1971	0.20% Ta_2O_5
June 1 and 2, 1972	0.15% Ta_2O_5

Purpose of investigation: In his letter of December 9, 1971, C.T. Williams, manager of Tantalum Mining Corporation of Canada Ltd., wrote:

"Although not apparent mineralogically, there has been a change in the ore as we mine downward and on the average the amount of very finely disseminated tantalite is increasing. Our recoveries are dropping and also it becomes increasingly more difficult to make our 45% to 50% Ta₂O₅ grade specification. The grade of the ore being processed is lower than that used in your work, but we have in the past had normal recovery on grades as low, or lower. We also have occasional problems with intermediate gravity minerals, such as tourmaline, ilmenite and occasional arsenopyrite which of course is nearly as dense as the wodgeite, but occurs at times locked with amphibolite gangue.

Our questions seem to be multiple. Firstly, will the so-called fine-grained ore we are presently encountering give the same response to the process you developed in 1967? If it doesn't, is it possible that changes might be made to the present flowsheet to maintain recovery and product grade at reasonable levels? If this is not possible, what other metallurgical process alternatives are there?"

Methods of concentration: Sand- and slime-deck tabling, low intensity magnetic separation and flotation of sulphide minerals.

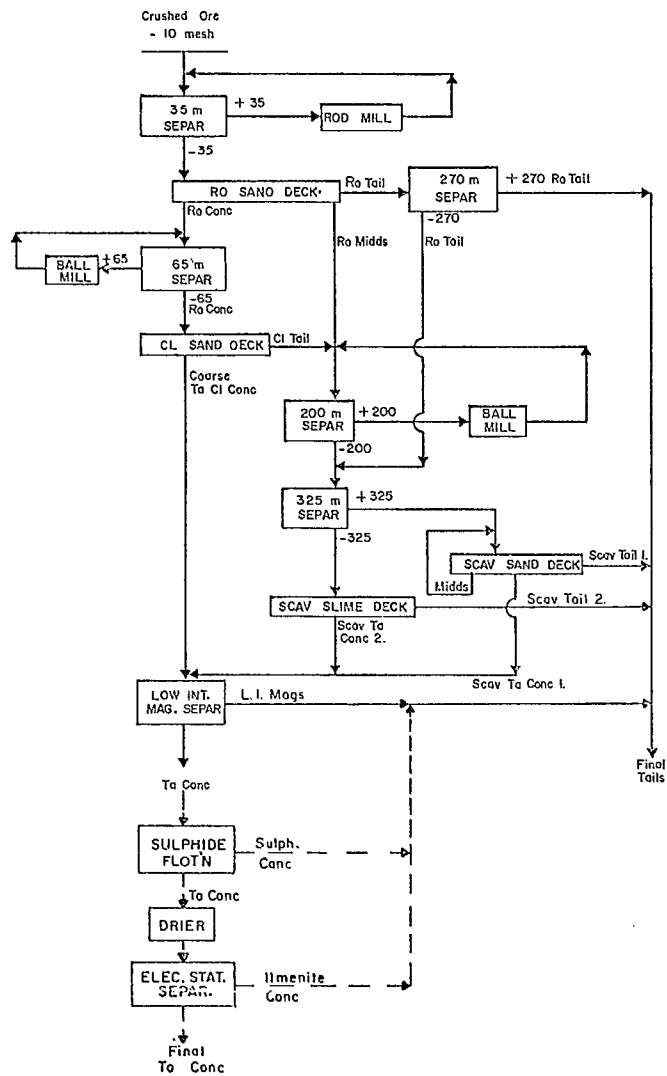
The flowsheet of this procedure is shown on page A-27.

Summary of results and conclusions: The following is a summary of the results that might be expected by supplementing the gravity operation at the Bernic Lake concentrator with slime-deck tabling of fines and re-ground middlings, followed by rejection of sulphides by flotation.

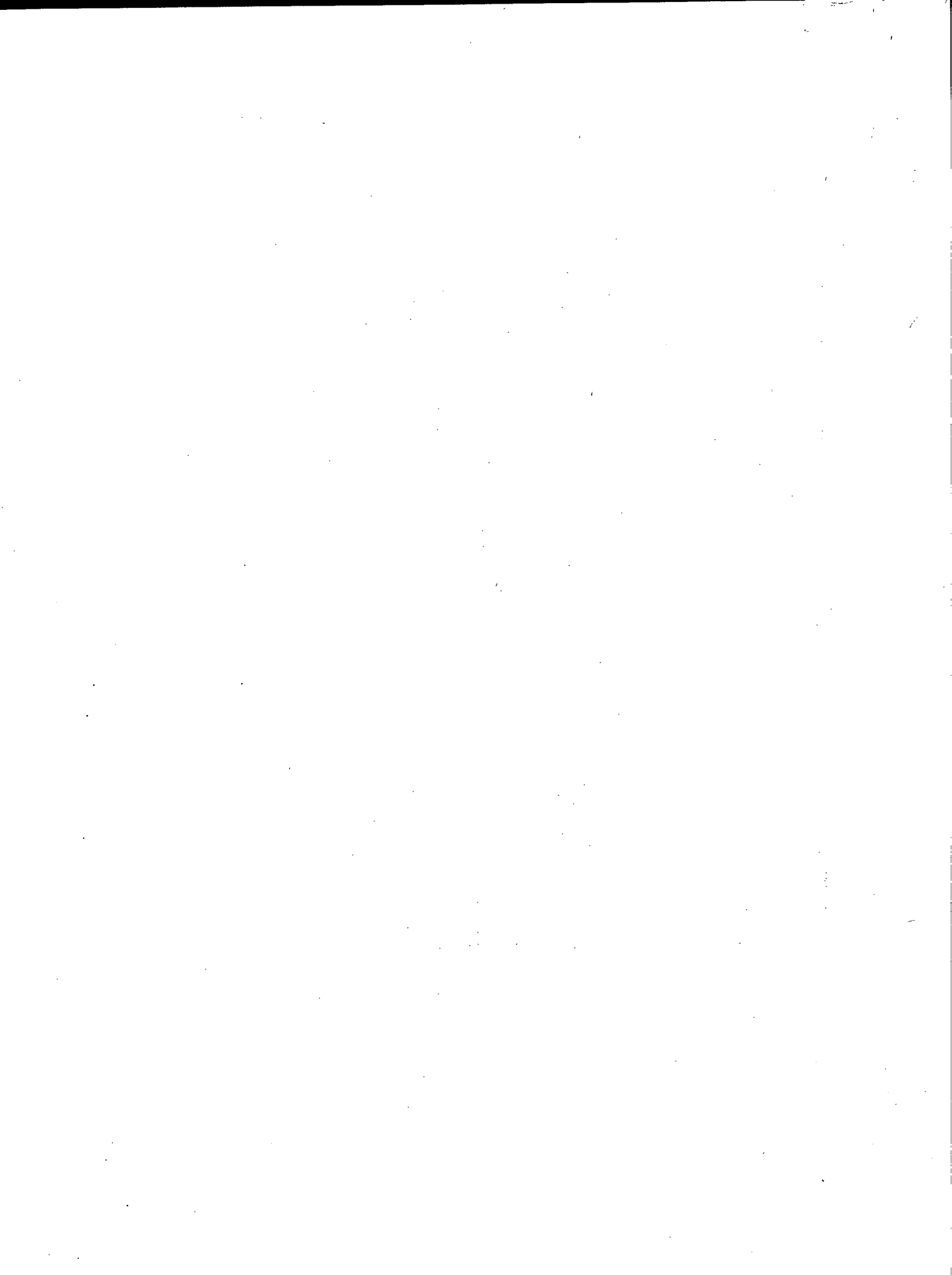
Date of sampling	Tantalum concentrate	
	% Ta ₂ O ₅ Assay	% Recovery
November 18, 1971	50.0	60.0
November 25, 1971*	51.12	66.0
June 1 and 2, 1972	52.65	55.5

*Rejection of sulphides not required

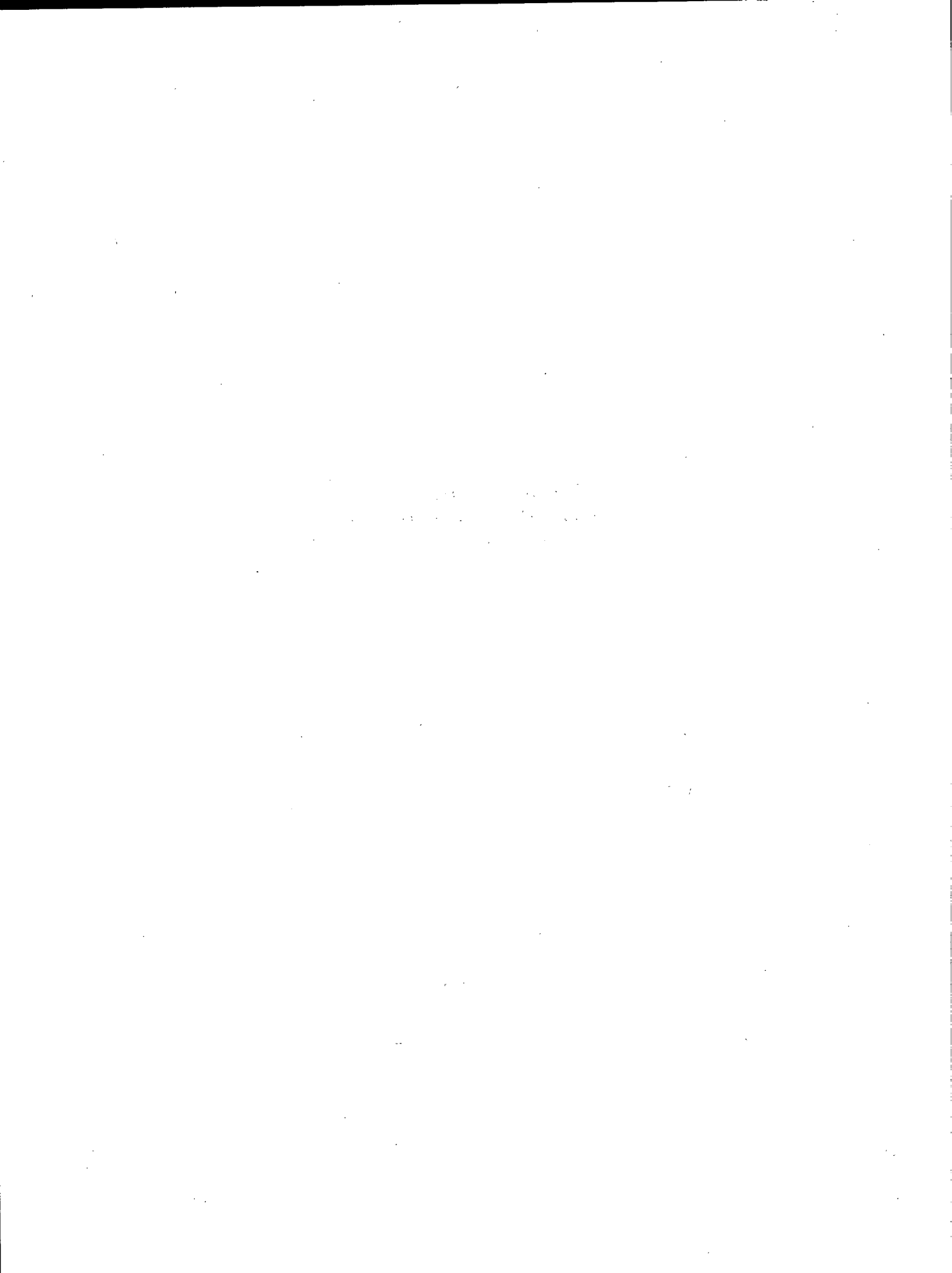
It was concluded that tantalum concentrates assaying 50% Ta₂O₅ with 53 to 66% tantalum recovery could be obtained from these refractory ores with the additional treatment illustrated in the flowsheet.



Flowsheet for Treatment of Finely Disseminated Tantalum Ores



APPENDIX B - PART 1
RESEARCH AND DEVELOPMENT ON NIOBIUM ORES
CANADIAN AND FOREIGN



CANADIAN RESEARCH AND DEVELOPMENT

Investigation Report No. MD3129, July 24, 1956

BENEFICIATION OF A NIOBIUM ORE FROM QUEBEC
COLUMBIUM LIMITED, SUBSIDIARY OF MOLYBDENUM
CORPORATION OF AMERICA, OKA, QUEBEC
USING CALCINATION AND SLAKING, FOLLOWED BY
STANDARD ORE CONCENTRATION PROCEDURES

by

D.E. Pickett

Ore sample tested: 6600 lb from Quebec Columbi-
um Ltd. near Oka, Quebec.

Mineralogy: Mineralogical examination of the ore
was not carried out.

On examination by hand lens this ore con-
tained calcite as the main gangue mineral with
apatite, mica, pyroxene and magnetite in minor
amounts. The niobium minerals - pyrochlore, ni-
ocalite and perovskite - were visible but sparsely
distributed through the gangue.

Analysis: This ore sample contained: 1.27%
Nb₂O₅, 1.33% SrO and 0.020% U₃O₈. Other
constituents were not analyzed.

Purpose of investigation: To produce a high
grade niobium concentrate amenable to chemical
extraction processes.

Methods of concentration: Calcining of the ore
followed by desliming, low and high intensity
magnetic separation, gravity concentration, elec-
trostatic separation and flotation.

Summary of results and conclusions: High-grade
niobium concentrates could not be produced from
this ore sample by the methods applied.

Mines Branch Investigation Report IR 59-20.

CONTINUOUS FLOTATION TESTS ON A NIOBIUM ORE
FROM THE COULEE-HEADWAY PROPERTY AT
OKA, QUEBEC

by

D.E. Pickett

Ore sample tested: Two ore shipments were re-
ceived for this investigation: 55 tons and 66.5
tons from the Oka district, Quebec, property of
Coulee Lead and Zinc Mines Limited, (later Colum-
bi-um Mining Products Limited).

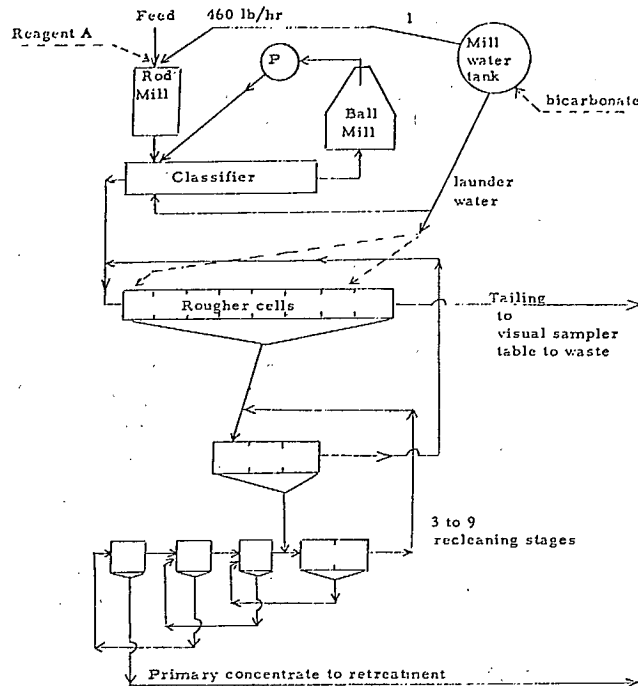
Mineralogy: See Mines Branch Investigation
Report 61-146, Appendix B, Part 2, page B-50.

Analysis: The average analysis for the 55 ton
sample was 0.324% Nb₂O₅; that for the 66.5
ton sample was 0.282% Nb₂O₅.

Purpose of investigation: The test was made to
investigate the operating variables of the flota-
tion process, particularly that involving the use
of bicarbonate solution as a modifier on a con-
tinuous basis as well as to provide concentrate
for extraction tests.

Method of concentration: Flotation. See flow-
sheet below.

Summary of results and conclusions: The best re-
coveries were obtained ... from an average feed
of 0.29% Nb₂O₅; 90% of the niobium was recov-
ered in a concentrate averaging 3.73% Nb₂O₅,
with an average tailing of 0.031%. No completely
satisfactory method for upgrading the 3% to 6%
Nb₂O₅ concentrate with a good recovery was
developed during the tests. The use of ammonium
bicarbonate was successful as was the combination
of reagents used as a collector.



Flowsheet for Continuous Flotation of Oka Ore

Mines Branch Investigation Report IR 59-17,
 March 2, 1959

PILOT PLANT TESTS ON A NIOBIUM ORE FROM
 NOVA BEAUCAGE MINES LIMITED,
 NORTH BAY, ONTARIO

by

D.E. Pickett

Ore samples tested: Four samples were received
 but no weights given.

Mineralogy: See Appendix B, Part 2, Investiga-
 tion No. MD3126, page B-51.

Analysis:

Shipment No.	Nb ₂ O ₅ %	U ₃ O ₈ %
1	0.73	0.05
2	0.69	-
3	0.81	-
4	0.62	-

Purpose of investigation: "The pilot plant tests
 were designed to obtain information about the
 process which could not be obtained in laboratory
 work."

Method of concentration: Sink-float; tabling
 tests, flotation and magnetic separation.

Summary of results and conclusions: Flotation, sink-float, tabling and magnetic separation tests were carried out on a sample containing approximately 0.75% Nb₂O₅ as pyrochlore.

Flotation concentrates containing up to 18% Nb₂O₅ were produced at recoveries up to 60%. Observations indicate that better recovery is possible.

The flotation process was proven to be a practical method of recovering pyrochlore concentrate. The economic limitations of overall niobium production from Beauceage ore can only be determined after doing more test work and a study of chemical extraction problems.

Preliminary testing indicated that sink-float would be valuable as a pre-concentration process. Tabling and magnetic concentration appear to have doubtful value.

Investigation Report IR 59-75, October 15, 1959

FLOTATION OF PYROCHLORE FROM
CANADIAN NIOBIUM ORES

by

D.E. Pickett

Ore sample tested: While the source of the ore in this series of tests was not specified, the presence of considerable niocalite would indicate that it came from Quebec Columbian Limited, Oka, Quebec. No weights are given.

Mineralogy: See Appendix B, Part 2, Mines Branch Investigation Report IR 58-55, page B-49.

Analysis: 1.27% Nb₂O₅; 0.02% U₃O₈, and 1.33% SrO. Approximate composition of the ore was calcite, 60%; apatite, 10%; diopside, 10%; mica, 10%; and niocalite, acmite, garnet, magnetite, pyrochlore and perovskite, 10%.

Purpose of investigation: To demonstrate in the flotation cell the behaviour of the niobium-bearing minerals as well as of minerals not containing niobium and to compare the results with bubble pick-up tests performed previously.

Method of concentration: Flotation, high intensity magnetic concentration and tabling.

Results and conclusions: While the pick-up behaviour was indicative of mineral behaviour, flotation test recoveries were not satisfactory due to the necessity of desliming and lack of selectivity among the collectors tested.

Sixth International Mineral Processing Congress, Transactions, June, 1963.

DEVELOPMENT OF A PROCESS FOR THE
CONCENTRATION OF OKA PYROCHLORE

by

H.L. Noblitt

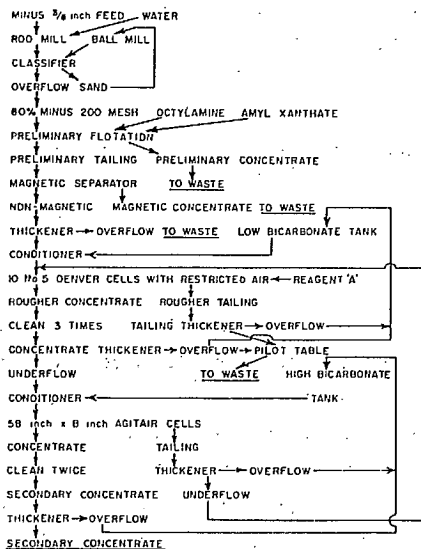
Ore sample tested: 120 tons of ore were treated in continuous testing. The ore came from the Columbian Mining Products property in the Oka district of Quebec.

Mineralogy: See Appendix B, Part 2, Mines Branch Investigation Report IR 61-146, page B-50.

Analysis: the average head analysis was 0.397% Nb₂O₅.

Purpose of investigation: This pilot plant investigation was performed to test, in continuous operation, the addition of secondary flotation of pyrochlore and perovskite with return of all middling products to rougher flotation as well as the effect on reagent consumption of a preliminary flotation of partly decomposed micas.

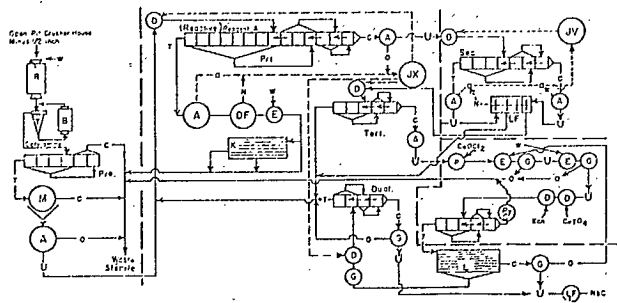
Methods of concentration applied: grinding to approximately all minus 65 mesh in water, floating off partly decomposed micas with a short chain monoamine; magnetic separation of the tailing, dewatering of the non-magnetic fraction, repulping in low bicarbonate solution (0.3% ammonium bicarbonate); floating with Reagent A, cleaning this concentrate three times with return of tailings to the previous cells, dewatering of the rougher tailing with return of low bicarbonate solution to the head tank; thickening of the primary concentrate with recovery of solution; repulping of primary concentrate with 3% ammonium bicarbonate, floating and two cleanings of the secondary rougher concentrate, with return of the cleaner tailings as in the low bicarbonate circuit; thickening of the cleaned secondary concentrate with return of the overflow to the high bicarbonate head tank. The pH in the primary circuit was buffered at 8.1. In the secondary circuit it was somewhat higher. The air supply to the cells was restricted to provide a froth no higher than one inch. Reagent consumption was approximately 1 lb of Reagent A and 0.4 lb/ton of octylamine per ton of ore treated. Bicarbonate concentrations were determined by titrating with hydrochloric acid with methyl orange as indicator. The flowsheet is shown below.



Flowsheet, pilot run 2

Summary of results: At the end of the test with all sections operating smoothly the primary rougher tailing contained 0.02% Nb_2O_5 , the preliminary concentrate 0.55%; the magnetic concentrate, 0.145% and the secondary concentrate 13 to 15% Nb_2O_5 . Overall recovery was approximately 85.5% but at times this rose to 92.5%. Over the period of a month, the pilot plant was kept in continuous operation.

Conclusions: At a grade of 13% Nb_2O_5 the concentrate contained 26.5% of combined pyrochlore and perovskite. Since 1960 enough progress has been made to assure the upgrading of this concentrate to a commercial grade without serious loss as any rejects from this upgrading process can be returned to the primary flotation feed. As there was no desliming, any pyrochlore that was liberated had an opportunity to float. The all alkaline circuit does not corrode iron but does corrode copper, nickel, cobalt and zinc. This process reduced to one-quarter the amount of niobium minerals and, of uranium and thorium being rejected to the tailings disposal area. With a minimum supply of air to the flotation cells, rejection of apatite was good. However, if it is desired to concentrate apatite it is only necessary to float the primary tailings again using a high froth to concentrate that mineral as well. The proposed flowsheet is given below.



Proposed production flowsheet

The legend of the flowsheet is given in the original paper.

Mines Branch Investigation Report IR 69-56,
June 1969

CONCENTRATION TESTS ON A PYROCHLORE ORE
FROM ARGOR EXPLORATIONS LIMITED

by

G.W. Riley

Samples tested: Samples of middlings from tabling the 14- to 28-mesh and 28- to 48-mesh fractions made at Lakefield Research Ltd were received. No weights were given. Fifty pounds of original ore were also received. Later a shipment of 5000 lb of ore was received for pilot plant use.

Mineralogy: No mineralogy was provided. Only pyrochlore and mica are mentioned.

Analysis: A drill core sample gave 0.54% Nb₂O₅. Tabling feed at Lakefield was reported to have contained 0.56% Nb₂O₅.

Purpose of the investigation: To find a means of pre-concentrating the ore by gravity methods to a 90% recovery of the Nb₂O₅ with a ratio of concentration of 5 to 1. It was proposed to table the 14- to 48-mesh fraction to produce a high grade concentrate and a low grade middling. The middling was to be tabled again to produce a high grade concentrate but this could not be done. Tests were proposed to find alternative methods.

Method of concentration: Jigging, spiralling and air tabling of a 5000-lb shipment of ore.

Summary of results: From the 14 to 48 mesh fraction tabling and jigging recovered over 90% of the Nb₂O₅ at a ratio of concentration of slightly more than 5 to 1. A spiral and jig treating the 14- to 30-mesh fraction recovered 87.6% of the Nb₂O₅ at a ratio of concentra-

tion of 4.5 to 1. Air tabling of the same fraction recovered 74.2% of the Nb₂O₅ at a ratio of concentration of 7.8 to 1. A spiral treating the raw minus 14 mesh ore recovered 83.7% of the total Nb₂O₅ at a ratio of 4.2 to 1.

Conclusions: The flotation feed obtained by combining the spiral and jig concentrates with the untreated minus 60-mesh material would amount to about 40% of the ore and would contain over 90% of the Nb₂O₅ in the ore.

Canadian Patent 599,760, June 14, 1960

FLOTATION PROCESS FOR CONCENTRATING
NIOBIUM-BEARING MINERALS

by

H.L. Noblitt

This patent refers to the discovery that when flotation of pyrochlore and niobium-bearing perovskite is practised in a solution of a soluble bicarbonate using a mixture of diamines and a wetting agent as collector, the recovery of desired mineral and the rejection of waste are both increased. The most readily available soluble bicarbonates are those of sodium, ammonium and potassium. Of these, ammonium is the most useful as it is relatively inexpensive, is readily available and, except for a slight ammoniacal smell around the cells, is non-toxic.

Example I consists of fifteen tests in which each of three different ore types from Oka, Quebec, were ground and all plus 35-mesh grains were removed. The solutions used on each ore were: distilled water; tap water acidified to contain 0.85 lb per ton of sulphuric acid; city tap water plus 6 lb per ton HN₄HCO₃; tap water plus 6 lb per ton NaHCO₃; and tap water plus 7.5 lb per ton of KHCO₃. The collector used was 2.8 lb per ton of feed of Reagent A: 1

part Duomeen T, 1 part Ultrawet DS plus 2 parts amine 220 reacted together and emulsified in water. Ore A was mostly dolomite with very little mica and pyrite. It was deeply weathered and iron-stained, (0.18% Nb_2O_5). Ore B was fresh with little mica and much calcite, pyroxene

and magnetite, (0.22 to 0.25% Nb_2O_5). Ore C was fresh, 60% calcite with much biotite and diopside, 6% apatite and lesser amounts of other carbonates and silicates (0.31% Nb_2O_5). The reagents used and the results obtained are given in the table below.

Modifier	Lb per ton of solution	% recovery	% Nb_2O_5	% Weight floated
Ore A				
1. Distilled H_2O	-	48.82	2.05	4.83
2. Sulphuric acid	0.85	84.23	0.91	17.96
3. NH_4HCO_3	6	84.83	2.85	6.62
4. $NaHCO_3$	6	81.26	5.70	2.37
5. $KHCO_3$	7.5	85.89	2.45	6.32
Ore B				
6. Distilled H_2O	-	90.96	0.785	29.61
7. Sulphuric acid	0.85	92.32	0.80	28.84
8. NH_4HCO_3	6	95.48	0.765	32.09
9. $NaHCO_3$	6	90.57	0.925	24.96
10. $KHCO_3$	7.5	87.16	0.705	33.19
Ore C				
11. Distilled H_2O	-	86.55	0.90	27.54
12. Sulphuric acid	0.85	86.82	1.06	26.00
13. NH_4HCO_3	6	90.02	1.00	28.52
14. $NaHCO_3$	6	88.20	1.115	25.10
15. $KHCO_3$	7.5	81.43	1.08	24.81

With bicarbonates, Ore A gave good recovery and good grade; acid gave good recovery but low grade; water gave low recovery and fair grade. With Ore B all recoveries were higher but grades were lower due to flotation of green silicates. With Ore C, all but $KHCO_3$ gave good recoveries with grades intermediate between Ores A and B.

Example III was a test in which 30 tons of Ore C were treated over 130 hours of continuous flotation at about 450 lb per hour. In the first 13 hours the solution contained 6.78 lb of

$KHCO_3$ per ton, adding 2.35 lb of collector per ton gave a recovery of 87.7% and a grade of 4.2% Nb_2O_5 after four cleanings. In the next 64 hours the solution contained 5.66 lb of NH_4HCO_3 per ton. With an average of 2.67 lb of Reagent A per ton of feed and 5 cleaning steps, recovery was 90.5% and concentrate grade was 3.65% Nb_2O_5 with rejection of 92.12% of the feed. In the next 12 hours the solution used contained 5.66 lb per ton $NaHCO_3$. The reagent addition was 3.33 lb per ton, recovery was 89.5% and concentrate grade was 3.62% Nb_2O_5 , with

waste rejection of 91.35%. In the remainder of the test NH_4HCO_3 solution was used as above with 2.41 lb per ton of collector to attain a recovery of 90.7%, an average grade of 4.0% with 7 cleaning steps. Waste rejection was 93.9%. Overall recovery was 90.0% at a grade of 3.89% Nb_2O_5 and a tailing of 0.031% Nb_2O_5 from 0.31%. With NH_4HCO_3 the pH was steady at 8.0 to 8.1.

FOREIGN RESEARCH AND DEVELOPMENT

U.S.S.R. Patent 66, 801, Aug. 31, 1946

CONCENTRATION OF PYROCHLORE

by

A.P. Diomidov and V.K. Babakh

Pyrochlore is floated with oleic acid as collector at a temperature not below 25°C in a medium of pH 8.4 to 9.6; Nb_2O_5 is extracted from the concentrate.

British Patent, 800,717, Sept. 3, 1958

SEPARATION OF PYROCHLORE FROM CALCITE, APATITE AND SILICA BY FLOTATION

by

H.G. Burks

Pyrochlore and silica are separated from calcite and apatite by froth flotation of an aqueous suspension of the mixed minerals with an aliphatic diamine having 14-20 carbon atoms in the molecule. The float is treated to destroy the diamine activity and the product is subjected to froth flotation with an aliphatic monoamine

containing 10-12 carbon atoms to separate the pyrochlore from SiO_2 . Preferably a wetting agent should be used in the first flotation. If iron is present, intermediate flotation or complexing of the iron ions may be required before the final separation from SiO_2 . Flotation of sovite ore containing 0.46% Nb_2O_5 using amine 220, 0.6; Duomeen T, 0.08; and Ultrawet 40 A, 0.3 lb/ton produced apatite-calcite tailings free of pyrochlore. The float was concentrated by another flotation without additions. Flotation of this concentrate with 10% Na_2SiO_3 and methyl isobutyl carbinol produced a froth containing FeS and mica. Magnetite and ilmenite were removed magnetically from the tailings, which were then conditioned with H_2SO_4 to pH 5.5-6, dodecylamine acetate, 1.2 lb/ton and sodium hexametaphosphate, 1.6 lb/ton and subjected to flotation to separate the pyrochlore from silica, coarse mica and other silicates. The froth concentrate includes about 70% of the pyrochlore in the ore and contains 45 to 50% plus small amounts of pyrrhotite, ilmenite, apatite, quartz and actinolite".

British Patent, 798,768, July 23, 1958.

FLOTATION OF NIOBIUM MINERALS

by

Kennecott Copper Corp.

Niobium minerals, such as columbite, pyrochlore and perovskite, are separated from finely ground ore, suspended as a pulp in water, by adding about 0.2 to 0.4% 8-quinolinol (Oxine), by weight of ore and aerating the pulp with agitation to produce a froth in which the minerals are collected. Reagent consumption is minimized by first removing the slimes as well as gangue constituents such as calcite, apatite, mica, magnetite and iron sulphides, dewatering the residue and repulping it with used reagent-bearing solu-

tion before flotation. Calcite and apatite may be removed by flotation with a fatty acid collector, mica and pyrite by flotation with a sulphonated petroleum oil and a xanthate and magnetite by magnetic separation.

By upgrading ore in this way and recycling used solution several times, recoveries greater than 90% can be attained with less than one-third of the reagent required for 90% recovery in one flotation of the original ore. Crude 8-quinolinol gives satisfactory results. It is made by mixing o-nitrophenol 5, o-aminophenol 8.3 and glycerol 18 g, slowly adding 8.2 mL concentrated sulphuric acid, boiling for 5 hours with a reflux condenser, cooling and diluting to 250 mL. About 60 to 80 mL of this solution may be used instead of 2 g pure 8-quinolinol with equal recovery of Nb_2O_5 .

Carbonatite-type niobium ore containing about 0.66% Nb_2O_5 was ground wet to 90% minus 200 mesh, deslimed and freed from iron sulphides and magnetite as described above. The pulp was filtered and 2 parts, weighing 1 kg each, were separately repulped, conditioned with 2.5 g 8-quinolinol and subjected to flotation. The two froth concentrates were combined, conditioned with 0.5 g 8-quinolinol and subjected to flotation. This was repeated three times, by using the solution from the preceding step for dilution water each time until a concentrate weighing 164.8 g and containing 6.6% Nb_2O_5 was recovered in the final froth. This represented a recovery of 81.84% of the Nb in the ore. Most of the tailings discarded contained 0.04% Nb_2O_5 . The final tailing weighed 15.8 g and contained 0.85% Nb_2O_5 representing 1% of the niobium in the ore.

Tsvethykh. Metal. i Zolota, (U.S.S R.) 1958

FLOTATION PROPERTIES OF PYROCHLORE,
ZIRCON AND ASSOCIATED MINERALS

by

G.M. Shapovalov and S.I. Pol'kin

To investigate the flotability of pyrochlore and zircon from commercial ore, flotation properties of the following pure minerals contained in this ore and presenting the main difficulties in their selective separation, were studied: pyrochlore, zircon, ilmenite, aegirite-augite, limonite and microcline. The use of Na_2S in flotation of pyrochlore and zircon with sodium oleate sharply increases selectivity of the process and for still better selection, Na_2S together with Na_2CO_3 should be used. The minerals fix 4 to 10 g Na_2S per ton. Preliminary treatment in $FeCl_3$ solution increases this figure to 7-27 g per ton. At pH less than 2, sodium alkyl sulphate is a selectively active collector; $FeCl_3$ activates aegirite-augite and mylonite at pH 4 to 9 in their flotation with sodium alkyl sulphate and $CaCl_2$ is a strong activator for pyrochlore, zircon, aegirite-augite and microcline in weakly acid and especially in alkaline medium. To prevent the activative action of Fe and Ca on the gangue, pyrochlore and zircon should be floated at a pH below 2. In flotation with sodium alkyl sulphate, Na_2SiO_3 depresses only pyrochlore. A mixture of cupric sulphate and Na_2SiO_3 is a depressor for limonite, microcline and aegirite-augite in flotation with alkyl sulphate. The same mixture in 1:2 ratio has a harmful depressive action on limonite and microcline in flotation with sodium oleate. (From Referat. Zhur., Met. 1959, Abstract No. 17130.)

Izv. Vysshikh Uchebn. Zavedenii, Tsvetn. Met. 5, 1962

FLOTATION OF COLUMBITE AND ASSOCIATED
MINERALS WITH DIFFERENT COLLECTORS

by

S.I. Pol'kin and Te-Ming Ch'eng

Conclusions:

1. Columbite is the most important source for the production of niobium. The fine impregnation and high brittleness of columbite often cause great losses in the concentration of columbite-bearing ores and in the finishing of crude concentrates by gravity methods; the use of flotation in combination with other methods can ensure maximum extraction in the concentration of columbite-bearing ores.
2. The flotation properties of columbite and columbite-bearing ores have still not been studied and for this reason, studies of columbite and some associated minerals have theoretical and practical importance.
3. It has been revealed experimentally that columbite has high floatability with sodium oleate, sodium isooctylphosphate, oxidized petrolatum and ANP-14 cation collector; the floatability of columbite is very close to that of garnet, which makes it difficult to separate them without selective reagents.
4. The floatability of zircon and columbite is rather close, but they can be separated in weak acid medium with oxidized petrolatum with consumptions of up to 4000 g/t columbite and garnet can also be separated from albite in these conditions.
5. It is possible to separate columbite, garnet and zircon from albite in flotation with sodium isooctylphosphate or sodium cetylsulfate in acid medium; the flotation activity of albite increased drastically when ANP-14 is

used; it can be separated into the froth product from columbite, zircon and garnet at pH less than 5.

Tsvetnye Metally. 39(9), U.S.S.R. 1964

MECHANICAL CARRYOVER OF SLIME PRODUCTS
BY FLOTATION

by

V.I. Klassen and S.A. Tikhonov

This article mentions the use of sodium alkyl sulphate, terpinol and sodium oleate. It is used on ore containing pyrochlore and albite and on another niobium-bearing ore.

Tr. Tsentv. Nauchn. Issled (Gornorozved Inst), U.S.S.R., 1964

METHOD OF CONCENTRATING FINE-IMPREGNATED
PYROCHLORE DUST ORES

by

S.N. Rossovskii

Oleic acid, sulphate soap, tallow oil or oxidized petrolatum can be used as collectors at a pH 4 to 6. Concentrates were cleaned three or four times in weakly acid medium. This increased concentration 15- to 20-fold in cleaned concentrate. Further treatment is made by either removing feldspar after acid treatment at pH 5 to 6 by flotation with oleic acid and magnetic separation of ionic product, or by flotation with an alkyl sulphate or cationic reagents. Then the Nb - Zr concentrate was separated. (Chem. Abstr. 1965, Vol. 63.)

Tsvetn. Metal, 36(5), U.S.S.R., 1963

FLOTATION SEPARATION OF PYROCHLORE,
ILMENORUTILE AND ZIRCON AND THE
MECHANISM OF ACTION OF REAGENTS

by

S.I. Pol'kin and P.I. Andreev

The effect of H_2SO_4 on the physical-chemical and flotation properties of pyrochlore, ilmenorutile and zircon in conjunction with sodium oleate (I), sodium octyl sulphate (II) and sodium silicate (III) was examined. Tagged H_2SO_4 with ^{35}S and I with ^{14}C were also used. Preliminary acid treatment increased markedly the amount of (I) attached to the mineral surface and this amount increased with the amount of (I) added. Acid treatment shifted the electrokinetic potential (EKP) toward the positive side and enhanced the attachment of (I) to the mineral surface. Acid treated ilmenite and zircon had a positive EKP and were floated with a markedly smaller amount of (I) than was pyrochlore which had a negative EKP. When crude collective concentrate was treated with H_2SO_4 , increasing the amount of acid increased the amount of (I) desorbed and decreased the floatability of minerals. Calculations showed, that for complete extraction, the mineral surface must be coated with a collector reagent film $>$ one molecular thickness. Increasing the amount of collector reagent attached to the surface increased the rate of flotation and the extraction of minerals into the froth product. Desorption of (I) with H_2SO_4 decreased the extraction sharply and H_2SO_4 acted as a depressant. The sulphate formed on the mineral surface by reaction with H_2SO_4 could not be washed off with water. When the minerals were first treated with (II) a larger amount of H_2SO_4 became attached to the surface. The oleate ion could displace the sulphate ion, forming metal oleates with the mineral surface. The regulating agent (III) reacted with

zircon, a silicate preventing the attachment of (I) and depressing the flotation. As pyrochlore and ilmenite were not silicates, they were not affected by (III) to the same extent and could thus be separated from zircon.

Nauchn. Irkutskii Gos. Nauchn-Issled. Inst.,
Redkirkh Metal No. 11, U.S.S.R., 1963

FLOTATION PROPERTIES OF PYROCHLORE

by

D.I. Kogan and L.V. Katashin

Surfaces of pyrochlore (1) from a pegmatite deposit and pyrochlore (2) from a carbonatite deposit containing SiO_2 , 3.50 and 3.15%; Nb_2O_5 , 65.35 and 63.90%; CaO , 15.00 and 16.60%; MgO , 0.50 and 0.91%; Fe_2O_3 , 0.07; FeO , 0.16 and 1.7%; TiO_2 , 4.70 and 3.50%; Na_2O , 6.60 and 5.73%; other, 1.71 and 1.27% respectively, have a negative charge. The pyrochlore (2) has a higher negative charge than pyrochlore (1). Flotation properties of pyrochlore, magnetite and carbonates depend on the value of the surface charge, which is controlled by the concentration of ions in the pulp. Pretreatment of the mineral surface with HF and HCl caused depression of pyrochlore, magnetite and carbonates because of increase of negative charge. Metal cations were washed from the mineral surface and new surface substances were formed during pretreatment with H_2SO_4 . The depressing effect of waterglass on minerals is caused by adhesion to the mineral surface of micelles of silicic acid. In this case the decrease of negative charge cannot suppress the wetting of the surface because of the high hydrophilicity of the micelles. Only the NH_4 ions, which are concentrated in the Stern layer, did not affect the floatability of pyrochlore, magnetite and carbonates. A combination of H_2SO_4 and NH_3 ions is capable of inducing selective

depression of pyrochlore and magnetite from carbonates. The Na_2S selectively affects pyrochlore permitting its use in selective flotation of pyrochlore and carbonates.

(IV) emulsified and saturated with oxygen and 175 g/ton Na_2SiF_6 . The products contained 11.3% ZrO_2 for a recovery of 69% and 15.2% TiO_2 for a recovery of 44.8%."

Izv. Akad Nauk. U.S.S.R., Met. Gorn. Delo, 1963

THE EFFECT OF EMULSIFICATION AND OXYGEN ON
THE PROPERTIES OF OLEIC ACID AS A
COLLECTOR IN SELECTIVE FLOTATION OF
PYROCHLORE-ZIRCON CONCENTRATE

by

S.I. Pol'kin, L.D. Plaksina and V.A. Chanturiya

"Flotation of pure pyrochlore (I), zircon (II) and ilmenorutile (III) with 100 to 2000 g/ton oleic acid (IV) as collector was studied in a laboratory flotation machine at pH 6.0. The minerals were pretreated with 30% H_2SO_4 and washed with water. When (IV) was added drop by drop, the recovery of (I), (II) and (III) was below 60%. By using emulsified (IV), 96% of (II) and 94% of (III) were recovered as froth products, while only 0.05% of (I) was extracted. Separation of (II) and (III) in the froth product was 98% and of (I) 5%. The best properties of (IV) were obtained after 30 minutes saturation with oxygen. In all tests the use of over 100 g had little effect on flotation results. Flotation of an ore concentrate containing 5.7% Nb_2O_5 ; 1.8% ZrO_2 and 2.5% TiO_2 was studied. Pretreatment with 370 g/ton H_2SO_4 gave a concentrate of (I) containing 35% Nb_2O_5 with a recovery of 62%. Subsequent separation of (II) and (III) was carried out by using 500 g/ton

Nauch. Tr. Irkutsk. Gos. Nauch-Issled, U.S.S.R.,
Inst. Red. Met. No. 13, 1965

TECHNOLOGICAL PROPERTIES OF
PYROCHLORE-CONTAINING SLIMES FROM A
CARBONATITE DEPOSIT

by

Yu I. Razvozhayev, and A.I. Khodyreva

An industrial process, including disintegration, classification and desliming of particle size minus 0.01 mm was developed for concentration of ores from the weathering crust of a carbonatite deposit. These ores have a complex mineralogical composition. After concentration by a combined procedure providing for gravity flotation of apatite and extraction of pyrochlore, the dephosphorized slimes of particle size minus 0.074 mm containing 1% Nb_2O_5 and 1.5% P were obtained. The concentrated slime product had a high dispersity. The first concentration of slimes for concentrating the pyrochlore to a Nb_2O_5 content of 2.4 to 2.6% can be done by direct flotation. The addition of regulators to the pulp had no appreciable effect. The presence of surface films of iron oxides on the pyrochlore and on the rock-forming minerals reduced the Nb concentration by a factor of over 2.4 to 2.6.

Sovrem. Sostoyanie Zadachi Selek. Flotatsii Rud, 1967, U.S.S.R., from Rev. Zh., 1967, Abstr. No. 11 G80.

DIFFERENTIAL FLOTATION OF WEATHERED CRUST
MINERALS OF NIOBIUM CARBONATITE ORES

by

M.P. Potekhina, S.I. Pol'kin and A.N. Danilina

During flotation of coarse Nb concentrate, niobium minerals are differentiated from oxidized iron minerals and apatite by using an acid treatment and flotation with the cationic collector ANP in a HF medium. In a niobium concentrate containing 38.4% Nb_2O_5 and 5% P_2O_5 , 88.5% of the Nb_2O_5 was extracted. (Chem. Abstr. Vol. 68, 1968, No. 116,538).

Nauch'Tr, Irkutsk, Gos. Nauch. Issled. Inst. Redk. Tsvet. Metally. No. 19, (U.S.S.R.), 1968

FLOTATION OF PYROCHLORE FROM SLIMES LEFT
AFTER GRAVITATIONAL CONCENTRATION OF
RARE METAL CARBONATITE ORES

by

L.V. Katashin and D.I. Kogan

The size of the material used for flotation was minus 0.074 mm, plus 0.020 mm. Two methods were tested: (1) direct flotation of pyrochlore and (2) separation of easily floated components of carbonates and depression of pyrochlore by sodium silicate and ammonium sulphate with subsequent flotation of pyrochlore. The concentrate contained 34.1% Nb_2O_5 .

Obogashch. Rud. 13(1), U.S.S.R. 1968.

CONCENTRATION OF COMPLEX PYROCHLORE-CONTAINING
ORES BY FLOTATION

by

D.I. Nedogovorov and S.N. Rossovskii

A process for the enrichment of a complex pyrochlore ore containing substantial amounts of aegirite, mica, calcite and other minerals with flotation properties very similar to those of pyrochlore involved two-stage flotation, desliming of the concentrate in a hydroclone, magnetic separation of the magnetic sands fraction; further flotation of the non-magnetic fraction and jet flotation of the primary and secondary slimes. This produced a final concentrate with 4 to 5% Nb_2O_5 , extraction of the niobium with the concentrate being less than or equal to 71% and a slime concentrate containing 0.46% Nb_2O_5 which is processed by a hydrometallurgical method.

Nauch. Tr. Irkutsk, Gos. Nauch. Issled. Inst. Redk. Tsvetnye Metally, (U.S.S.R.), 1968

FLOTATION EXTRACTION OF PYROCHLORE FROM
SLIMES OF WEATHERING CRUST OF
FERRUGINOUS ORES

by

Yu I. Razvozhayev

The conditions were determined with subsequent separation of iron oxides and hydroxides and with concentration of the pulp from chemical separation. The composition of the pyrochlore

concentrate may be improved by HCl as a solvent for iron oxides and hydroxides. The consumption of HCl is 900 kg/t pyrochlore product. The optimum consumption of Na_2SiF_6 for flotation was 10-15 kg/t of pulp. In the first flotation product containing 20% Nb_2O_5 , 80% of the Nb was extracted. The separation of amphibolite and ilmenite was carried out by flotation in a 5% solution of H_2SO_4 . Thus 70.6% of the niobium was separated in the pyrochlore product and 13.8% Ti and 5% Nb in the ilmenite product.

Obogashoh. Rud. 13(5), U.S.S.R., 1968.

EXTRACTION OF NIOBIUM-CONTAINING MINERALS
FROM TAILINGS OF GRAVITATIONAL CONCENTRATION
BY THE IM-50 REAGENT

by

V.S. Salikov, K.F. Serdyuk and N.P. Khabotova

On the basis of results obtained on pilot plant equipment, a method is given for the extraction of Nb_2O_5 minerals from the tailings of gravitational concentration by flotation of pyrochlore using the IM-50 Reagent (Hexamate or Hydroxamate). The resulting concentrate consisted of 4 to 5% pyrochlore, 60-70% ilmenite, 13-15% titanite and 3-6% zircon and other minerals, i.e., the concentrate contained 85% of minerals with 0.5 to 62.5% Nb_2O_5 ; 3-5% minerals with 0.06-0.08% Nb_2O_5 and only 8-12% feldspar without Nb_2O_5 . The synthesis of IM-50 consisted of two stages: (1) the recovery of methyl ethers of carboxylic acids, and (2) the condensation of the ethers with hydroxylamine sulphate in acid medium. The reagent, IM-50, is a yellow brown liquid with a strong alkaline reaction. (Chem. Abstr. Vol. 21, 41, 358), 1969.

Tr. Nauch. Tekh-Konf Inst., Posv 50-Letiyu Velikoi Okt. sots. Revol., U.S.S.R., 1968. From Ref. Zh., Khim. 1969, Abstr. No. 84151.

IMPROVEMENT OF FLOTATION CONCENTRATION SYSTEMS
FOR NIOBIUM-TANTALUM ORES

by

S.I. Gorlovskii and N.P. Khabotova

A flotation technique for niobium-containing ores and tailings from gravity concentration was developed using the IM-50 Reagent, including sulphide-calcite flotation, desliming, rougher flotation with IM-50 Reagent and several cleaning operations. The effectiveness of the technique was due to conditioning the flotation feed at a pH of 4.2 - 4.5 and a fast increase to 5.8 - 6.0 with Na_2CO_3 or NaOH during flotation. The cleaning operations were conducted at a pH below 2.

Obogashoh. Rud., 15(3), U.S.S.R., 1970.

FLOTATION BENEFICIATION OF RARE
METAL CARBONATITES

by

B.V. Levinskii and L.I. Nikonova

Dimethyl alkylammonium chloride provided higher selectivity than the ANP reagent (primary salts of amines) in separation of pyrochlore and apatite to form separate concentrates. This was proven during experimental flotation of a Ta-Nb ore containing 75% carbonates as calcite and ankerite, 13% silicate minerals as pyroxene,

aegirite, feldspars and quartz, 7% apatite and 4.5% mica. Of the Ta and Nb, 80% are bound in hachettolite and 15-18% in pyroxene and mica. The total content of commercial components is 0.11% Nb_2O_5 , and 2.47% P_2O_5 .

Obogashch. Rud. 19(4), U.S.S.R., 1974.

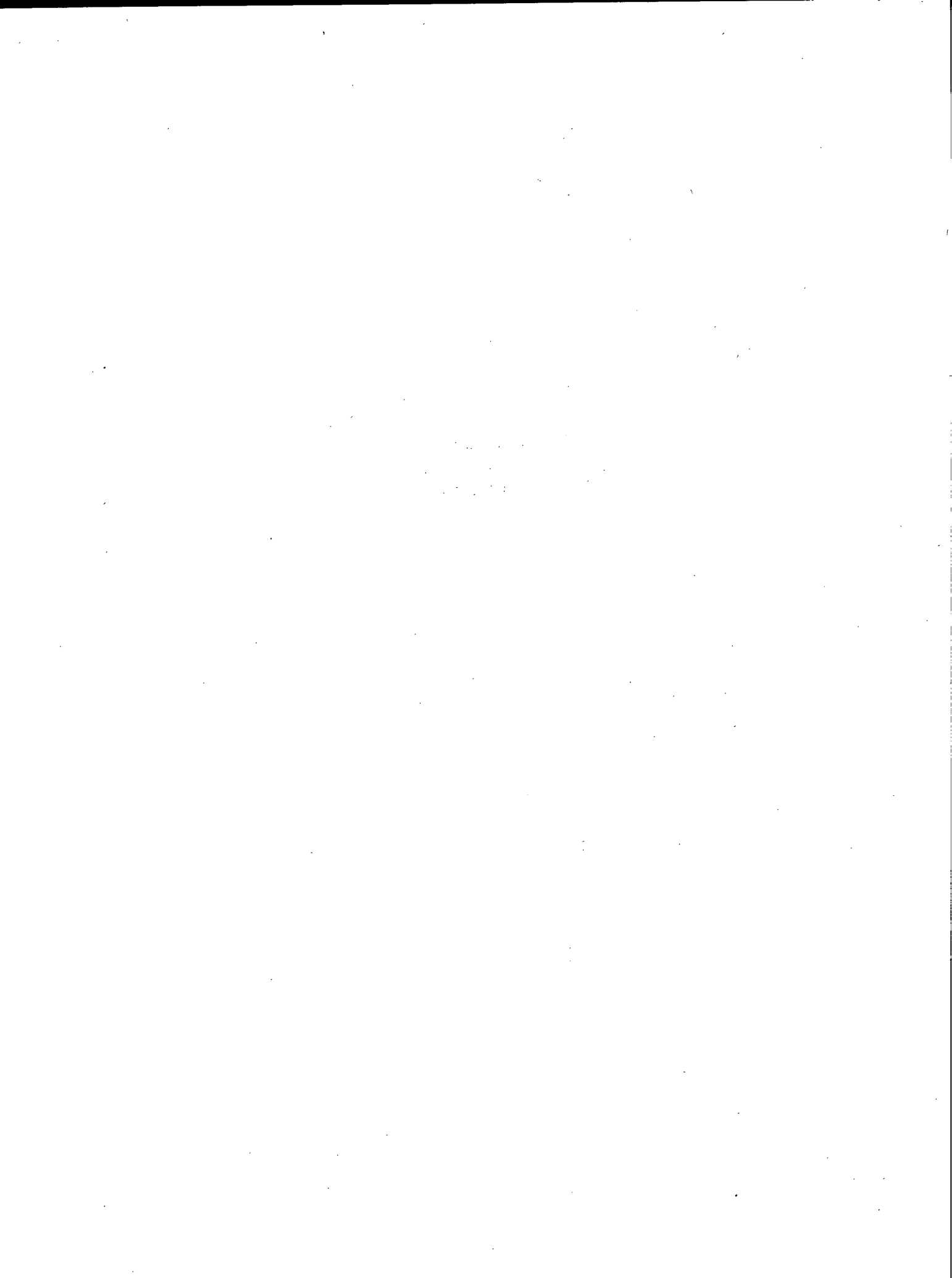
FLOTATION OF TITANIUM-TANTALUM-NIOBIUM
MINERALS WITH REAGENT IM-50 USING A
CIRCULATING WATER SUPPLY

by

S.I. Gorlovskii, B.A. Zav'yalov, A.A. Seledkov,
I.D. Ustinov, N.P. Khobotova and V.S. Shemyakov

A method was developed for purification of the circulating water used during flotation of Ti-Ta-Nb minerals by IM-50 flotation agent. The method is based on the precipitation of mixed iron oxyhydrate, hydroxamate and carboxylate flakes, formed by the addition of 0.5 g/L $FeSO_4$ at nearly neutral pH of 7.5 - 8.0 adjusted by alkali. The high rate and degree of flake precipitation was obtained by the use of flotation tailings as solid phase carriers. The laboratory and industrial experiments showed that water, purified by this method of settling, can be recirculated and used efficiently for ore flotation. The method probably can be used in flotation of cassiterite, tungsten and other ores and in purification of waste waters. (Chem. Abstr. Vol. 83, 1975, No. 101062r).

APPENDIX B - PART 2
MINERALOGICAL INVESTIGATIONS ON CANADIAN
NIOBIUM ORES



CRYSTAL STRUCTURE OF PYROCHLORE

Pyrochlore has been classified with kop-

pite, pyrrhotite, schneebergite and others (12). It has cubic axes, belongs to space group $O_h^7 - Fd\bar{3}m$ and has the general formula: $R_2Q_2X_7$. There are eight formulae in the unit cell as shown in table below.

Mineral Name	Formula			$a, \text{Å}$	x
	R	Q	X		
Koppite	(Ca,Ce,Na,K) ₂	(Nb,Fe) ₂	(O,OH,F)	10.37	0.19
Pyrochlore	(Na,Ca,Y,Ce,Th,U) ₂	(Ti,Ta,Nb) ₂	(O,OH,F)	10.33 to 10.38	1/5-1/6
Pyrrhotite	(Ca,Na,Fe) ₂	(Nb,Ta,Ti) ₂	(O,OH,F) ₇	10.38 to 10.41	
Schneebergite	(Ca,Na,Fe) ₂	(Sb) ₂	(O,OH) ₇	10.296	

Ion Positions

(0 0 0; $0 \frac{1}{2} \frac{1}{2}$; $\frac{1}{2} 0 \frac{1}{2}$; $\frac{1}{2} \frac{1}{2} 0$) +

16 R in (c) : (1/8, 1/8, 1/8; 1/8, 7/8, 7/8;)

16 Q in (d) : (5/8, 5/8, 5/8; 5/8, 3/8, 3/8;)

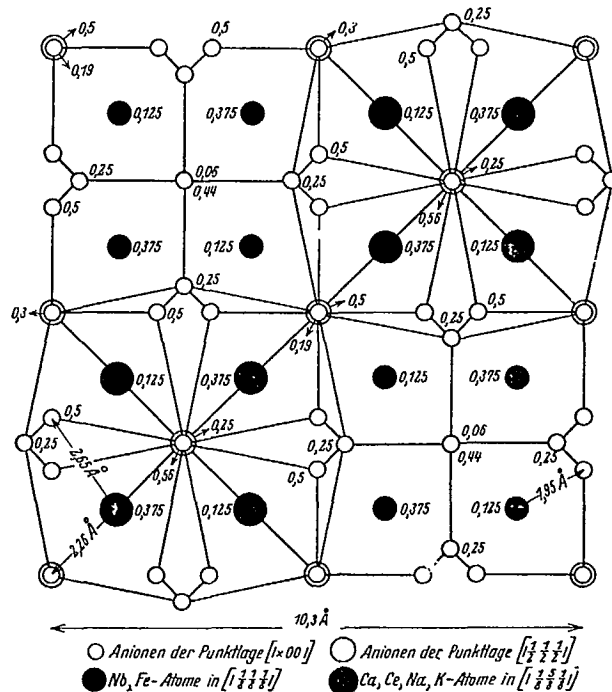
4 X I in (b) : ($\frac{1}{2} \frac{1}{2} \frac{1}{2}$; 3/4, 3/4, 3/4).

48 X II in (f) : (x 0 0; \bar{x} 0 0; $\frac{1}{4} + x, \frac{1}{4}, \frac{1}{4}$; $\frac{1}{4} - x, \frac{1}{4}, \frac{1}{4}$) x = 0.19

R to X I 2.26. R to X II 2.65. Q to X II 1.95. X I to X II 3.21.

X II to X II 2.64 and 2.76 Å. All of these distances are in Angstrom units.

R to R 3.666 Å.



Pyrochlore Ion Positions

Geological Survey of Canada, 1954, Paper 54-5.

NOTES ON GEOLOGY AND MINERALOGY OF
THE NEWMAN NIOBIUM-URANIUM PROSPECT

by

R.B. Rowe

The host rock (acmite-potash feldspar-uranium pyrochlore rock) is described as follows: "Acmite, feldspar, calcite, biotite, pyrite and uranium pyrochlore can be identified in hand specimens of this rock which is dark in colour, generally equi-granular and medium-grained. The acmite is dark green, forms subhedral to euhedral crystals up to 8 mm in length and commonly comprises 60 to 90% of the rock. Reddened feldspar occurs as aggregates of anhedral crystals and is present in amounts up to about 30%. Calcite occurs as veinlets and as anhedral crystals interstitial to acmite and feldspar and generally comprises less than 10% of the rock. A few crystals of brown biotite occur in places. Uranian pyrochlore is visible as disseminated crystals up to about 2.5 mm in maximum dimension. These crystals vary in lustre from vitreous to resinous to sub-metallic."

Average composition by volume

Mineral	Per cent
Acmite (including fibrous amphibole)	69.5
Feldspar (mostly potash feldspar)	15.0
Carbonate	5.9
Pyrite	2.7
Uranian pyrochlore	2.1
Opaque minerals (mostly various iron oxides)	1.6
Soda - hornblende	1.4
Chlorite	0.7
Sericite	0.7
Biotite	0.4
Monazite, apatite, fluorite (a few crystals)	

There would appear to be a grinding and liberation problem here.

Interim Mineral Dressing Test Report, No. 716
ML, March 16, 1955.

A MINERALOGICAL STUDY OF ORE FROM THE
NORTH ZONE OF THE NEMEGOS (ONTARIO)
PROPERTY OF MULTI-MINERALS LIMITED

by

E.H. Nickel

The most abundant minerals in the north zone material are: pyroxene, feldspar and magnetite. Minerals occurring in smaller amounts include ilmenite, pyrrhotite, pyrite, chalcopyrite, sphalerite, apatite, zircon, calcite, biotite, analcite and pyrochlore.

Ore dressing methods hold very little promise for the concentration of the valuable constituents of this material. Much of the pyrochlore is fine-grained and the grain borders are so irregular that a high degree of freedom can only be obtained by extremely fine grinding. The mechanical separation of ilmenite and magnetite is practically impossible because of their fine-grained intergrowth and even the clean separation of these minerals from the gangue can probably not be accomplished at coarse grinds because of the irregularity of the grain boundaries. This ore appears to have little to recommend it at present.

Investigation No. MD3112, January 26, 1956.A MINERALOGICAL STUDY OF PYROCHLORE ORE FROM
OREBODY NO. 8 OF THE NEMEGOS (ONTARIO) PROPERTY
OF MULTI-MINERALS LIMITED

by

E.H. Nickel

The minerals present in Oreboddy No. 8 are chiefly pyroxene, feldspar, nepheline and magnetite, with smaller amounts of ilmenite, pyrrhotite, apatite, zircon, calcite, biotite, analcite, pyrochlore and traces of other sulphides.

The ore mineral, pyrochlore, occurs chiefly as irregular aggregates in feldspar and nepheline. It is quite fine-grained and appreciable liberation does not take place above 200 mesh. The brittle nature of this mineral causes it to be crushed very readily concentrating it in the finest sizes. It is unlikely, therefore, that the pyrochlore of this deposit can be successfully concentrated by gravity methods.

Investigation No. MD3155, October 23, 1956.A MINERALOGICAL STUDY OF CARBONATE-TYPE
PYROCHLORE ORE FROM THE NEMEGOS (ONTARIO)
PROPERTY OF MULTI-MINERALS LIMITED

by

E.H. Nickel

This is a companion piece to the preceding report.

Pyrochlore is the only niobium-bearing mineral identified in the ore and occurs in disseminated crystals in the gangue minerals. The principal gangue minerals are calcite, apatite and pyroxene. The calcite is notable for its strontium content and the apatite for its strontium and rare earth contents. The majority of the gangue minerals are liberated at 35 mesh and

the pyrochlore at about 100 mesh.

Mines Branch Investigation Report 58-55, 1958.A MINERALOGICAL INVESTIGATION OF RARE EARTH
CARBONATE ORE IN DRILL CORE FROM THE OKA,
QUEBEC, PROPERTY OF QUEBEC COLUMBIUM LIMITED

by

E.H. Nickel

The diamond drill core described in this report contains from 1.5 to 4.5% rare earth oxides and appreciable amounts of manganese, barium and strontium. The rare earth minerals are ancylite, bastnaesite and monazite. They are characterized by an extremely fine grain size and are commonly intergrown with other minerals.

The chief mineral in the core is a coarse crystalline dolomite. Other minerals, including those present in small amounts, are strontianite, barite, siderite, calcite, apatite, pyrite, pyrrhotite, marcasite, sphalerite, galena, quartz, biotite, pyrochlore and a zeolite.

The Canadian Mineralogist, Vol. 6, 1957-1961,
p. 610-633.

A STUDY OF PYROCHLORE AND BETAFITE

by

D.D. Hogarth

Pyrochlore and betafite from several types of Canadian occurrences were investigated. Nine new analyses for twenty constituents and eleven analyses for eight constituents are presented. A general formula $A_{16-x}B_{16}(O,OH)_{48}(F,OH)_8$ is proposed for the pyrochlore-betafite series with x representing vacant sites in the unit cell. Differential thermal and thermogravimetric curves indicate two states of water.

Betafite and thorian pyrochlore are metamict, but recrystallization can begin well below the exothermic reaction indicated on the D.T.A. curves. The cell edge of ignited minerals tends to decrease as titanium and iron increase. Molecular weights derived from density-cell edge data correspond qualitatively to those calculated from analyses. Observed X-ray intensities agree with calculated values, but the role of iron is uncertain. Frequency diagrams suggest a natural division of pyrochlore and betafite at 15% uranium.

Mines Branch Investigation Report IR 61-146,
December 13, 1961.

A MINERALOGICAL INVESTIGATION OF NIOBIUM ORE
FROM COLUMBIUM MINING PRODUCTS LIMITED,
OKA, QUEBEC

by

E.H. Nickel

The ore from the Columbian Mining Products Limited property at Oka consists largely of calcite, pyroxene, biotite and apatite, with calcite predominating. The niobium-bearing minerals are pyrochlore and niobian perovskite. Pyrochlore is more abundant in the higher-grade samples while niobian perovskite predominates in the lower-grade samples. The Nb_2O_5 content of the pyrochlore ranges from 39.7% to 55.2% and that of the perovskite varies from 14.5% to 44.9%. A number of chemical relationships and physical properties of these minerals have been investigated with a view to relating niobium content to other determinable features. It was found that the niobium content of the pyrochlore is related inversely to its radioactivity and titanium content and that the niobium content of

the perovskite is related directly to the $Nb_2O_5:TiO_2$ ratio in the ore.

Other minerals found in the ore include dolomite, magnesite, siderite, strontianite, monticellite, nepheline, analcite, natrolite, garnet, magnetite, hematite, pyrite, pyrrhotite, chalcopyrite, feldspar, zircon, barite and fluorite.

Mines Branch Investigation Report IR 70-7, 1970.

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

by

E.H. Nickel and R.G. Pinard

A mineralogical investigation was made of samples of diamond drill core from a carbonatite deposit near St-Honoré, Quebec, which were submitted by one of the members 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.

Investigation No. MD3126, June 29, 1956.

A MINERALOGICAL EXAMINATION OF ORE FROM
BEAUCAGE MINES LIMITED, NORTH BAY, ONTARIO

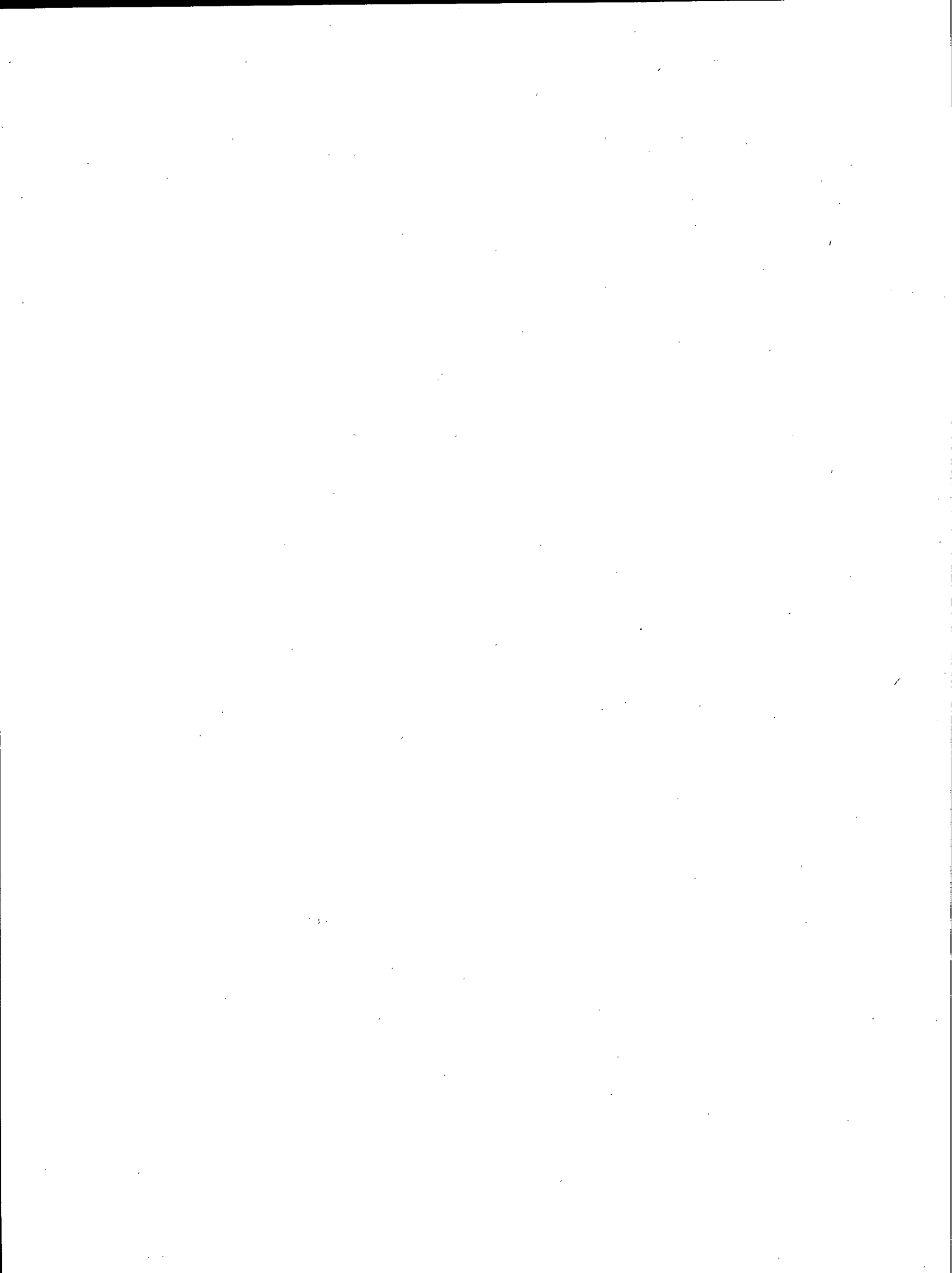
by

E.H. Nickel

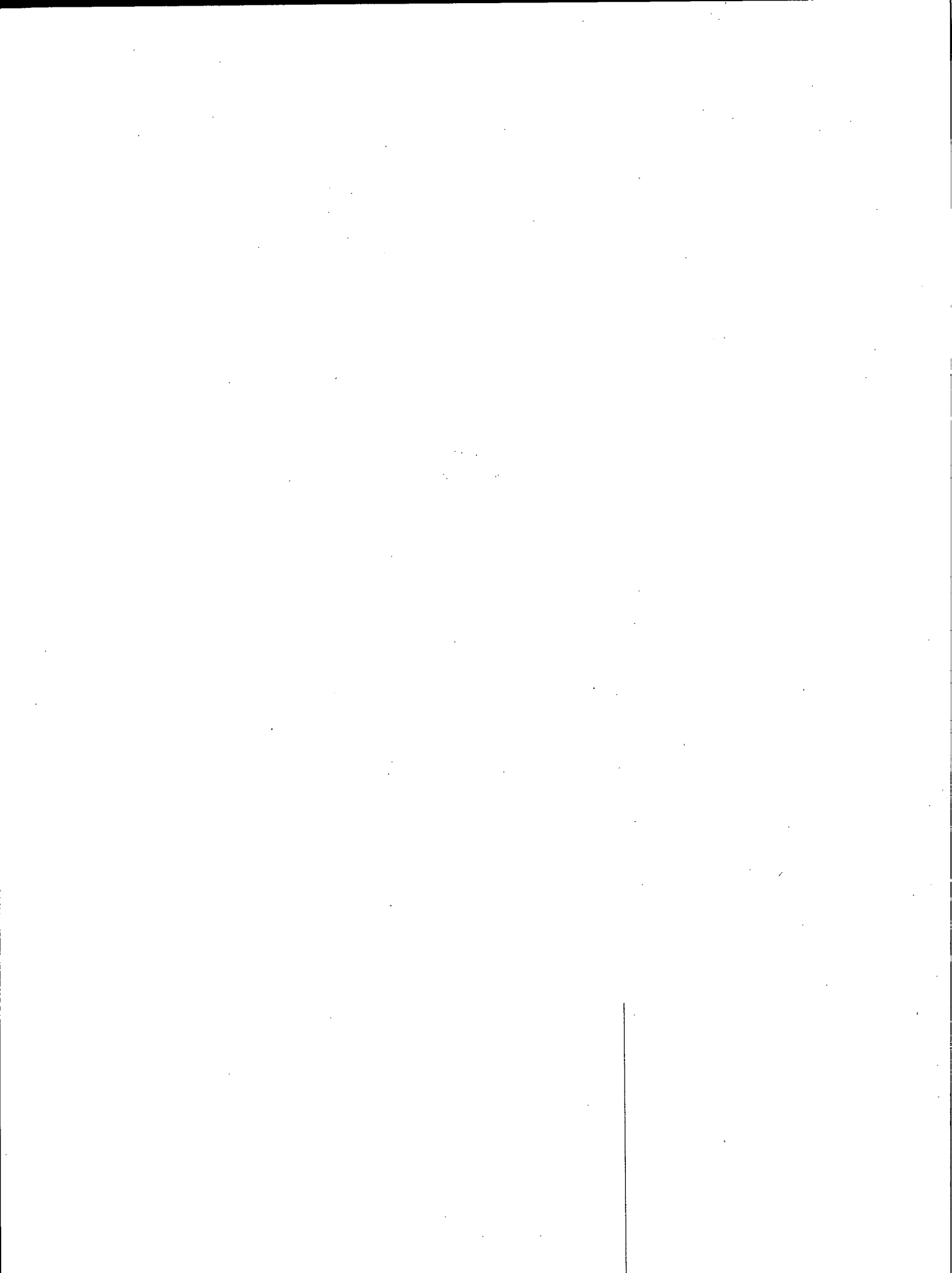
Pyroxene, biotite, feldspar and calcite are the most abundant constituents and total more than 90% of the minerals present. The ore mineral pyrochlore, making up about 1% of the ore, is the only niobium and uranium-bearing mineral identified. The estimated mineral abundances in the three samples investigated are shown in the following table:

Estimated abundance of minerals in
samples 3603 and 3604 and shipment C

Mineral	3603%	3604%	C%
Pyroxene	40	29	26
Biotite	21	18	17
Feldspar	15	37	14
Calcite	15	7	24
Apatite	2	2	7
Sulphides	3	3	5
Magnetite	1	1	4
Ilmenite	1	1	1
Pyrochlore	1	1	1
Other	1	1	1
	<u>100%</u>	<u>100%</u>	<u>100%</u>



APPENDIX B - PART 3
NIOBIUM PRODUCTION



Foreign ProductionNorway

"Columbium (Niobium) and tantalum" by R.J. Jones, Mineral Resources Division; EMR; Memorandum Series No. 135; 1957.

The first commercial production of pyrochlore concentrates from a primary source came from about 65 miles southwest of Oslo, near Ulefoss in Telemark county. A part of the Söve carbonatite area was mined, beginning in 1953. One of the higher grade sections has this composition:

Compound	%	Compound	%
CaO	43.5	CO ₂	36.4
MgO	1.2	Nb ₂ O ₅ + Ta ₂ O ₅	0.30
FeO	1.1	Fe ₃ O ₄	5.0
3(CaO.P ₂ O ₅)CaF ₂	8.5	FeS ₂	0.5
MnO	0.5	Silicates, etc.	3.0

The ore is crushed, ground and classified. The sized products are tabled, producing a pyrochlore-apatite concentrate and a tailing that is mostly carbonate. The pyrochlore-apatite concentrate is treated with nitric acid, producing 50% Nb₂O₅ concentrate and further treated with a nitric acid-phosphoric acid solution which produced a phosphate fertilizer.

In another approach it was proposed that the ore be treated by crushing, grinding to minus 6 mesh and calcining in a fluid-bed roaster (9). The calcine would be slaked first in air and then in water, producing hydroxide milk, which was to be processed further, as well as a low carbonate fraction, about 6.5 to 7.5% of the original weight of the feed. In test work, the calcine was tabled to remove mica and produce a heavy fraction which was given magnetic separation. The non-magnetic fraction was mostly apatite and niobium minerals. The heavy fraction was treated with nitric acid as in the other process. It was also proposed to treat the non-magnetic fraction electrostatically to improve working conditions and use less expensive sulphuric acid to produce

phosphate fertilizer. Because this process needed more expensive machinery it was never applied.

Brazil

Ministry of Mines and Energy, Bulletin (Boletim) No. 136, (Portuguese), 1970. "Pyrochlore of Barreiro, Araxa."

Attention was first drawn to the Barreiro carbonatite by indications of radioactivity. The government exploration agency, Prospec, contracted for aeroscintillometer, aeromagnetometer and aerial photography mapping. This gave the surface limits of the deposit. Diamond drilling to 110 m indicated continuation to depth. In 1959 DEMA (Distributors and Exporters of Minerals and Fertilizers) did surface and near-surface sampling by means of adits and pits. Concentration test work was begun at Barreiro. At this time, reserves, calculated to 50-m depth, were approximately 300,000,000 tons with a grade of 3% Nb₂O₅ (Paraiso, 1963). Drilling to 111 m was stopped in ore.

The ore is earthy, coloured light yellowish maroon. It appears to be similar to the capping at Panda Hill, Tanzania. In 1957 Guimares gave the mineral composition as:

Mineral	%	Mineral	%
Ilmenite	30 - 35	Apatite	Traces - 10
Limonite	30 - 35	Pyrochlore	2 - 6
Barite	1 - 11	Quartz	1 - 4

The deposit has a surface stratum, with a thickness of about 15 m in the flat parts thinning to nothing on the hills where it is considered as waste due to difficulty in concentration, in spite of it being no different chemically from the neighboring rock. This is probably the "hardpan", well known in Africa and to some extent in the southern United States.

Mining is by open cut without explosives. The ore is residual capping of the carbonatite where carbonates, silica and other minerals have been removed by millions of years

of oxidation and leaching. A bulldozer, a loader and two trucks are used. Mining began in 1961. In a section containing 5 to 6% Nb₂O₅, the flowsheet consists of:

- (1) crushing,
- (2) grinding in a ball mill to 150 mesh
- (3) magnetic separation,
- (4) cycloning,
- (5) flotation (including separation of TiO₂),
- (6) leaching with caustic soda to remove P and S,
- (7) drying.

The final product has an average grade of 58% Nb₂O₅ and maximum limit of 0.4% SiO₂, 0.04 P and 0.04% S with nearly 1.7% thorium. DEMA has exported this thorium and is obliged to return to the Energy Board an equivalent in U 235.

DEMA produces ferro-niobium in its own plant by thermal reduction. The official name of the project is: "Companhia Brasileira de Mineraçao e Metalurgia".

Production:

Year	Tons ferro-niobium	Tons of Nb ₂ O ₅ concentrates
1964		28
1965		1 304
1966	619	3 795
1967	420	7 200
		kg Nb ₂ O ₅ concentrates
1968		3 020 000
1969		6 257 000
		Tons pyrochlore concentrates
1971		1 640
1972		4 884
1973		6 445
1974		3 740
1975		2 895

The average analysis of the ore is:

Compound	%	Compound	%
Nb ₂ O ₅	3.50	Ce ₂ O ₃	1.62
P ₂ O ₅	3.33	La ₂ O ₃	2.45
TiO ₂	3.30	SO ₃	8.46
SiO ₂	2.86	MgO	0.43
ZrO ₂	0.14	CaO	0.33
ThO ₂	0.16	BaO	17.50
CO ₂	2.06	MnO	1.21
Al ₂ O ₃	1.19	U ₃ O ₈	<0.01
Fe ₂ O ₃	44.00	H ₂ O	6.07
FeO	1.00		

The average mineral content of ore is:

Pandaite	1 - 6%	Goyazite	1 - 6%
Magnetite	2 - 6%	Monazite	5 - 10%
Ilmenite	1 - 4%	Rutile-anatase	1 - 3%
Goethite	40 - 50%	Quartz-anatase	1 - 4%
Barite	14 - 30%	Zircon	0.5-1%

Flotation is thought to be through the use of Cyanamid Reagent 3037. Recovery is approximately 66%. The flotation concentrate is washed and then leached with hot hydrochloric acid. The mixture is filtered on a drum filter. The filter cake is mixed with 20% of its weight of caustic soda (in flocks) which is worked into the concentrate, the temperature rising to 60°C. In the roaster which follows, the temperature rises to 200°C, the concentrate is digested and phosphor and sulphur are removed. Also, sodium of the NaOH substitutes for barium to some extent in the pyrochlore, increasing the Nb₂O₅ grade. After double washing the concentrate is dried at 600°C.

Canadian ProductionEngineering and Mining Journal, October, 1961.ST. LAWRENCE COLUMBIUM PROJECT
STARTS PRODUCTION IN QUEBEC

by

Staff Reporters

Late in August 1961, production of pyrochlore concentrates began at the property of St. Lawrence Columbiu and Metals Limited at Oka, 32 km west of Montreal. Ore reserves were reported to be 62.6 million tons of 0.4% Nb₂O₅ to a depth of 500 feet. Of this, 17 million was in the block adjacent to the mill and being mined by open pit methods. The ore contained approximately 1% pyrochlore, 75-80% calcite, 6-10% diopside, 3-4% apatite, 3-4% magnetite, 3-4% mica, and 1-2% pyrite. As can be seen from the flowsheet, the process was roughly: crushing, grinding, screen sizing, tabling, magnetic separation, pyrite flotation, pyrochlore flotation, tabling, drying, and storage. Approximately 60% of the feed was removed by the tables as a calcite product which was stored in a tailings disposal area east of the mill. Pyrochlore flotation was by "an amine collector and a catalyzer" at pH 5 to 6.5. The last table was largely a visible control instrument. Water was supplied by wells. Analyses of pyrochlore and pyrochlore

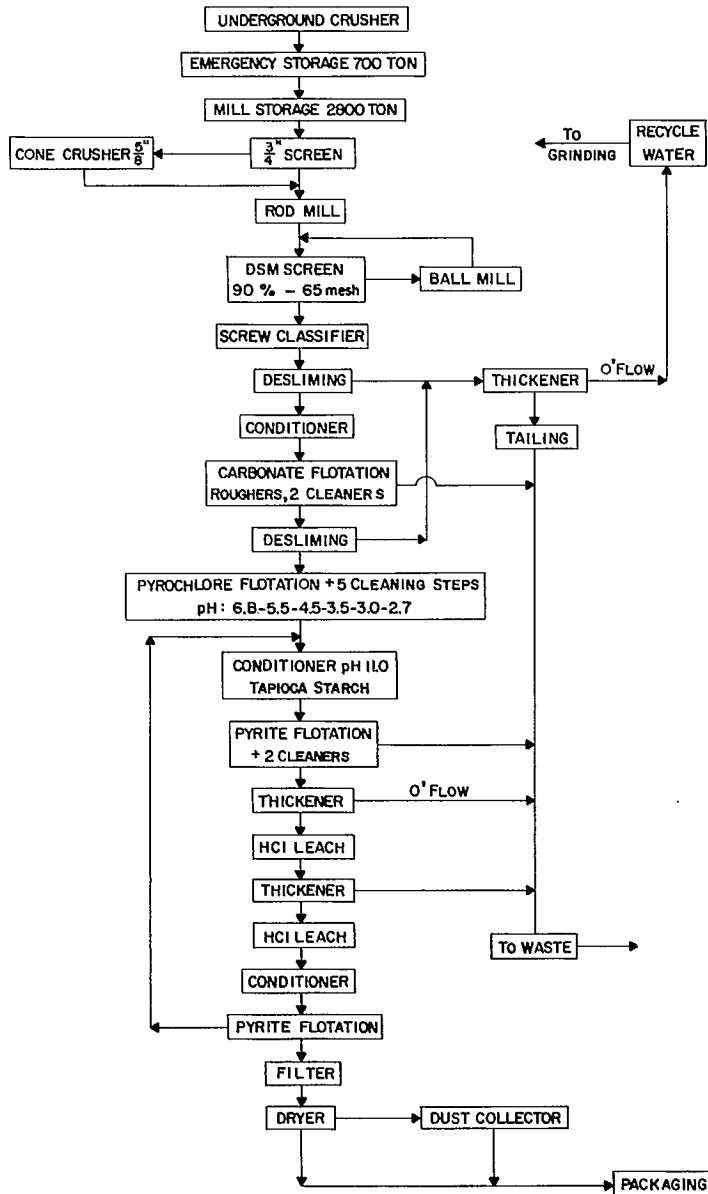
concentrates are given below.

Analysis	Pyrochlore	Pyrochlore concentrates %
Nb ₂ O ₅	60.0	52+
Ta ₂ O ₅	0.5	0.09 - 0.5
TiO ₂	4.5	4.0 - 6.5
Fe ₂ O ₃	2.70	2.7 - 5
Na ₂ O	3.50	3.3 - 3.70
K ₂ O	0.60	0.20 - 0.80
SrO	0.60	0.42 - 1.2
CaO	15.08	15.0 - 20.0
MgO	1.00	0.0 - 2.10
MnO ₂	0.37	0.07 - 0.74
ZrO ₂	1.0	0.80 - 1.3
U ₃ O ₈	0.10	0.05 - 0.20
ThO ₂	0.23	0.07 - 0.30
Y ₂ O ₃	0.12	not determined
Gd ₂ O ₃	0.2	0.0 - 0.2
CeO ₂	3.0	3.0 - 6.0
La ₂ O ₃	0.7	n.d.
SiO ₂	0.08	0.2 - 0.6
F	3.69	2.5 - 4.05
H ₂ O+	1.24	n.d. - 0.6
P ₂ O ₅	n.d.	0.1 - 0.6
S	n.d.	0.1 - 0.4
WO ₃	n.d.	Traces
SnO ₂	n.d.	Traces

September 5, 1961

The pH of the pulp is raised to 11 with NaOH and it is conditioned with 0.1 lb/t of tapioca starch to depress pyrochlore. Pyrite is floated and cleaned twice with potassium amyl xanthate; 75% of the pyrite is removed. The remaining cleaner pyrochlore concentrate contains about 25% by weight of pyrite. The pyrite flotation tailing is thickened to 55% solids and then leached with 4 lb/t hydrochloric acid. The small amount of apatite is soon dissolved. The slurry

is diluted in another thickener and the underflow is pumped to a second leaching stage where 0.5 lb/t of acid is added. The leached slurry at a pH of about 0.5 is conditioned with $CuSO_4$. The pH is adjusted to 10.5 with NaOH and xanthate is added to float the sulphides. The flotation tailing is filtered, dried and bagged. The Niobec flowsheet is given in figure below. The reagents used and metallurgical balance of the mill are recorded in the tables on page B-62.



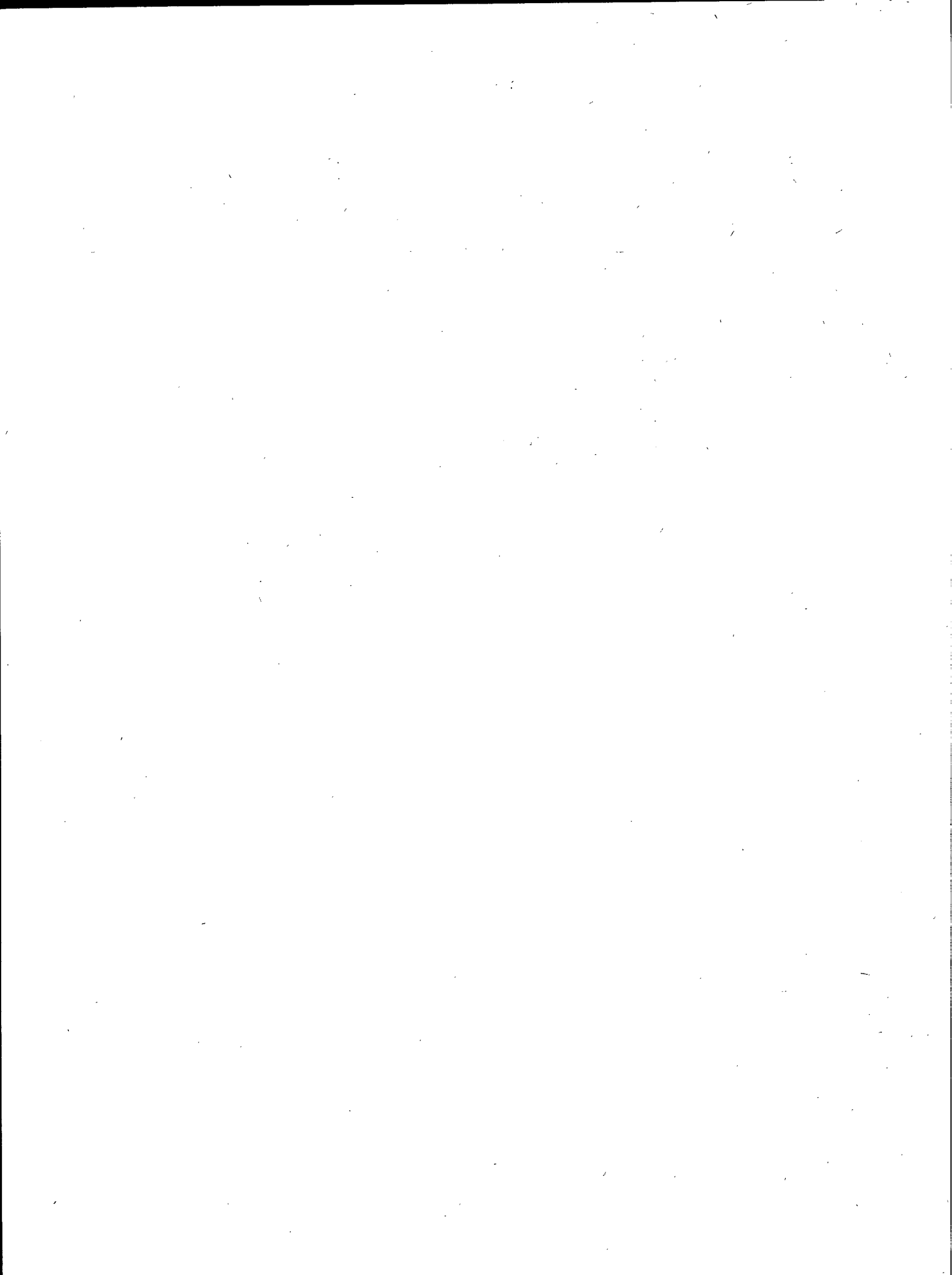
Reagents used

Reagents	Use	Consumption lb/-n	Solution strength, %	Arrival
Acintol FA-3	Calcite collector	0.54	100	Bulk
Lannagol	Emulsifier	0.17		
Na ₂ SiO ₃	Softener	0.79	10	Bulk
Duomac T	Pyrochlore collector	0.41	3.75	Drum
CES-265	Emulsifier	0.17	1.25	Drum
Oxalic acid	Depressant	1.02	5.0	Bulk
Fluorosilicic acid	Depressant	1.02	5.0	Bulk
Copper sulphate	Activator	0.06	1-2	Bags
K.A.X.	Sulphide collector	0.02	10	Drum
T. starch	Depressant	0.15	3	Bulk
NaOH	pH regulator	1.06	10	Bulk
HCl	Lixiviant	4.42	31	Bulk
DF-250	Frother	0.001	10	Drum
Percol 455	Flocculent	0.01	0.15	Bags

Metallurgical balance; Niobec mill

Product	Weight, tpd	Grade, % Nb ₂ O ₅	Distribution, %
Slimes	253	0.51	9.39
Carbonate concentrate	429	0.06	1.87
Rougher tailings	538	0.08	3.13
Cleaner tailings	716	0.28	14.38
Pyrite concentrate	11	1.24	0.99
Pyrochlore concentrate	15.5	62.41	70.24
Mill feed	1962.5	0.70	100.00

APPENDIX B - PART 4
NIOBIUM EXTRACTION TESTS



Mines Branch Investigation Report IR 58-84EXTRACTION OF NIOBIUM FROM A TABLE MIDDLING
PRODUCT OF QUEBEC COLUMBIUM LTD., OKA, QUEBEC

by

G. Thomas, T.R. Ingraham and W.J. Romanowicz

Sample tested: This was a table middling product.Mineralogy: Pyrochlore, perovskite, niocalite, apatite and pyroxenes were the main minerals.Analysis: The feed contained 4.8% Nb₂O₅.Purpose of the Investigation: It was proposed to extract the niobium minerals and when in solution the niobium would be precipitated and the precipitate calcined to pure Nb₂O₅.Summary of results: Preliminary leaching with nitric acid removed apatite. Leaching with HF or phosphoric acid gave 90% extraction at prohibitive costs. Recovery of niobium was too low with sulphuric acid due to the presence of considerable niocalite, a niobium silicate.Conclusions: Any of the above methods might be applied to concentrates containing pyrochlore and perovskite but not to those with an appreciable content of niocalite.Mines Branch Investigation Report IR 60-123THE PREPARATION OF NbCl₅ FROM ORE CONCENTRATES
OF COLUMBIUM MINING PRODUCTS LIMITED,
OKA, QUEBEC

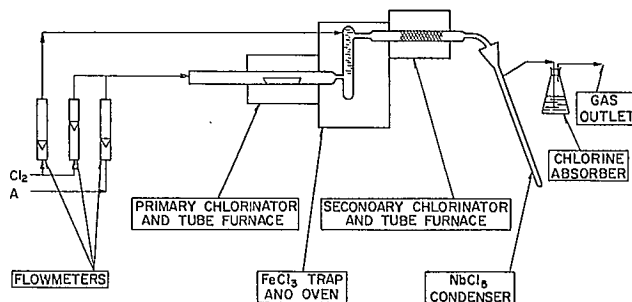
by

J.P. Whalley, T.R. Ingraham and P. Marier

Sample tested: The sample was a flotation concentrate, further upgraded by tabling.Mineralogy: Approximately 50% of the sample weight was pyrochlore and perovskite. No niocalite appeared to be present. The remainder was largely apatite, diopside and mica.Analysis: (See the following table).

Concentrate analysis

Compound	%	Compound	%
Rare earths			
Nb ₂ O ₅	24.7	and ThO ₂	5.1
CaO	17.6	ZrO ₂	1.1
SiO ₂	14.1	BaO	trace
Fe ₂ O ₃	12.3	SrO	0.26
MgO	7.5	Y ₂ O ₃	0.04
TiO ₂	6.3	V ₂ O ₅	0.05
Al ₂ O ₃	3.1		

Purpose of the investigation: To determine whether the concentrate was amenable to chlorination and whether the NbCl₅ would be suitable for the production of Nb metal.Method of extracting: Chlorination in the presence of carbon at between 600 and 700°C. Condensation of the evaporated NbCl₅ and its reduction by hydrogen. The arrangement of apparatus used is given below.

Apparatus used in chlorination experiments

Results: The concentrate was chlorinated without difficulty with an excellent yield. Practically all of the other elements were chlorinated too but their separation was not difficult. Condensed $NbCl_5$ was converted to Nb metal by hydrogen reduction.

Conclusions:

- (1) Concentrate prepared from Columbian Mining Products Limited ore was amenable to chlorination.
- (2) Reagent consumption and conditions for the production of niobium pentachloride were: 0.81 g of chlorine per gram of concentrate (4.6 g per gram of niobium), 1.0 g of carbon per gram of concentrate, at a temperature of between 600 and 700°C for 30 to 120 minutes for concentrate particles of 90% minus 200 mesh. (It is evident that with a concentrate approaching 50-52% Nb_2O_5 the consumption of chlorine would decrease.)
- (3) The niobium pentachloride was suitable for the production of niobium metal.

The analysis of impurities in the niobium produced is given below.

Metallic impurities detected in niobium

Sample No.	Elements %					
	Ta	Fe	Mg	Si	Ag	Cr
1	n.d.*	0.02	0.03	0.03	0.002	0.003
2	n.d.	0.02	0.006	0.006	trace	n.d.

*n.d. - not detected

Internal Report EMA 64-3

THE PRODUCTION OF HIGH PURITY NIOBIUM OXIDE
FROM PYROCHLORE-PEROVSKITE CONCENTRATE

by

F.J. Kelly and W.A. Gow

Sample tested: Pyrochlore-perovskite flotation concentrate.

Mineralogy: As in previous report IR 60-123.

Analysis

Chemical analysis of flotation concentrate samples								
Sample No.	Nb_2O_5 (%)	Ta_2O_5 (%)	TiO_2 (%)	CaO (%)	Fe (%)	P_2O_5 (%)	SiO_2 (%)	ZrO_2 (%)
1	26.5	0.89	7.41	17.46	--			
2	48.9	0.28	5.33	19.0	5.47	0.15	0.4	1.33

Purpose of the investigation: To explore the possibility of producing high grade Nb_2O_5 for metal production or other uses.

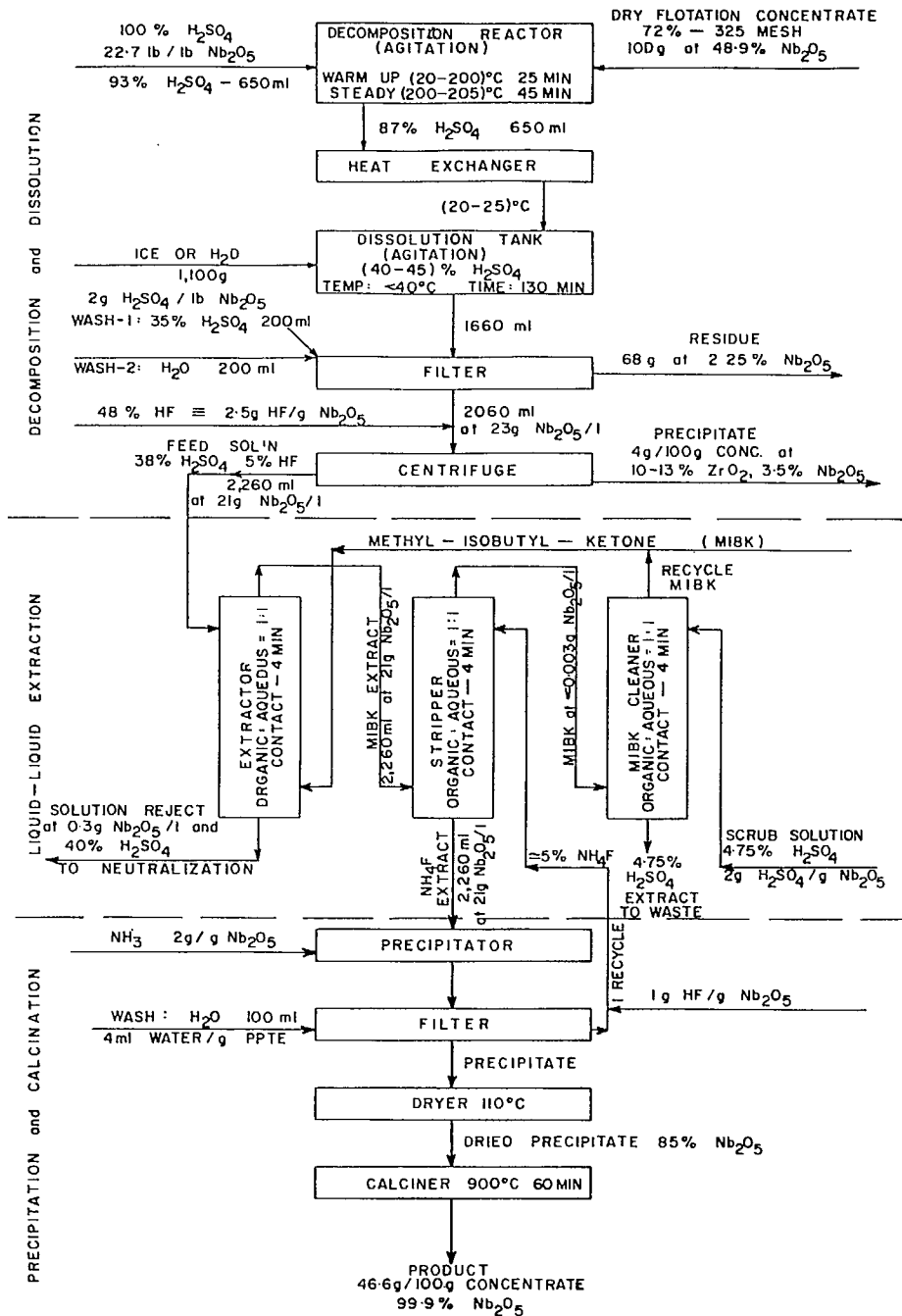
Method of treatment: Solution in hot concentrated sulphuric acid to a paste. Solution of the paste in 40% H_2SO_4 by adding ice followed by filtration and washing with acid. The solution was acidified further with 5% HF and was then shaken with methyl isobutyl ketone. The two solutions were separated and the organic phase was shaken with ammonium fluoride solution in water. Separation of the two liquids removed the niobium to the precipitation step by ammonia after filtration. The precipitate was filtered, washed, dried and calcined. The analysis of the final niobium pentoxide is given below.

Semi-quantitative spectrographic analysis
of the calcined precipitate

	%
Nb_2O_5	99.9 (chemical analysis)
Si	0.005
Mg	0.05
Fe	0.01
Al	0.01
Ti	trace
Ta	trace
Mn	0.003
Ca	0.03

The laboratory procedure applied is presented in the flowsheet on page B-67.

Results and conclusions: At least 95% of the niobium content of No. 2 sample was recovered as a precipitate analyzing 99.9% Nb_2O_5 .



Schematic presentation of the laboratory procedure used to produce Nb_2O_5 from flotation concentrate

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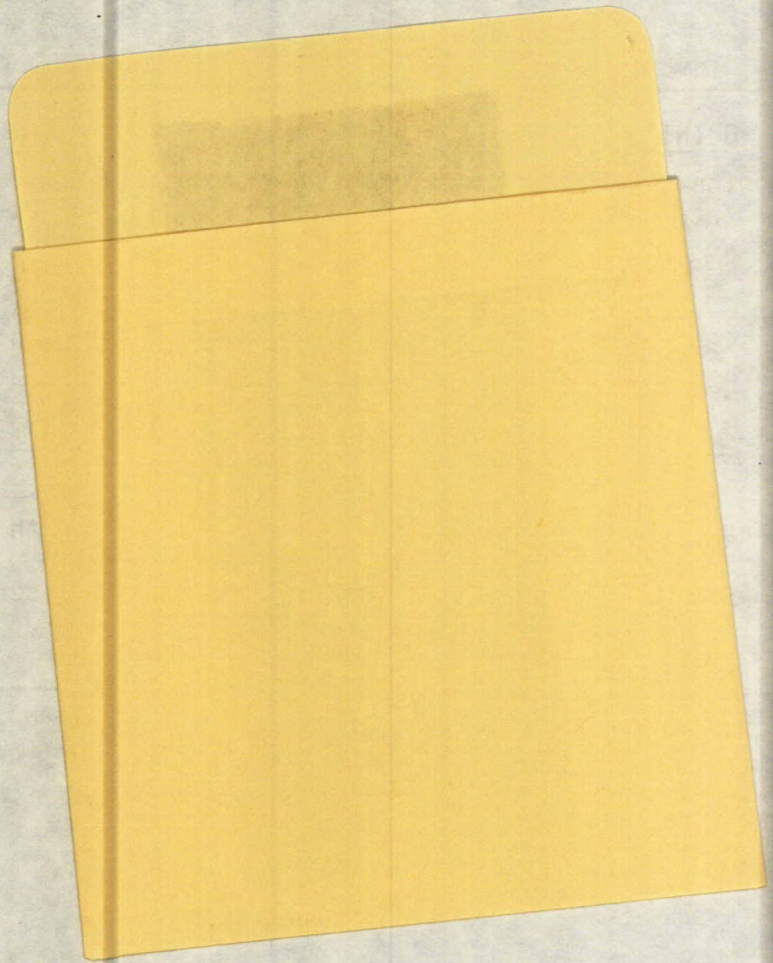
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