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

by

F. J. KELLY & W. A. GOW

EXTRACTION METALLURGY DIVISION

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

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F. J. Kelly* and W. A. Gow**

SUMMARY

This paper discusses the laboratory investigations made at the Mines Branch in Ottawa to determine the feasibility of producing by hydrometallurgical means, high purity niobium oxide from a flotation concentrate containing the niobium-bearing minerals, pyrochlore and perovskite. The feed material used for this work was flotation concentrate made from ore from the Oka, Quebec property of Columbian Mining Products Limited.

The test work indicated that the most promising route from mineral to purified oxide was decomposition of the flotation concentrate and dissolution of the niobium in concentrated sulphuric acid; purification and concentration of the dissolved niobium by a liquid-liquid extraction step using methyl isobutyl ketone as the extractant; recovery of the niobium from the organic extractant with ammonium fluoride solution from which the niobium oxide was precipitated with ammonia; and calcination of the washed precipitate. By following this procedure, laboratory testing resulted in over 95 per cent recovery of the niobium in a precipitate analysing 99.9 per cent Nb_2O_5 . The indicated reagent cost on the basis of the laboratory study was in the order of \$1.00 to \$1.25 per pound of niobium recovered.

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INTRODUCTION

Over the past few years Columbian Mining Products Limited has been engaged in the development of a niobium-bearing mineral deposit in the vicinity of Oka, Quebec. One phase of this program resulted in a flotation process capable of producing concentrates containing up to 50 per cent Nb_2O_5 . Flotation pilot plant investigations under the direction of the Company's consultant, Mr. H. L. Noblitt, showed that it was not possible to produce concentrates analysing more than 50 per cent Nb_2O_5 without experiencing a prohibitive drop in recovery. In comparison, niobium-bearing flotation concentrates containing at least 60 per cent Nb_2O_5 are currently available from other world sources. In view of this situation, the Mines Branch was asked by Mr. Noblitt to undertake an investigation directed towards the production by hydrometallurgical means of a high purity niobium salt from the Oka flotation concentrates. If the investigation showed an operation of this type was economically feasible, the competitive world position of the Oka producers might be improved.

As an aid in the planning of the requested investigation, the Company supplied the Mines Branch with the results of some exploratory work done by Battelle Memorial Institute in 1958 on this same problem (1). The Battelle work, done on a low grade flotation concentrate analysing 6 per cent Nb_2O_5 , showed that the niobium was leached with 96 per cent sulphuric acid at a temperature of 200-225°C. The leach liquor was diluted with water then boiled to precipitate the niobium by hydrolysis. The precipitate after calcination analysed 92 per cent Nb_2O_5 . The process suggested by the Battelle work was considered to be unsatisfactory because of the impractically high dilution required to effect hydrolysis and precipitation of the niobium, and because the final precipitate was not sufficiently pure. In addition, the results were inconclusive since the feed material was a low grade concentrate which at 6 per cent Nb_2O_5 would not be used in practice. However, the Battelle work did establish that concentrated sulphuric acid was effective in decomposing and leaching the pyrochlore and perovskite minerals.

A method for overcoming the two objections to the Battelle process was suggested by recent work done by the United States Bureau of Mines on the separation and recovery by liquid-liquid extraction of niobium and tantalum from sulphuric acid solutions (2). In this work methyl isobutyl ketone was used to produce high purity niobium and tantalum salts from a hydrofluoric acid-sulphuric acid solution of the two metals.

On the basis of the Battelle and U. S. Bureau of Mines studies, a test program was devised to determine the most practical conditions for sulphuric acid decomposition and dissolution of the niobium-bearing concentrate and to determine if methyl isobutyl ketone could be used for recovery of the dissolved niobium as a high purity salt.

Two samples of flotation concentrate were supplied for this work. Mineralogical examination showed the only niobium-bearing minerals present in these concentrates were pyrochlore and perovskite in a proportion of about three parts pyrochlore to one part perovskite. The chemical analyses of these concentrates are shown in Table 1.

TABLE 1

Chemical Analysis of Flotation Concentrate Samples

Sample No.	Nb ₂ O ₅ (%)	Ta ₂ O ₅ (%)	TiO ₂ (%)	CaO (%)	Fe (%)	P ₂ O ₅ (%)	SiO ₂ (%)	ZrO ₂ (%)
1	26.5	0.89	7.41	17.46	--	--	1.75	--
2	48.9	0.28	5.33	19.0	5.47	0.15	0.4	1.33

DECOMPOSITION AND DISSOLUTION

Procedure

The general procedure used to effect dissolution of the niobium minerals consisted of two operations which, in this paper, are referred to as the decomposition and dissolution steps. In the decomposition step the flotation concentrate was contacted with strong, hot, sulphuric acid. After decomposition, the decomposed product was taken up in a more dilute sulphuric acid solution at temperatures up to 40°C; a procedure referred to as the dissolution step. In most of the tests the dry, ground concentrate was mixed with 93 per cent sulphuric acid in the decomposition step. However, in this paper, weight proportions of sulphuric acid:niobium oxide or sulphuric acid:concentrate are expressed in terms of 100 per cent H₂SO₄.

In the decomposition tests in which the acid:concentrate weight ratio was 3:1 or less, the mixture was heated in porcelain crucibles in a muffle furnace. In these tests, prior to heating, the mixture had the consistency of paste while the decomposed product, after heating, was dry or only slightly moist. In the tests in which the acid:concentrate weight ratio was 4:1 or greater, the mixture, being a fluid slurry, was heated in a glass reaction vessel and stirred continuously during the heating period.

The two samples, being flotation concentrates, were about 70 per cent minus 325 mesh. Consequently, the samples were not subjected to any additional grinding prior to decomposition.

The procedure employed in the dissolution step depended on the procedure followed in the decomposition step. Where the concentrate had been decomposed in the muffle furnace with low acid:concentrate weight ratios, the dry or almost dry decomposed product was taken up in 65 per cent sulphuric acid solution. Where the concentrate had been decomposed in the glass reaction vessel at high acid:concentrate weight ratios, the slurry, on completion of the heating period, was cooled to room temperature and gradually diluted with water or melting ice until the acid concentration of the solution was reduced to 40 to 45 per cent H_2SO_4 . The slurry was stirred continuously during the cooling and dilution steps and for a period following dilution during which the niobium was taken up in solution.

After the dissolution step, the slurries were filtered and the filter cakes washed twice; once with 35 per cent H_2SO_4 solution and then with water. The primary and wash filtrates were combined for the liquid-liquid extraction test work.

Results

In this work two samples of concentrate designated as Samples No. 1 and No. 2, containing 26.5 and 48.9 per cent Nb_2O_5 respectively were used as feed. The lower grade Sample No. 1 was used at the start of the investigation since it was the only one available at that time. Although it was realized that Sample No. 1 was of lower grade than anticipated from the flotation operation, it was considered that the results obtained from work on it would be indicative of the conditions applicable to the higher grade concentrate when it became available. To provide a better basis for comparison of the acid requirements of the two samples studied, the following discussion refers to acid:niobium oxide rather than acid:concentrate weight ratios.

In the first series of tests, mixtures of 93 per cent H_2SO_4 and concentrate, in proportions ranging from 3.8 to 11.3 weight parts of acid per part of Nb_2O_5 , were heated in a muffle furnace for periods of from 0.5 to 3 hours at a temperature of $215^\circ C$ to effect decomposition of the niobium minerals. The heating period was measured from the time the mixture was placed in the furnace which was pre-heated to the required temperature. On completion of the decomposition step, the decomposed material was taken up in 65 per cent sulphuric acid solution to form a slurry, containing 15 ml of acid solution per gram of original concentrate, which was stirred for a 3 hour

period. The slurry was then filtered and the residue, after washing, analysed for Nb_2O_5 .

The results of some of these tests are given in Figure 1, and show that the Nb_2O_5 extraction increased from 50 per cent to 87 per cent as the weight ratio of $\text{H}_2\text{SO}_4:\text{Nb}_2\text{O}_5$ increased from 3:8 to 11.3:1. The data shown in Figure 1 were obtained from the tests done using a heating period of 0.5 hour. The results of tests done at heating periods of over 0.5 hour showed that prolonged heating was detrimental to dissolution of the niobium.

Since the graph of Figure 1 was rising, another series of tests was done using $\text{H}_2\text{SO}_4:\text{Nb}_2\text{O}_5$ weight ratios ranging from 15.1 to 26.4:1. In these tests, due to the fluidity of the acid-concentrate mixture, the heating was done in a glass reaction vessel for periods ranging from 40 min to 90 min at a temperature of 200-205°C. During the heating period the slurry was mechanically stirred. Since it required about 25 minutes to bring the slurry up to the decomposition temperature, the slurry was at the maximum temperature for periods of from 15 to 60 min. On completion of the decomposition step the slurry was allowed to cool to room temperature and then diluted with a volume of water such that the acid concentration of the solution was reduced to 35 per cent H_2SO_4 . At this point the liquid-solid composition of the slurry was in the proportion of 9 ml solution per gram of original concentrate. Agitation was continued for 3 hr after which the slurry was filtered and the residue washed. The results of the tests done using a heating period of 40 minutes (15 minutes at 200-207°C) are given in Figure 2. This graph shows that the maximum extraction obtained was 92 per cent at an $\text{H}_2\text{SO}_4:\text{Nb}_2\text{O}_5$ weight proportion of 22.6:1. The results of tests done using longer retention times at the decomposition temperature (205°C) again showed that prolonged heating reduced the extraction efficiency. A mineralogical study of the residue from the test in which 92 per cent extraction was obtained showed the presence of undecomposed grains of perovskite and little evidence of niobium re-precipitation from solution.

At this point in the program, Sample No. 2, containing 48.9 per cent Nb_2O_5 , became available, and the investigation was continued using this more representative concentrate sample. The first series of tests using the high grade sample was done to determine the minimum $\text{H}_2\text{SO}_4:\text{Nb}_2\text{O}_5$ weight proportion necessary for efficient decomposition and dissolution. The procedures used were similar to those used in Sample No. 1, except that in the dissolution step the dissolution solution was diluted to 45 per cent H_2SO_4 with melting ice instead of to 35 per cent H_2SO_4 with water as was used in Sample No. 1. The results showed that at an $\text{H}_2\text{SO}_4:\text{Nb}_2\text{O}_5$ ratio of 22.7:1 and using a total heating time of 40 minutes, the extraction levelled off at about 92 per cent. This acid requirement is comparable to that found necessary for the decomposition of Sample No. 1.

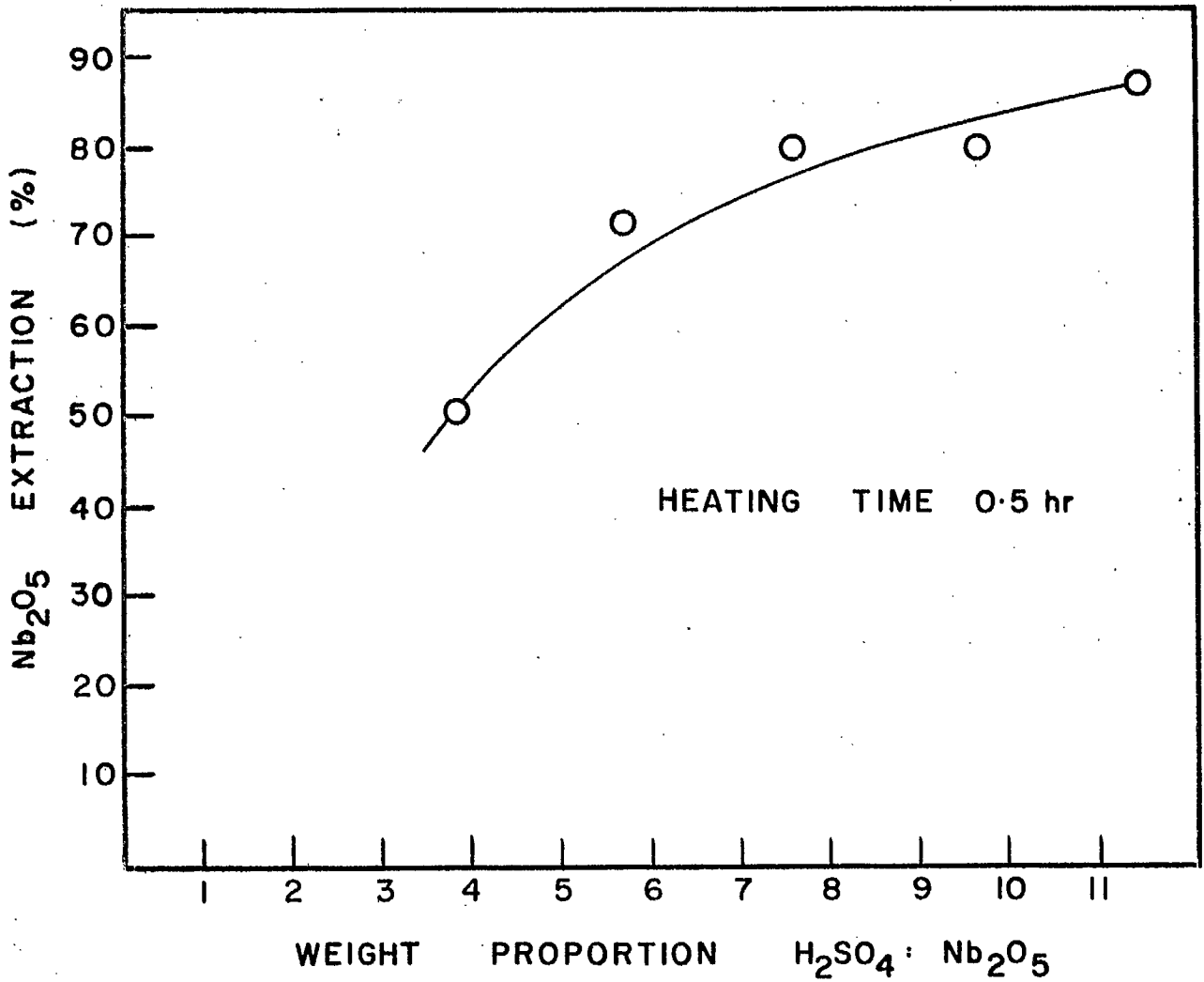


FIGURE 1 EFFECT ON SAMPLE I OF LOW ACID:
 Nb_2O_5 WEIGHT PROPORTIONS IN
DECOMPOSITION

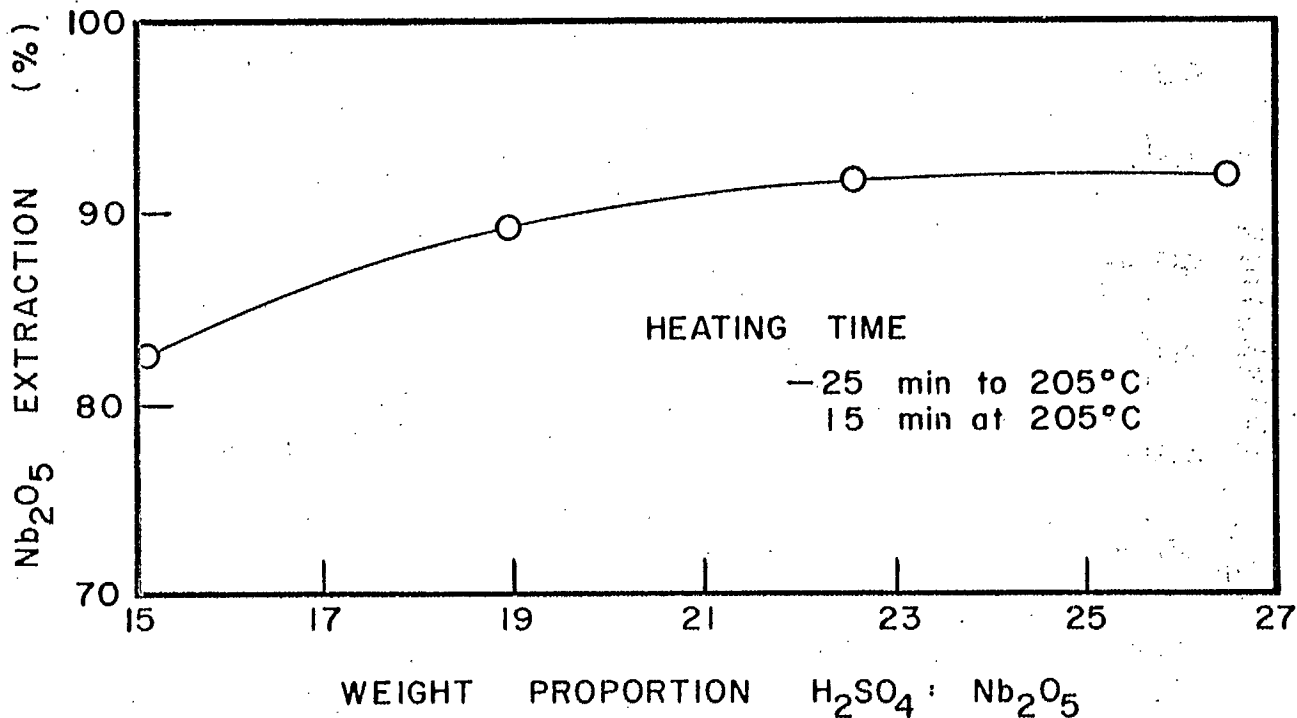


FIGURE 2 EFFECT ON SAMPLE 1 OF HIGH ACID : Nb₂O₅ WEIGHT PROPORTIONS IN DECOMPOSITION

The effect of heating time on the extraction of niobium for Sample No. 2 was investigated. The results of this work are shown in Figure 3. In these tests both the extraction of niobium and the degree of decomposition of the niobium mineral were determined; the latter by mineralogical study. Figure 3 shows that with decomposition periods of up to 45 minutes (70 minutes including warm-up), both per cent extraction and per cent decomposition increased with the former lagging 3-4 per cent behind the latter. However, when the 45 minute period was exceeded, the extraction showed a sharp decrease although decomposition continued to increase. From this it is evident that prolonged heating had an undesirable influence on the product of the decomposition step by resulting in a less soluble niobium compound; although decomposition of the niobium minerals was almost complete.

In all the test work described commercial grade, 93 per cent H_2SO_4 had been used for decomposition of the minerals. To determine the importance of high acid concentration in the decomposition step, tests were done in which the starting concentration of the acid used for decomposition ranged from 80 to 93 per cent H_2SO_4 . The results showed that with a starting concentration of 85 per cent H_2SO_4 , the extraction dropped 20 per cent from the extraction obtained when the starting acid concentration was 93 per cent H_2SO_4 . When the acid used for decomposition was 80 per cent H_2SO_4 , the extraction dropped an additional 10 to 15 per cent. These results indicate that very high acid concentrations are required in the decomposition step if extraction of niobium is to be satisfactory. In connection with this, it was observed that the acid concentrations of the solutions at the end of the decomposition of Sample No. 1 were 83 per cent H_2SO_4 compared to 87 per cent H_2SO_4 concentration in the solutions after the decomposition of Sample No. 2. This difference in acid concentrations during decomposition may explain why niobium extractions were higher from the higher grade concentrate, and why undecomposed perovskite was found in the residues from Sample 1.

The importance and operating characteristics of the dissolution step were indicated by the test work. The results showed that on completion of the decomposition step only two-thirds of the niobium was in solution, and that a 2 to 3 hour retention time in the dissolution step was required to dissolve the remaining one-third. The dissolution step was shown to be effective in allowing time for the precipitation of calcium sulphate which had been taken into solution during decomposition. Tests also showed that extension of the dissolution retention time beyond 3 hours was of no advantage. The most critical operation in the dissolution step was control of the pulp temperature while diluting the pulp after decomposition. If the dilution was done too quickly, the pulp temperature rose rapidly, and there was visible evidence of niobium re-precipitation if the temperature rose much above $40^\circ C$. The use of melting ice as the diluent in this step provided an effective means of temperature control in these small-scale tests.

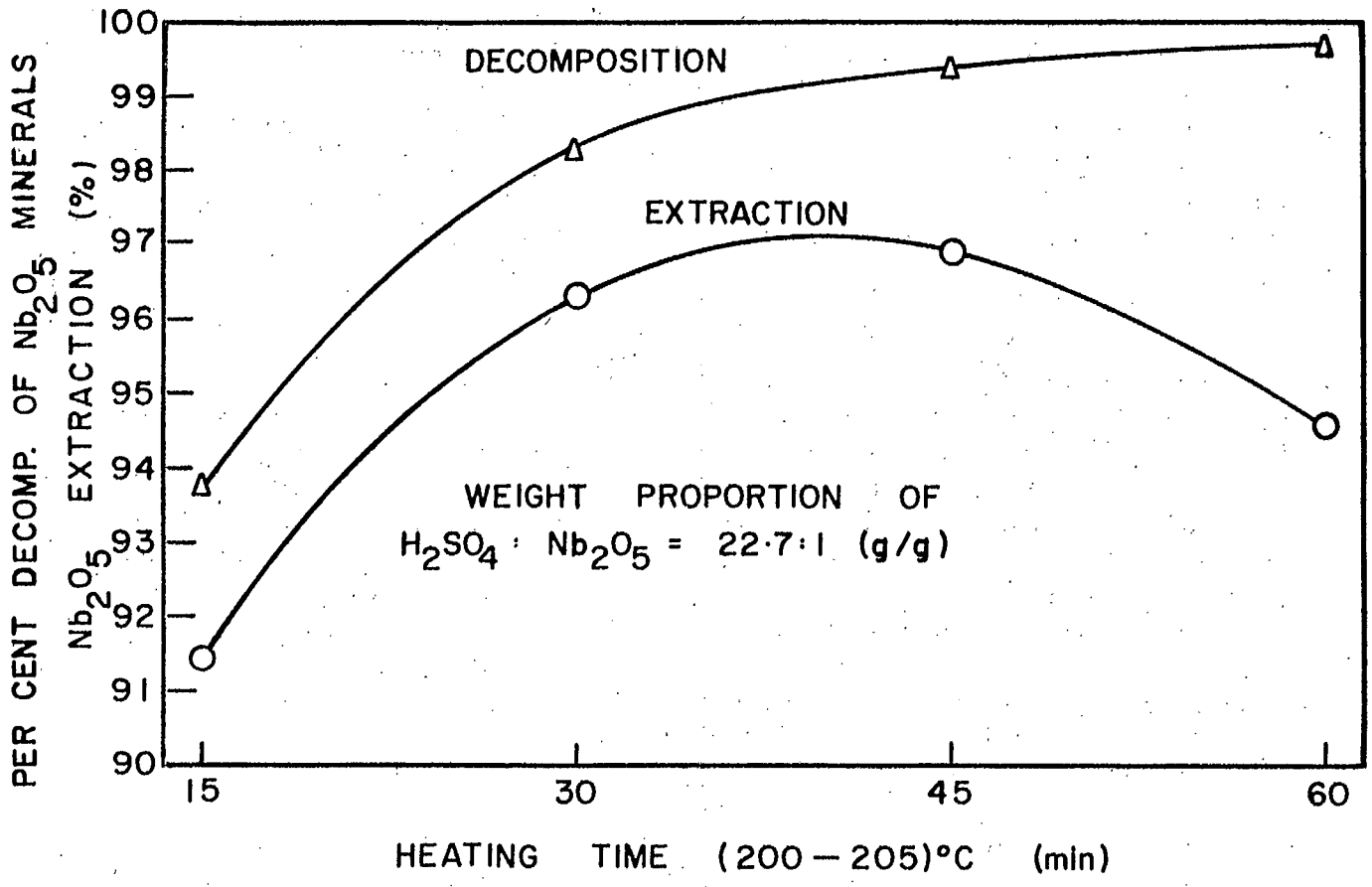


FIGURE 3 Nb₂O₅ EXTRACTION AND PER CENT DECOMPOSITION OF Nb₂O₅ MINERALS vs DECOMPOSITION TIME SAMPLE - 2

After completion of the dilution step, the niobium-bearing solution was recovered by filtration. The filter cake was washed with 35 per cent sulphuric acid using about 3 ml wash solution per gram of dry filter cake. Finally the cake was washed with a similar volume of water. It was observed that in the initial pulp filtration and in the subsequent washing operations filtration was rapid.

Table 2 is a summary in tabulated form of the best procedure for recovering the niobium in solution from Sample No. 2. The Nb_2O_5 distribution obtained by using these conditions is also given.

LIQUID-LIQUID EXTRACTION AND PRECIPITATION

Procedure

The general procedure followed in the solvent extraction and precipitation operations was composed of five steps. First, hydrofluoric acid was added to the combined niobium-bearing dissolution filtrate and wash solutions to a concentration of 5 per cent HF. After addition of the hydrofluoric acid, equal volumes of the acid solution and methyl isobutyl ketone (MIBK) were shaken together. On standing for a short period, the aqueous and organic phases separated clearly and the niobium-barren aqueous phase could then be drawn off from the bottom of the funnel. The niobium was stripped from the organic phase by shaking the MIBK with an equal volume of 5 per cent aqueous ammonium fluoride solution. After allowing the mixture to settle, the NH_4F solution, now containing the niobium, was drawn from the bottom of the funnel and treated to precipitate the niobium. In most tests, the now barren MIBK from the stripping operation was contacted with an equal volume of 4.75 per cent H_2SO_4 solution and recycled to the subsequent extraction step. The $\text{Nb}_2\text{O}_5\text{-NH}_4\text{F}$ solution from stripping was neutralized with ammonia to precipitate the purified Nb_2O_5 which was then filtered, washed, dried and calcined. The barren solution from precipitation was re-acidified with hydrofluoric acid and recycled to subsequent stripping operations. In this work polyethylene equipment was used throughout.

Results

The feed solution to the MIBK extraction step analysed about 23g Nb_2O_5 /l and 40 per cent H_2SO_4 . The principal impurities and their approximate concentrations were Fe - 4 g/l, Ca - 0.2 g/l, TiO_2 - 0.05 g/l, P_2O_5 - 0.5 g/l, ZrO_2 - 0.5 g/l, F - 0.8 g/l, Ta - 0.03 g/l and SiO_2 - 0.05 g/l. ⁵ When hydrofluoric acid was added to this solution to a concentration of 5 per cent HF, a white precipitate formed. About 2g of precipitate was produced per litre of solution. On analysis the precipitate

TABLE 2

(a) Conditions for Decomposition and Dissolution Steps
in Treatment of Sample No. 2

Decomposition

Concentrate Nb ₂ O ₅ analysis	48.9%
Grind	70% minus 325 mesh
Retention time	45 min
Temperature	200-205°C
100% H ₂ SO ₄ req'd	22.7 lb/lb Nb ₂ O ₅ in conc
93% H ₂ SO ₄ req'd	650 ml/100g conc
Final H ₂ SO ₄ concn	87%

Dissolution

Sulphuric acid concn	45%
Ice added for dilution	1100 g/100g conc
Retention time	3 hr
Temperature	< 40°C

Filtration and Washing

Residue, dry weight	68% of conc wt
Acid wash, H ₂ SO ₄ concn	35%
Acid wash, volume	200 ml/100g of conc
Water wash, volume	200 ml/100g of conc
Combined filtrate, volume	2060 ml/100g of conc
Combined filtrate, H ₂ SO ₄ concn	40%

(b) Nb₂O₅ Distribution in Decomposition and Dissolution Products

Sample No. 2

	<u>Nb₂O₅ Analysis</u> <u>(g/l or %)</u>	<u>Distribution</u> <u>(%)</u>
Dissolution filtrate	28.5)	92.3)
Acid wash filtrate	9.2)	4.0)
Water wash filtrate	0.8)	0.4)
Residue	2.25	3.2
Flotation conc	48.9	100.0
	23.0	96.7

was found to contain about 13 per cent ZrO_2 , 0.1 per cent HfO_2 and 3 per cent Nb_2O_5 . This represented less than 0.5 per cent of the niobium in the feed solution. The precipitate, being difficult to filter, was recovered by centrifuging, and the clarified solution extracted with MIBK. Adjustment of the feed solution to 5 per cent HF required 2.5 lb HF/lb Nb_2O_5 .

The test work done on the MIBK extraction step covered MIBK:aqueous volume ratios of 1:1 and 2:1, contact times of 2 and 4 minutes, recycling of the MIBK after stripping and whether two contact stages were necessary. The results showed that an MIBK:aqueous volume ratio of 1:1 was sufficient to recover 99.95 per cent of the niobium with a high degree of selectivity from an aqueous solution analysing about 21 g/l Nb_2O_5 . About half of the tantalum present in the feed solution was extracted with the niobium. These results were obtained with a 4 minute contact time and were slightly better than those obtained with a 2 minute contact time. The tests to investigate the effect on extraction of recycling the MIBK showed that the extraction of niobium levelled off at 99 per cent by the fifth cycle. Organic solvent losses are difficult to estimate from small scale tests since the measured loss is usually much higher than that experienced in larger scale continuous operations. The measured loss in these tests was in the order of 0.5 lb MIBK per lb Nb_2O_5 .

The test work done to investigate the removal of niobium from the MIBK with 5 per cent NH_4F solution showed that with an organic:aqueous volume proportion of 1:1, 99.9 per cent of the niobium was transferred to the aqueous phase in 4 minutes. The ammonium fluoride solution for this step was prepared by acidifying the ammonium hydroxide barren from the precipitation operation in the previous cycle with hydrofluoric acid to a pH of about 3.5. The amount of HF required for this was 1.0 lb per lb Nb_2O_5 produced.

After the niobium was removed from the organic phase, the MIBK was scrubbed with a 4.75 per cent H_2SO_4 aqueous solution using volume proportions of MIBK:aqueous of 1:1. This procedure is necessary to remove from the MIBK ammonium salts which would interfere with the extraction operation when the MIBK was recycled. This step also removed tantalum, present in a concentration of 0.02 g/l. The sulphuric acid used in preparing the scrub solution amounted to 2 lb H_2SO_4 per lb Nb_2O_5 . The scrubbed MIBK was reused for subsequent extraction of feed solution.

After it had been used to recover the niobium from the MIBK, the ammonium fluoride solution had a pH value of about 3.5. When anhydrous ammonia was added to raise the pH to 8.0, 99.9 per cent of the contained niobium precipitated. About 2 lb NH_3 per lb Nb_2O_5 recovered was required for this pH adjustment. The precipitate was recovered by vacuum filtration using a Buchner funnel. It was observed that the precipitate filtered rapidly. The barren filtrate was acidified to pH 3.5 with hydrofluoric acid and reused

to recover the niobium from the MIBK extract from the subsequent test. The filtered precipitate was washed with a volume of water equivalent to about 4 ml water per gram of precipitate and then dried at 100°C.

After being dried the precipitate analysed 85 per cent Nb_2O_5 . Spectrographic analysis of the precipitate showed that it contained about 0.10 per cent impurities in the form of metals and silica. In addition, it was apparent that the fluorine and ammonia consumed in the precipitation operations were contributing to the impurity of the precipitate. On the presumption that these latter impurities could be driven off with heat, the precipitate was calcined at 900°C for one hour. After calcination, the precipitate analysed 99.9 per cent Nb_2O_5 .

Table 3 shows the distribution of niobium throughout the entire solution treatment operation. The overall recovery in the precipitate of the niobium present in the original feed solution is shown to be 98.5 per cent. In continuous operation the niobium in the two recycled products would not be lost and the overall recovery would be slightly higher than indicated. Table 4 is a spectrographic analysis of the high grade precipitate. It is shown that the total impurity level was about 0.10 per cent.

DISCUSSION

The test work has defined a procedure for the production of high purity niobium oxide from pyrochlore-perovskite flotation concentrate. In the work done the overall recovery of niobium from the flotation concentrate was 95.2 per cent in a product containing 99.9 per cent Nb_2O_5 . Figure 4 is a schematic presentation of the laboratory procedures used along with material balances and reagent consumptions. This schematic and the procedural details and results described in the preceding sections serve as a basis for discussion.

The test work in this investigation was limited to small scale laboratory studies in which the many variables, particularly those related to the liquid-liquid extraction operation, were not exhaustively investigated. However, the scope of the work was such that it is possible to assess the applicability of the process to a specific operation. Sufficient information was obtained in reagent consumptions, retention times, solution balances, compositions of solutions, recovery and grade of final product to provide data for an economic feasibility study to determine if further process development is warranted.

TABLE 3

Indicated Nb₂O₅ Distribution
in the Products of Solvent Extraction and Precipitation

Product	Nb ₂ O ₅ Analysis	Nb ₂ O ₅ Dist (%)
ZrO ₂ precipitate	3.5%	0.3
Aqueous phase after extraction on the MIBK	0.3 g/l	1.0
4.75% H ₂ SO ₄ scrub solution	0.004 g/l	0.05
Recycled MIBK	< 0.003 g/l	0.05
Recycled barren from ammonia precipitation	0.01 g/l	0.1
Precipitate (uncalcined)	85%	98.5
Feed to MIBK extraction	23 g/l	100.0

TABLE 4

Semi-Quantitative Spectrographic Analysis
of the Calcined Precipitate, %

Nb ₂ O 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

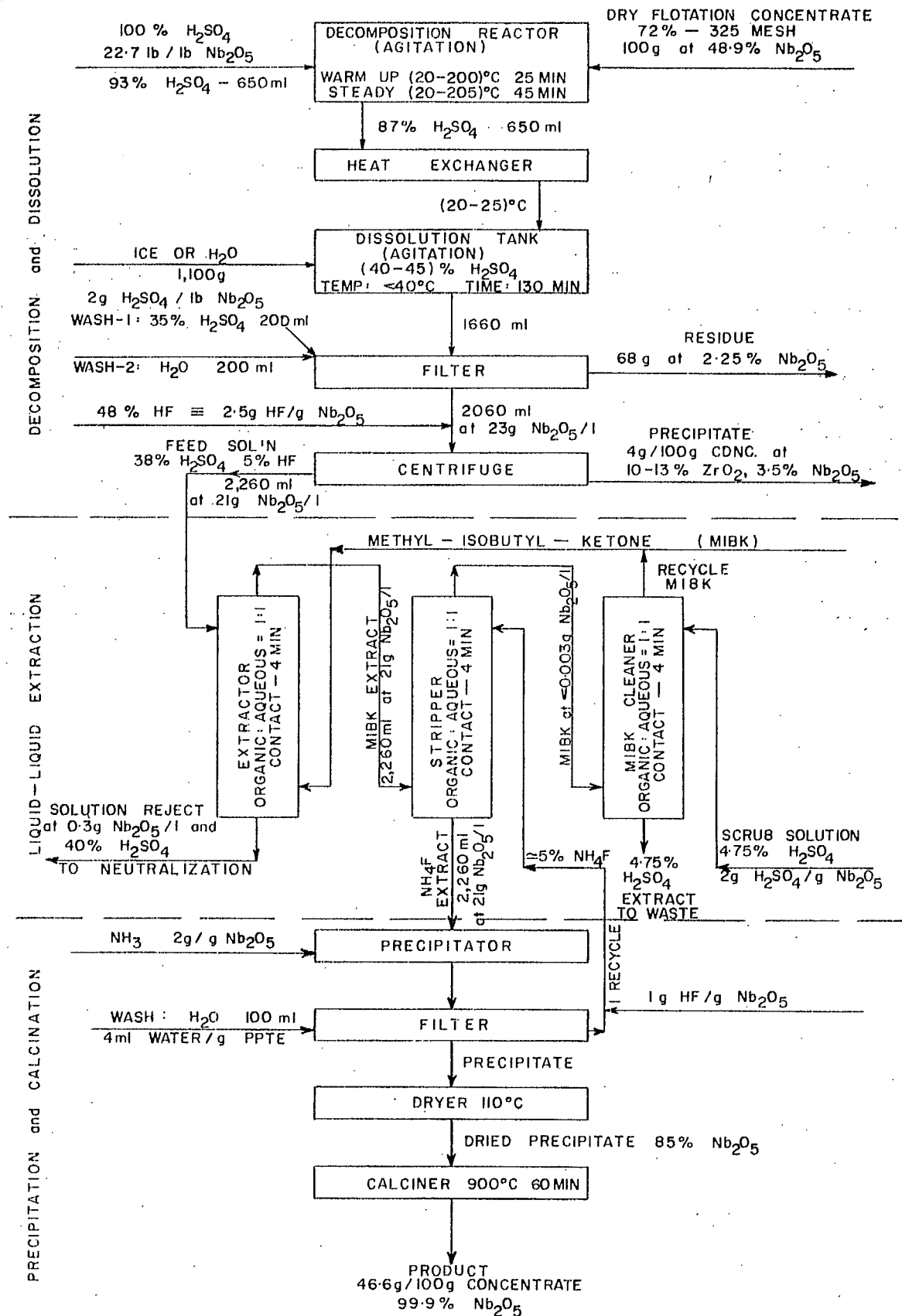


FIGURE 4 SCHEMATIC PRESENTATION OF PROCEDURE USED IN LABORATORY TO PRODUCE Nb₂O₅ FROM FLOTATION CONCENTRATE

The indicated cost of reagents based on the laboratory results is in the order of \$1.25 per pound of Nb_2O_5 recovered. Table 5 gives a breakdown of this figure. It is thought that this Nb_2O_5 reagent cost is probably a maximum figure, and that further investigation would show ways in which it could be reduced. There is reason to believe that the HF concentration in the aqueous feed to liquid-liquid extraction could be lowered below the 5 per cent level used in this work. Each 1 per cent reduction in the HF concentration at this point would reduce the cost by \$0.08/lb Nb_2O_5 . Since the aqueous feed to liquid-liquid extraction contains only 20g Nb_2O_5 /l, it might be possible to reduce the organic:aqueous volume ratio below 1:1, the minimum used in this investigation. A reduction in the organic:aqueous ratio would in turn reduce the volumes of ammonium fluoride and sulphuric acid solutions required to recover niobium from the MIBK and to scrub the MIBK prior to recycling. Possibly a part of the aqueous barren solution from the MIBK extraction step could be used to wash the residue. The MIBK loss experienced with laboratory testing would almost certainly be reduced considerably in a larger scale operation.

TABLE 5

Breakdown of Reagent Costs

	lb/lb Nb_2O_5	\$/lb reagent	\$/lb Nb_2O_5
93% H_2SO_4 for decomposition	24.4	.011	0.263
93% H_2SO_4 for res washing	2.0	"	0.022
93% H_2SO_4 for MIBK scrub	2.2	"	0.024
70% HF to liquid-liquid ext feed	3.6	0.11	0.396
70% HF to precipitation barren	1.5	0.11	0.165
Anhydrous ammonia to pptn	2.0	0.05	0.100
MIBK loss	0.5	0.17	0.085
Lime to neutralize acid wastes	25.0	0.07	<u>0.175</u>
Total			1.235

The ZrO_2 precipitate formed when HF is added to the combined dissolution and wash filtrates is not economically significant since the ZrO_2 recovered amounts to only 0.001 lb ZrO_2 per lb Nb_2O_5 recovered. In practice it might be more economical to add the HF to the dissolution slurry and thus discard the ZrO_2 precipitate with the leach residue. This would eliminate the relatively costly centrifuging step shown in the schematic.

The liquid-liquid extraction operation is highly selective for niobium and through its use a premium grade niobium oxide was produced. The final product analysed 99.9 per cent Nb_2O_5 by chemical analysis which was confirmed by spectrographic analysis. The purity of this product is as good or better than that of currently available analytical grade Nb_2O_5 and would make it ideally suited for feed material for niobium metal production (4). Consequently, this process would be particularly useful if an integrated process going from concentrate to metal were to be considered.

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