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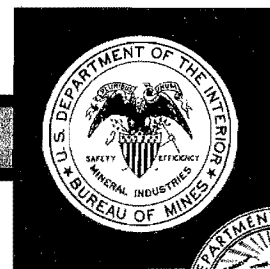
**U.S. BUREAU OF MINES  
E. 315 MONTGOMERY AVE.  
SPOKANE, WA 99207**

# **Column Flotation of Multiple Products From a Fluorite Ore**

**By M. R. Peterson, L. J. Duchene, and M. B. Shirts**

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**BUREAU OF MINES**



**UNITED STATES DEPARTMENT OF THE INTERIOR**

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**Report of Investigations 9309**

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Manuel Lujan, Jr., Secretary**

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## CONTENTS

	<i>Page</i>
Abstract .....	1
Introduction .....	2
Fluorspar .....	2
Potential byproducts .....	2
Beryl .....	2
Mica .....	2
Silica sand .....	2
Flotation .....	3
Experimental materials and equipment .....	3
Flotation columns .....	3
Bubble generator .....	3
Procedure .....	4
Crushing and grinding .....	4
Fluorite flotation circuit .....	5
Mica flotation circuit .....	6
Silicate flotation circuit .....	6
Zeta potential studies procedures .....	6
Discussion of results .....	7
Fluorite flotation .....	7
Process economics of fluorite circuit .....	9
Beryl flotation .....	9
Conclusions .....	10
References .....	11
Appendix A.—Glossary .....	12
Appendix B.—Column flotation parameter testing .....	14
Appendix C.—Fluorite column flotation economics .....	18

## ILLUSTRATIONS

1. Flotation column diagram showing beneficiation zones .....	4
2. Schematic of U.S. Bureau of Mines bubble generator .....	4
3. Flowsheet of Fish Creek CCFU fluorite circuit .....	5
4. Flowsheet of Fish Creek CCFU byproducts circuit .....	6
5. Column profile of fluorite, silica, and calcite upgrading .....	8
6. Zeta potentials of beryl and quartz .....	9
7. Zeta potential of quartz .....	10
8. Zeta potential of beryl .....	10
B-1. Effect of column length on fluorite grades and recoveries .....	14
B-2. Effect of retention time on fluorite grades and recoveries with varying column length .....	14
B-3. Variation of fluorite grades and recoveries with feed injection location .....	15
B-4. Effect of retention time on fluorite grades and recoveries with varying feed injection location .....	15
B-5. Effect of tailings recirculation on fluorite grades and recoveries .....	16
B-6. Effect of froth depth on fluorite grades and recoveries .....	16
B-7. Effect of column wash water on fluorite grades and recoveries .....	16
B-8. Comparison of fluorite grades by particle sizes for column and conventional flotation .....	16
B-9. Comparison of fluorite recoveries by particle sizes for column and conventional flotation .....	17

## TABLES

1. Chemical analyses of Fish Creek sample .....	3
2. Material balance to Fish Creek CCFU .....	5
3. Size distribution of fluorite flotation sample .....	5
4. Comparison of conventional bench and continuous column flotation optimum results .....	7
C-1. Estimated capital cost, fluorspar column flotation .....	18
C-2. Major items of equipment, fluorspar column flotation .....	18

## TABLES—continued

	<i>Page</i>
Equipment cost summary:	
C-3. Feed preparation section, fluorspar column flotation .....	19
C-4. Flotation section, fluorspar column flotation .....	19
C-5. Product handling section, fluorspar column flotation .....	20
C-6. Water recovery section, fluorspar column flotation .....	20
C-7. Raw material and utility requirements, column flotation .....	21
C-8. Estimated annual operating cost, fluorspar column flotation .....	21

### UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

cm/s	centimeter per second	$\mu\text{m}$	micrometer
cm <sup>3</sup> /min	cubic centimeter per minute	mg	milligram
d/yr	day per year	Mgal	10 <sup>3</sup> gallon
°C	degree Celsius	min	minute
°F	degree Fahrenheit	mL	milliliter
ft	foot	mL/min	milliliter per minute
ft/s	foot per second	mm	millimeter
ft <sup>2</sup>	square foot	mm <sup>2</sup>	square millimeter
gal	gallon	mm Btu	10 <sup>6</sup> British thermal unit
h	hour	mV	millivolt
hp	horse power	pct	percent
h/d	hour per day	psig	pound per square inch, gauge
in	inch	s	second
kW·h	kilowatt hour	st	short ton
lb	pound	st/d	short ton per day
lb/h	pound per hour	yr	year
lb/st	pound per short ton		

# COLUMN FLOTATION OF MULTIPLE PRODUCTS FROM A FLUORITE ORE

By M. R. Peterson,<sup>1</sup> L. J. Duchene,<sup>2</sup> and M. B. Shirts<sup>3</sup>

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## ABSTRACT

The U.S. Bureau of Mines investigated column flotation for recovery of a high-grade fluorite ( $\text{CaF}_2$ ) concentrate and byproduct concentrates from the Fish Creek fluorite deposit in Eureka County, NV. The recovery scheme consisted of (1) grinding the ore to minus 48 mesh, (2) fluorite rougher and cleaner flotation, (3) desliming the rougher fluorite flotation tailings at 20  $\mu\text{m}$ , (4) mica flotation, and (5) silicate rougher flotation. Acid-grade fluorspar, mica, free silica ( $\text{SiO}_2$ ) sand, and low-grade beryl ( $\text{Be}_3\text{Al}_2\text{Si}_6\text{O}_{18}$ ) concentrate were produced in a 100 lb/h continuous column flotation unit (CCFU). The highest results achieved were 96.6-pct  $\text{CaF}_2$  recovery in a first cleaner concentrate containing 99.7 pct  $\text{CaF}_2$ . Beryl recovery was as high as 92 pct at a grade of 5.3 pct  $\text{BeO}$ . The silica sand, which assayed 98 pct  $\text{SiO}_2$ , was recovered as the rougher beryl flotation tailings. Collector reagent consumption was reduced 45 pct for fluorite and 58 pct for silicate flotation in comparison with bench-scale conventional flotation. All products were significantly improved in both grade and recovery over conventional flotation results, and the recovery scheme was simplified by removal of several stages of flotation in the fluorite and silicate flotation steps.

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## INTRODUCTION

### FLUORSPAR

Fluorspar, the commercial name for the mineral fluorite, defines material that contains a marketable fluorite concentration; or an aggregate of rock (or ore) with the potential to be upgraded to a fluorite content sufficient to allow industrial application. Fluorspar is marketed in three grades, which have different physical specifications and applications (1):<sup>4</sup>

*Metallurgical grade.*—Also known as "metspar" or "lump spar," is used as a flux (slag forming material) in the steel industry. The required effective fluorite content of 60 to 70 pct is calculated by subtracting 2.5 times the percentage of silica from the percentage of fluorite content. Silica content is limited to 5 to 6 pct, sulfides must be less than 0.50 pct, and lead content below 0.25 pct. The silica limit is relaxed to 15 pct SiO<sub>2</sub> outside the United States.

*Ceramic grade.*—Also known as "glass" and "enamel" grade, must contain a minimum of 95 pct CaF<sub>2</sub>, with a maximum of 2.5 pct SiO<sub>2</sub>. This material must be fine grained for use in opaque and flint glass, in clay bricks, and in vitreous enamel coatings for metal articles.

*Acid grade.*—This is used in the manufacture of hydrofluoric acid and should contain a minimum of 97 pct CaF<sub>2</sub> and no more than 1.1 pct SiO<sub>2</sub>, 1.25 pct CaCO<sub>3</sub>, and 0.03 pct S. Acid-grade fluorite receives the highest price. Uses include the manufacture of Teflon<sup>5</sup> fluorocarbon polymer coatings, fluorine chemicals, synthetic cryolite, refrigerants, and insecticides.

The United States has maintained a high-import reliance (86-90 pct based on apparent consumption) of fluorite for use in the steel, aluminum, uranium, water fluoridation, and chemical industries as a fluorine source. Fluorspar consumption in 1987 was 709,500 st; with a reported acid-grade consumption of 451,000 st, and 147,000 st of metspar (2). Owing to its strong electronegative chemical characteristics, no substitute has been found for most applications of fluorine, making fluorspar a strategic material.

Fluorine consumption was expected to experience a sharp drop due to an international agreement to freeze and later reduce consumption of chlorinated fluorocarbons (CFC's) after a hole in the atmospheric ozone was observed. Despite restrictions on CFC's and the agreement, fluorine consumption increased 12.5 pct to 797,800 st in 1988, including a 30-pct increase in fluorite imports. New substitutes for CFC's being developed will require more fluorine per molecule and may stabilize or increase consumption (3).

<sup>4</sup>Italic numbers in parentheses refer to items in the list of references preceding the appendix at the end of this report.

<sup>5</sup>Reference to specific products does not imply endorsement by the U.S. Bureau of Mines.

At projected consumption rates, currently exploited fluorspar deposits of the world are expected to be depleted by the end of the century. As many domestic and foreign deposits of fluorite are exhausted, alternates such as marginal deposits, secondary sources, or multicomponent ores will have to be developed. One such marginal deposit is the Fish Creek fluorite deposit of Eureka County, NV, which contains ore reserves estimated from 2.5 to 5 billion st of ore at a grade in excess of 10 pct CaF<sub>2</sub>. This represents the United States largest known fluorite deposit.

### POTENTIAL BYPRODUCTS

#### Beryl

The Fish Creek ore also contains potential byproduct minerals of beryl, mica, and silica sand. Pure beryl mineral contains 5 pct Be or 14 pct BeO. Beryllium is used as an alloy and metal in aerospace and defense uses requiring lightweight, high strength, and high-thermal conductivity in structural and electronic equipment applications. Beryl and beryllium oxide are also used as moderators for nuclear radiation. There are no substitutes that can match beryllium's performance in these products. The 318 st of beryllium consumed in 1987 were valued near \$145 million. Use of beryllium in high-speed circuits and electronic components in cars is rapidly increasing. The National Defense Stockpile is increasing its supply by 60,000 lb (4).

Most exploration drill cores taken from the Fish Creek deposit were not analyzed for beryllium content, but all samples used in this study assayed between 0.1 and 0.2 pct BeO. If consistent throughout the deposit, this would greatly augment present world beryllium reserves.

#### Mica

Practical uses for mica (muscovite) are determined by particle size, 100- to 200-mesh scrap, and flake mica is used primarily in building and construction applications such as asphalt products, texture paints, ceiling tiles, concrete block filler, and wall joint cement. Finer sized mica (160-to 325-mesh) is used for paints, and as an additive for plastics to increase strength, heat resistance, or dielectric properties (5).

#### Silica Sand

Silica has a wide variety of applications, which grow in proportion to its purity. Abrasives, casting sands for foundries, furnace linings, filter media, glass, ceramics and specialty chemicals all have markets. Prices are low for raw sand, and the marketability of sand is greatly dependent on transportation costs. The most detrimental



contaminant is iron, limited to 0.025 and 0.15 pct  $\text{Fe}_2\text{O}_3$ , respectively, for white and amber glass manufacture (6).

## FLOTATION

Development of domestic low-grade fluorite resources has been retarded by reliance upon imports from foreign sources, and by inadequate technology for concentrating its low-grade ores. A method for recovering fluorite and byproducts from the Fish Creek ore was developed by the U.S. Bureau of Mines (7-8) based on previous Bureau research on similar ores (9) using bench-scale froth (conventional) flotation; however, the ore responded poorly to continuous operation. Column flotation has been shown to provide superior results when used in many applications currently limited to conventional flotation processes. An increase in concentrate grade, product recovery, or

decrease in the number of flotation stages required to achieve product grade, or a combination of these effects has been documented on several ores examined at a Bureau facility (10-14). A study of parameter effects with fluorite ore was carried out (14) and is summarized in appendix B. The advantages of column flotation include improved energy efficiency, utilization of natural settling and separation, reduced turbulence in the flotation cell, and closer control of critical flotation parameters such as bubble size, cell air content, airflow, retention time, froth depth, and froth cleaning. A reduction in reagent consumption using column flotation in comparison with conventional flotation is common. Because of the enhanced results produced by column flotation with other ores, fluorite column flotation tests were performed on a Fish Creek ore sample.

## EXPERIMENTAL MATERIALS AND EQUIPMENT

A 10,000-lb sample was obtained from the Fish Creek fluorspar deposit near Eureka County, NV, which also contains beryllium and muscovite byproduct values. The sample was crushed and screened through 1/4 in, thoroughly blended, after which representative portions were submitted for chemical and petrographic analysis. The chemical analyses are shown in table 1.

Table 1.—Chemical analyses of Fish Creek sample, percent

Constituent	Composition
Fluorite ( $\text{CaF}_2$ ) . . . . .	14.3
Beryl ( $\text{BeO}$ ) . . . . .	.2
Calcite ( $\text{CaCO}_3$ ) . . . . .	5.12
Total Silica ( $\text{SiO}_2$ ) . . . . .	66.5
Total Iron ( $\text{Fe}$ ) . . . . .	1.37

Petrographic analysis of the fluorite beryllium sample indicated that it contained major amounts of quartz ( $\alpha\text{-SiO}_2$ ), fluorite, and muscovite (light-colored mica [ $\text{KAl}_3\text{Si}_3\text{O}_{10}(\text{OH})_2$ ]). Minor amounts of hematite ( $\text{Fe}_2\text{O}_3$ ), limonite (hydrous iron oxides), potassium-feldspar ( $\text{KAlSi}_3\text{O}_8$ ), beryl, calcite ( $\text{CaCO}_3$ ), and chlorite (hydrous silicates of aluminum, ferrous iron, and magnesium) were observed. No reliable chemical method was available for determining muscovite concentration, therefore, petrographic analysis was used to estimate mica content. Petrographic estimates for quartz and mica were 28 and 30 pct, respectively. Liberation size for all minerals was 48 mesh.

### FLOTATION COLUMNS

Flotation was accomplished in four 2.5-in diam columns. The rougher fluorite flotation column was 18 ft high; the other three columns (cleaner fluorite, and mica and beryl rougher flotation columns) were 12 ft high. Each column was composed of flanged sections with 1.125-in-diam ports

located at 1-ft intervals along its length. Solenoid-actuated sample valves with stainless steel spring loaded plungers were designed and constructed at a Bureau facility and fitted into the ports. These valves permitted rapid, consistent extraction of a representative pulp or froth sample. A concentric cylindrical overflow weir was mounted on the top section of each column. A conical bottom with a 0.5-in-diam port was attached to the bottom section of each column. Figure 1 presents a diagram of a flotation column.

### BUBBLE GENERATOR

The Bureau's bubble generation system was used to aerate the flotation column. The bubble generator was a 6-in-tall by 2-in-diam clear cylindrical Plexiglas acrylic plastic chamber with 1-in thick walls. This is illustrated in figure 2. The generator had a removable Plexiglas acrylic plastic top attached to the generator body with an O-ring seal. A spacer supported a fritted glass disk in the chamber to prevent short circuiting of air. The remaining chamber volume was filled with 0.04-in-diam glass beads to improve air-water contact. Teflon fluorocarbon polymer chips or quartz gravel, coarsely sized at plus 6 mesh (plus 0.132 in) were substituted for the glass beads with satisfactory results.

A section of 28-mesh screen was placed over each orifice to prevent loss of glass beads. House air was regulated to 60 psig and introduced through the side port of the bubble generator. Water, pressurized to 60 psig by a turbine-blade pump, was introduced through the top bubble generator orifice. Air and water were mixed in the contact chamber; the pressurized mixture exited the chamber through the bottom port and was injected into the column through an aluminum or Teflon fluorocarbon polymer tip with a 0.04-in-diam orifice. The bubbles were

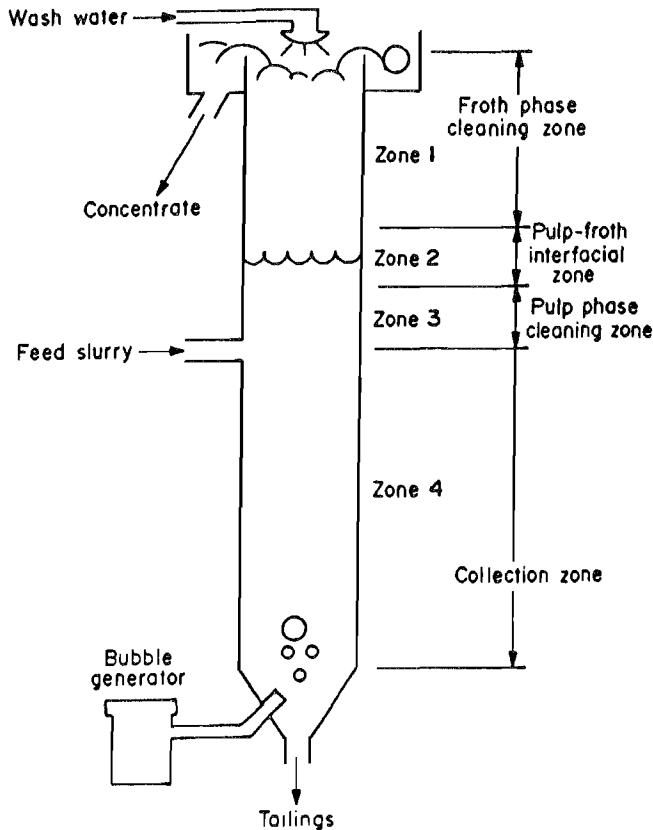


Figure 1.—Flotation column diagram showing beneficiation zones.

still-photographed inside the column alongside a microgrid and measured after development. The generator design allowed for bubble size control from less than 0.004- to more than 0.12-in average bubble diameter (0.1- to 3-mm) by adjusting air and waterflow rates and by adding Dowfroth 400 frother. Previously published work on this parameter investigation (10, 14-15) showed coarse bubbles (3- to 5-mm diam) produced the best results on the Fish Creek fluorite ore, and were therefore used for all testing.

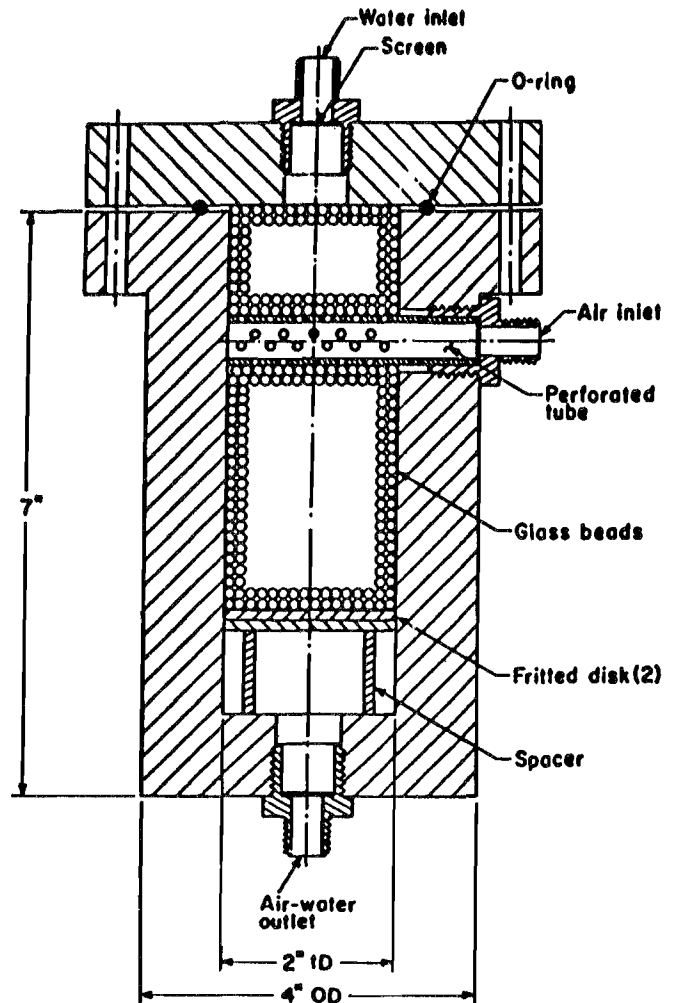


Figure 2.—Schematic of U.S. Bureau of Mines bubble generator.

An airflow rate of 4,500 cm<sup>3</sup>/min and a waterflow rate of 800 mL/min were used to generate the 3- to 5-mm-diam bubbles for this testwork.

## PROCEDURE

The finalized CCFU flowsheet is given in figures 3 and 4. Table 2 includes the material balance for a 2,000-st/d plant used for the economic evaluation appearing in

appendix C. The data are derived from pilot plant results obtained under preferred, sustained operation conditions.

## CRUSHING AND GRINDING

In the CCFU run-of-mine (as-received) ore, initially crushed to minus 1/4 in, was preclassified at a rate of 100 lb/h on a 48-mesh vibrating screen. The plus 48-mesh material was fed directly to the 24- by 36-in rod mill and ground at 50 pct solids using a standard rod charge. The

minus 48-mesh material was added to the rod-mill discharge. The undersized and ground oversized material were slurried at 25 pct solids. The grind size distribution of the flotation feed solids is given in table 3.

Table 2.—Material balance to Fish Creek CCFU based on 2,000 st/d feed

Stream <sup>1</sup>	Solids, st/d	Fluorite, st/d	Assay, pct CaF <sub>2</sub>	Beryl, st/d	Assay, pct BeO
1. Feed ore .....	2,000	286	14.3	20	0.2
2. Plus 48-mesh ore .....	1,500	214.5	14.3	15	.2
3. Minus 48-mesh ore .....	500	71.5	14.3	5	.2
4. Flotation fee .....	2,000	286	14.3	20	.2
5. Rougher CaF <sub>2</sub> concentrate .....	330	273.1	82.77	.15	.01
6. Rougher CaF <sub>2</sub> tailings .....	1,670	12.9	.77	19.85	.24
7. Cleaner CaF <sub>2</sub> concentrate product ..	280.1	271.7	97	.05	<.01
8. Cleaner CaF <sub>2</sub> tailings .....	49.9	1.4	2.98	.1	.04
9. Slimes .....	400	6	1.5	2.2	.11
10. Mica flotation feed .....	1,270	6.9	.54	17.65	.28
11. Mica concentrate product .....	666	2.4	.36	.9	.03
12. Mica tailings .....	604	4.5	.74	16.75	.55
13. Magnetics .....	13	.2	1.58	.19	.29
14. Nonmagnetics .....	591	4.3	.72	16.56	.56
15. Recycle water .....	NAP	NAP	NAP	NAP	NAP
16. Silicate flotation feed .....	591	4.3	.72	16.56	.56
17. Silicate concentrate .....	62.6	4.2	6.71	16.33	5.22
18. Silicate tailings .....	528.4	.1	.01	.23	.01

NAP Not applicable.

<sup>1</sup>Stream numbers correspond to circled numbers in figures 3 and 4.

Table 3.—Size distribution of fluorite flotation sample

Size, mesh	Weight percent
Plus 48 .....	4.4
Minus 48 Plus 65 ....	9.5
Minus 65 Plus 80 ....	6.6
Minus 80 Plus 100 ...	6.5
Minus 100 Plus 150 ..	11.4
Minus 150 Plus 200 ..	13.4
Minus 200 Plus 270 ..	12.5
Minus 270 Plus 325 ..	5.6
Minus 325 Plus 400 ..	3.9
Minus 400 .....	<u>26.2</u>
Composite .....	100.0

## FLUORITE FLOTATION CIRCUIT

Preferred conditions for conventional flotation of the Fish Creek ore determined previously using bench-scale recovery scheme development and mineral parameter testwork (7-8), were as follows: 3 lb/st sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), 3 lb/st N-sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>), 2 lb/st oleic acid (C<sub>18</sub>H<sub>34</sub>O<sub>2</sub>); with a 15 min conditioning time for each reagent and a flotation pH of 9. When these conditions were directly applied to initial continuous column flotation tests, they were found to be excessive and could be reduced to conditioning with 2.9 lb/st Na<sub>2</sub>CO<sub>3</sub> for 3 min, followed by 2 lb/st Na<sub>2</sub>SiO<sub>3</sub> for 3 min. Oleic acid (dissolved in ethanol) was then added at 1.14 lb/st and conditioned for 3 min, while the pH was maintained near the natural ore pH of 9 with the addition of Na<sub>2</sub>CO<sub>3</sub> and hydrochloric acid (HCl) as needed.

A cascading pilot plant setup was employed to maximize the use of gravity feeding from stage to stage and minimize mechanical difficulties in pumping between process stages. The conditioned fluorite rougher feed material was

pumped at 25 pct solids through an agitated feed surge tank to an entry port located 11 ft from the column base using a peristaltic pump and flexible tubing. Mineralized froth continuously flowed from the top of the fluorite rougher column to the feed port located 7 ft from the base in the fluorite cleaner column. Final fluorite concentrate continually flowed by gravity from the cleaner column to a pan-type filter for recovery. Tails from the cleaner column were pumped out, filtered, and discarded.

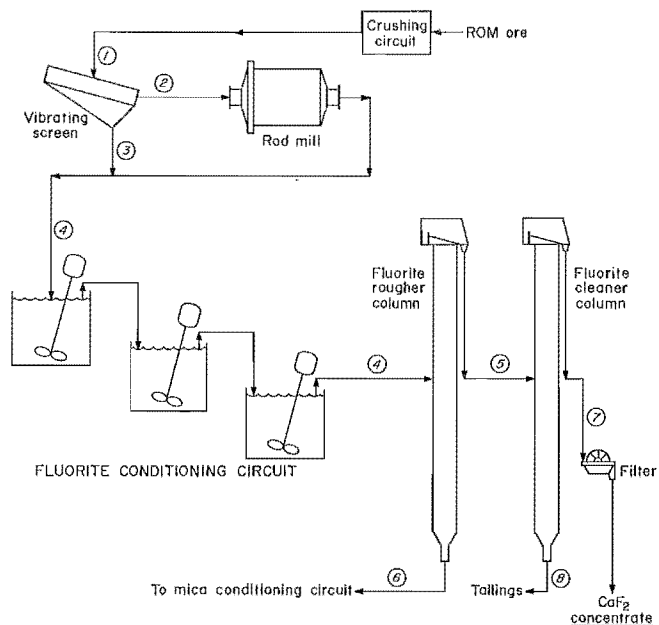


Figure 3.—Flowsheet of Fish Creek CCFU fluorite circuit. Circled numbers correspond to stream numbers in table 2.

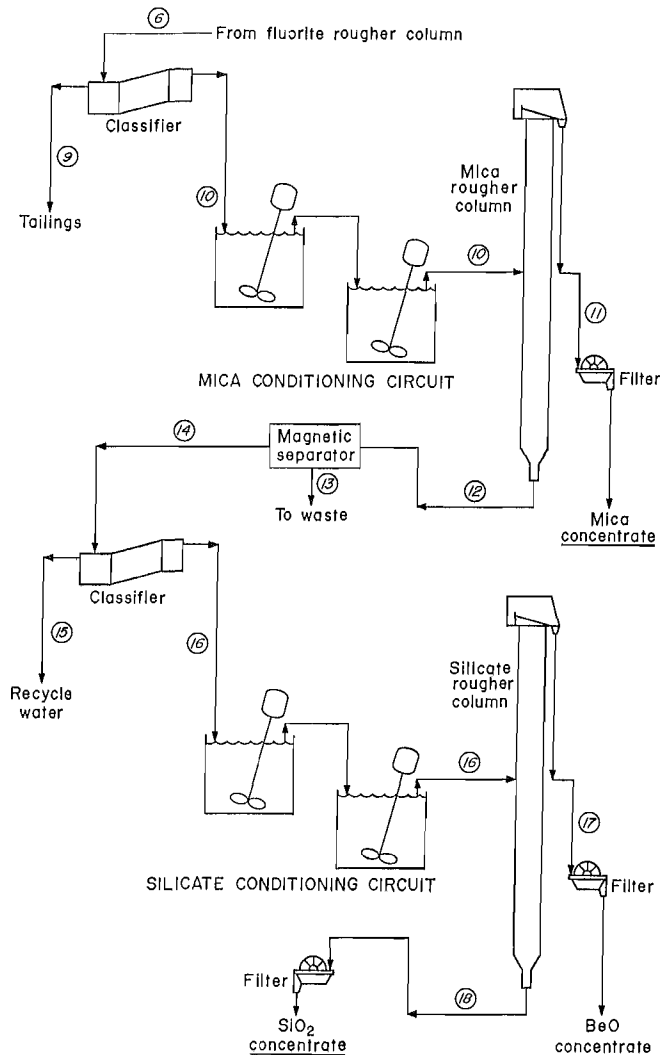


Figure 4.—Flowsheet of Fish Creek CCFU byproducts circuits. Circled numbers correspond to stream numbers in table 2.

### MICA FLOTATION CIRCUIT

The tailings from the rougher fluorite float were pumped from the base of the column to a spiral classifier for desliming at  $20\ \mu\text{m}$ . The minus  $20\ \mu\text{m}$  slime material at approximately 7 pct solids was sent to filtration, while the plus  $20\ \mu\text{m}$  material was diluted to approximately 20 pct solids and fed to the byproduct recovery circuit (fig. 4), starting with the mica conditioning and flotation circuit.

The preferred method for treatment in the mica circuit consisted of conditioning the deslimed rougher fluorite flotation tailings with 5 lb/st sulfuric acid ( $\text{H}_2\text{SO}_4$ ) for 3 min, followed by 0.75 lb/st primary ether amine acetate (MG-98A) dissolved in ethanol for 3 min, while controlling pH at 2.0 to 2.8 with the addition of sodium hydroxide (NaOH) as needed. This compares with the conventional

flotation method in which the feed was conditioned 3 min each with 5 lb/st  $\text{H}_2\text{SO}_4$  followed by 0.4 lb/st MG-98A dissolved in ethanol (7-8). The conditioned mica flotation feed material was pumped at approximately 20 pct solids through flexible tubing from the conditioner to a port located 7 ft from the column base using a peristaltic pump. Mineralized froth was continuously removed from the top of the mica rougher column and gravity fed to filtration. The tailings from mica flotation were pumped from the base of the column to a magnetic separator for tramp iron removal. After magnetic iron removal, the slurry was sent to a spiral classifier for dewatering ahead of silicate flotation conditioning.

### SILICATE (BERYL) FLOTATION CIRCUIT

The preferred method for silicate flotation in the CCFU was to stage condition the spiral classifier product with 4 lb/st hydrofluoric acid (HF) and 0.05 lb/st sodium fluoride (NaF) for 3 min, followed by additions of 0.7 lb/st tallow amine acetate (Armac-T) dissolved in ethanol, and 7.5 lb/st acetic acid ( $\text{CH}_3\text{COOH}$ ) conditioned for 3 min. The conditioned silicate feed material was pumped at approximately 20 pct solids through flexible tubing from a steady feed tank to a port located 7 ft from the column base using a peristaltic pump. Mineralized froth (silicate concentrate) was continuously removed from the top of the silicate rougher column and gravity fed to filtration. Silicate rougher flotation column tailings consisted almost exclusively of free quartz, a potential glass sand feed material, was pumped directly to filtration.

The original recovery scheme developed by bench-scale conventional flotation testwork involved conditioning with 5 lb/st HF for 3 min, followed by conditioning for 20 s with 1.2 lb/st tallow amine acetate for rougher silicate flotation. The rougher silicate concentrate was then conditioned with 1.2 lb/st sodium hypochlorite (bleach) to remove the amine collector followed by sorption of the organic chemicals onto 12 lb/st activated charcoal, rinsing, and filtration. This was followed by cleaner flotation of the beryl using an emulsion of 0.39 lb/st oleic acid with 0.15 lb/st Triton X-45 emulsifier at a pH of 6.2 using sulfuric acid to depress gangue silicates, and 0.05 lb/st NaF for beryl activation. No beneficiation was observed in the CCFU when this method was employed.

### ZETA POTENTIAL STUDIES PROCEDURES

The zeta potential testwork was carried out with pure beryl and quartz samples from Wards Minerals, Rochester, NY. The samples were ground to minus 200 mesh in a ceramic mortar and pestle. In each test, 200 mg of sample was added to 100 mL of pH adjusted, deionized water containing the desired concentration of reagent(s). After 5 min of conditioning, the mixture was transferred into the Riddick cell for zeta potential measurement. All pH adjustments were made with either HCL or NaOH.

## DISCUSSION OF RESULTS

A 100-lb/h CCFU was operated on the bulk Fish Creek sample (figs. 3-4), and removed four products: fluorite, mica, silica sand, and a beryl silicate concentrate. The best results achieved for the CCFU are summarized in table 4 along with a comparison with bench and continuous column flotation results using the same flotation procedure. Reagent additions were unavailable for the conventional silicate flotation circuit. Preferred reagent dosages for the CCFU were 2.9 lb/st  $\text{Na}_2\text{CO}_3$ , 2.0 lb/st  $\text{Na}_2\text{SiO}_3$ , and 1.14 lb/st  $\text{C}_{18}\text{H}_{34}\text{O}_2$  to the fluorite circuit (pH 9); 5 lb/st  $\text{H}_2\text{SO}_4$  and 0.75 lb/st MG-98A in the mica circuit (pH 2.8); 4.0 lb/st HF, 0.7 lb/st tallow amine acetate, 7.5 lb/st  $\text{CH}_3\text{COOH}$ , and 0.05 lb/st NaF after magnetically removing the tramp iron from the mica flotation tailings (pH <3).

Table 4.—Comparison of conventional bench and continuous column flotation optimum results

Stage	Reagents, lb/st	Grade, pct	Recovery, pct
<b>CONVENTIONAL BENCH</b>			
Fluorite ...	$\text{Na}_2\text{CO}_3$ ... 3	98.1 $\text{CaF}_2^1$	94
	$\text{Na}_2\text{SiO}_3$ ... 3		
	Oleic ... 2		
Mica .....	$\text{H}_2\text{SO}_4$ ... 5	90 mica	90
	MG-98A ... .7		
Silicate .....	HF ... 5	2.83 BeO	83.5
	Armac-T ... 1.2		
	NaF ... .05		
<b>CONTINUOUS COLUMN</b>			
Fluorite ...	$\text{Na}_2\text{CO}_3$ ... 2.9	99.7 $\text{CaF}_2$	96.6
	$\text{Na}_2\text{SiO}_3$ ... 2		
	Oleic ... 1.1		
Mica .....	$\text{H}_2\text{SO}_4$ ... 5	90 mica	90
	MG-98A ... .75		
Silicate .....	HF ... 4	5.27 BeO	92
	Armac-T ... .7		
	NaF ... .05		
	$\text{HC}_2\text{H}_3\text{O}_2$ ... 7.5		

<sup>1</sup>Second cleaner concentrate.

Fluorite rougher column plug flow retention times based on slurry feed rate and collection zone volume (column volume between feed injection port and air sparger) were approximately 5 min compared with conventional flotation retention times of 15 min. Flow to subsequent processing stages was maintained as necessary to match discharge from the fluorite rougher column.

Using the preferred reagents and conditions in the CCFU, excellent results were achieved. Average cleaner fluorite concentrates of 98.3 pct  $\text{CaF}_2$  (acid grade) with recoveries of 94.9 pct were achieved. The highest grade and recovery achieved were a 99.7-pct  $\text{CaF}_2$  concentrate with 96.6 pct recovery. Silica content was less than 0.5 pct;  $\text{CaCO}_3$ , S, and Pb assays were below detection limits (0.1, 1.0, and 0.03 pct, respectively). It was estimated by petrographic analysis that a 90-pct mica concentrate was

produced with more than 90 pct recovery. The silicate flotation stage produced two concentrates. The tailings, a silica sand product, always assayed more than 96 to 99 pct  $\text{SiO}_2$ . Iron content around 0.2 pct  $\text{Fe}_2\text{O}_3$  prohibits use of this material in glass making, but it would be suitable for use in the manufacture of abrasives, sodium silicate chemicals, and other products. The silicate concentrate produced using the preferred reagent scheme contained 92 pct of the beryl at a grade of 5.3 pct BeO.

The flow scheme for recovery of fluorite and byproducts from the Fish Creek ore was developed in bench-scale testwork reported in Bureau's report of investigations (RI) 9028 (7). Operation of the CCFU resulted in elimination of (1) two cleaner flotation stages in the fluorite flotation circuit, and (2) a cleaner stage of beryl flotation as well as a reagent removal system requiring sodium hypochlorite treatment, filtration, and activated charcoal treatment. The magnetic iron removal was moved from the rougher silicate concentrate to the mica flotation tailings stream. Column flotation also resulted in higher grades of fluor-spar, mica, and beryl products with better recovery.

### FLUORITE FLOTATION

Water temperature was found to be an important factor in continuous flotation of fluorite. This effect was unreported in bench studies, probably because of the rapid adjustment to room temperature of small ore charges in contact with flotation machines. Highest recoveries were achieved while maintaining the circuit feed water at 75° F. An 18° F reduction in temperature resulted in a recovery drop of 20 pct or more. The effect seen on the concentrate grade was not as pronounced.

Batch column testing significantly improved the results achieved in bench conventional flotation of the Fish Creek ore. With the columns, adjustment of operating parameters resulted in increased concentrate grades. Manipulation of parameters such as froth depth, wash water addition rate, aeration rate, bubble size, and feed injection location (retention time) had pronounced effects on column performance and metallurgy and were easily incorporated into the CCFU.

Coarse bubbles with moderate air holdups of approximately 12 to 15 pct were chosen as optimal, based on test results prior to CCFU operation. Use of fine bubbles and high-air holdups increased recoveries, but decreased grades as calcite also floated with the fluorite under these conditions.

Column profile samples from a continuous test were analyzed for fluorite, silica, and calcite content (fig. 5). Fluorite was upgraded to the concentrate stream (top of the column), while silica and calcite were rejected to the tailings stream (base of the column). The most efficient separation of fluorite from silica occurred in the froth phase cleaning and pulp-froth interfacial zones (figs. 1 and

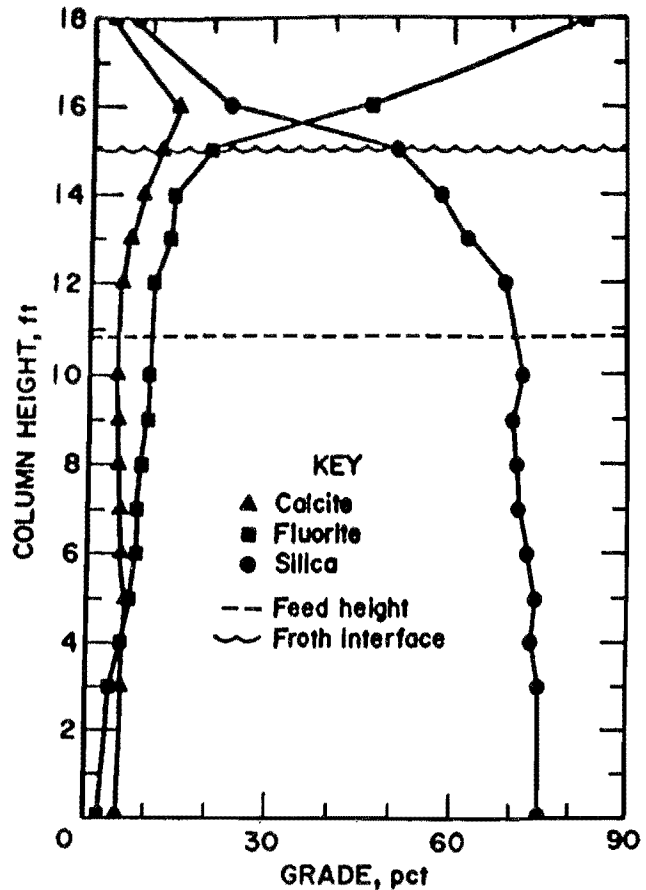
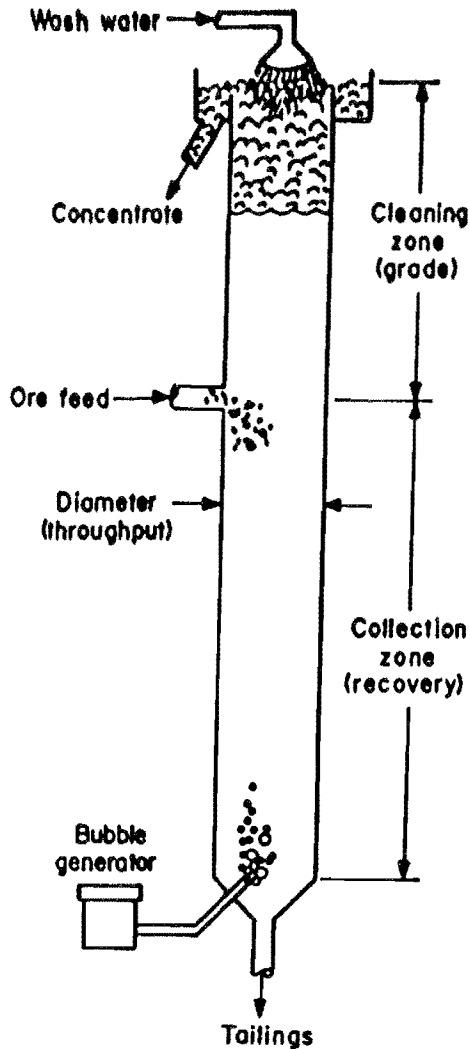


Figure 5.—Column profile of fluorite ( $\text{CaF}_2$ ), silica ( $\text{SiO}_2$ ), and calcite ( $\text{CaCO}_3$ ) upgrading.

5). This suggests that the specific function of zones 1 and 2 is upgrading, linking them strongly to mineral concentrate grade, while zones 3 and 4 predominantly act as collection zones, linking them specifically to mineral recoveries.

Calcite behavior during column flotation was particularly interesting because the flotation response of calcite mirrored that of the collected fluorite and that of the rejected silica at different points within the column. Calcite concentrated with the fluorite in the lower three column beneficiation zones. Calcite rejection was accomplished in the froth phase cleaning zone. Unlike these results in the CCFU, continuous and bench conventional froth flotation were unable to reject the entrapped calcite.

The fluorite rougher column employed low wash water and shallow froth beds (<2 ft) to achieve good grades with high-fluorite recoveries. The fluorite cleaner

column used 6 ft of froth and high wash water addition rates (>100 pct of feed slurry flow rate) and achieved substantial upgrading of the fluorite with rejection of the silica and calcite. Feed was injected high in the fluorite rougher flotation column (to achieve high recovery) and lower in the fluorite cleaner column (where bubble particle attachment was rapid) where grade was the primary objective. With this, 99 pct  $\text{CaF}_2$  grades were attainable.

Maintenance of high-fluorite recoveries was necessary for improved downstream byproduct grades. Conventional flotation would have required multiple stages of scavenger and cleaner flotation to equal the higher grades achieved using column flotation. Use of Plexiglas acrylic plastic in construction of the columns also allowed close monitoring of froth heights and bubble generator performance. Close control of aeration rate in the mica flotation column prevented production of an uncontrollable froth (which

occurred under high aeration conditions with the amine flotation chemistry) and resulted in higher purity mica concentrates because of less gangue entrainment.

### PROCESS ECONOMICS OF FLUORITE CIRCUIT<sup>7</sup>

Based on the promising results achieved with column flotation, the Bureau conducted a cost evaluation on column flotation to recover fluorite only from the Fish Creek deposit. The evaluation concluded that a 2,000-st/d column flotation mill could operate with an estimated 12 pct return on investment after taxes primarily due to the higher price commanded by the high-purity fluorite product.

This process evaluation considered the marketability of fluor spar only because markets for mica, beryl, and glass sand were too small to justify processing the entire tailings stream for byproducts. The evaluation was formulated with conservative estimates to assure reliability, and did not take land investment, mineral depletion allowance, or plant owner's supervision cost into account. A Marshall and Swift (M & S) index of 897.0 was used for the evaluation estimates, performed in the third quarter of 1989. Details of the economic evaluation for column flotation of fluorite are presented in appendix C.

### BERYL FLOTATION

Several problems were encountered in the flotation of beryl in the CCFU. Iron minerals and iron stained quartz were difficult to reject with the cationic (amine) collector system. Even with the use of HF to condition the mineral surfaces, close control of collector addition had to be maintained to produce a concentrate superior to that produced using conventional froth flotation methods.

Even the presence of high levels of iron ions in the water had to be prevented to obtain selectivity in beryl flotation. Iron contamination from mild steel conditioning tanks interfered with beryl flotation selectivity and had to be replaced with stainless-steel tanks. Iron activation of minerals in silicate flotation has been reported by other investigators (16-17), as well as the difficulties in beryl flotation with fatty acids and other collectors (18-20). No process has reported success in producing a suitable beryl concentrate (BeO content of 10- to 11-pct, where pure beryl has a BeO content of 14 pct) strictly using flotation. All beryllium production has been accomplished with hydrometallurgical processes from bertrandite and high-grade beryl sources.

Beryl concentration from the low-grade ore in the CCFU was also hampered by the greatly decreased flow rates at the end of the process, and the time required to accumulate enough material to begin the unit operation. Separation of iron-stained quartz and iron minerals was

difficult due to poor selectivity of the collector (tallow amine acetate).

Incorporation of the initial recovery scheme (7), including a reagent removal scheme followed by beryl flotation of the silicate concentrate with an oleic acid emulsion, did not improve CCFU beryl grades. In bench flotation, a 5.96-pct BeO concentrate with a recovery of 65.8 pct of the beryl values was achieved; but in CCFU operation the cleaner beryl flotation increased grade from 1.76 pct BeO to only 3.35 pct BeO with poor recovery. The continuous conventional system based on the original scheme was unable to exceed 1 pct BeO due to material control problems and inferior recoveries.

Zeta potential studies were performed on pure quartz and beryl samples to find conditions allowing improved quartz-beryl separation. Effects of HF, NaF, and  $C_{18}H_{34}O_2$  emulsion were examined with varying pH and also varying concentrations of HF and  $C_{18}H_{34}O_2$  emulsion. Results of this basic research were not completely applicable to beryl flotation of the actual ore because of the use of pure minerals and the absence of iron in the tests.

The surface charges of beryl and quartz were nearly identical without reagents (fig. 6), with the only difference lying in the pH range of 2.0 to 2.5, where beryl still has a

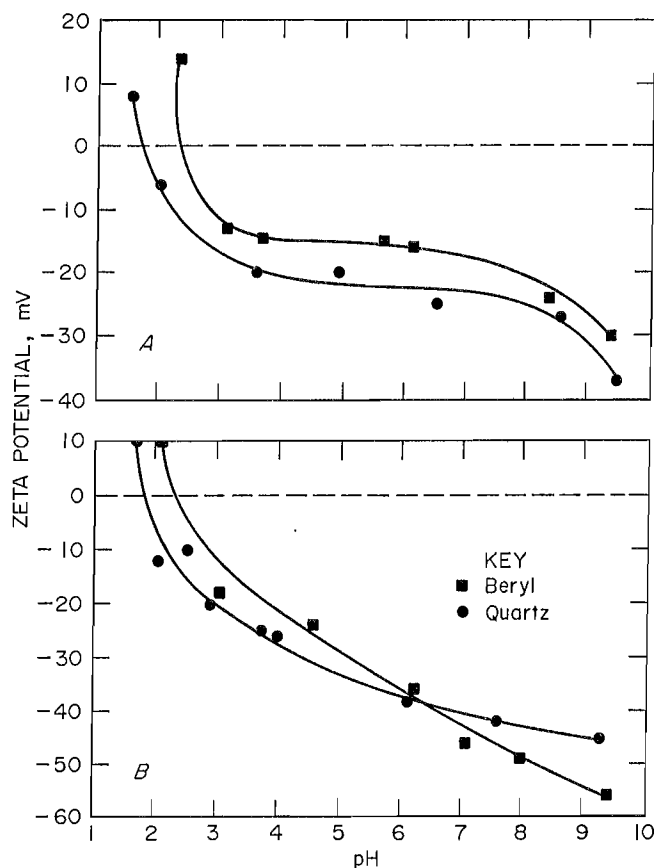


Figure 6.—Zeta potentials of beryl and quartz. A, Without reagents, B, with 0.39 lb/st of oleic emulsion.

<sup>7</sup>Process evaluation was conducted by T. A. Phillips, Staff Engineer, Research Directorate, U.S. Bureau of Mines, Washington, D.C.

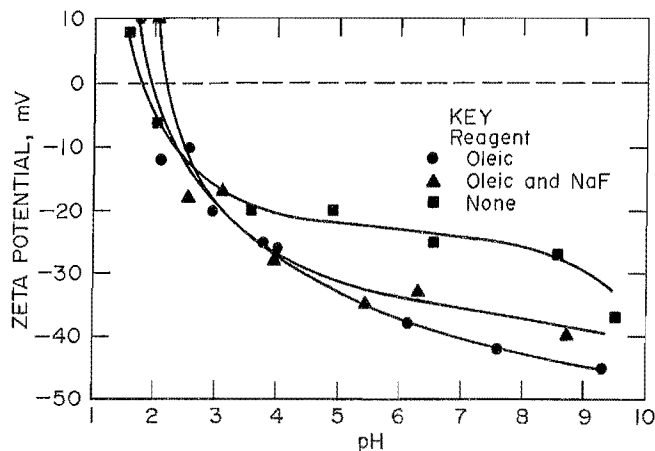


Figure 7.—Zeta potential of quartz with 0.05 lb/st NaF and 0.39 lb/st oleic acid emulsion.

slightly positive surface. In the presence of 0.39 lb/st oleic emulsion (fig. 7), the surfaces remain similar, but the beryl surface charge was more negative above a pH of 4 due to collector adherence to the surfaces. Adherence to the quartz surface was also quite strong showing oleic acid to be nonselective between beryl and quartz, supporting data from the CCFU.

Fluoride additions had the strongest effects on the mineral surfaces. Below a pH of 2.3, quartz showed an extended range of positive surface in the presence of 0.05 lb/st NaF and 0.39 lb/st oleic emulsion (fig. 7). This indicates a resistance to oleic acid attachment to the surface. Use of HF decreases the pH range in which beryl exhibits a positive surface charge (fig. 8). Anionic flotation of beryl would be possible in this region, but acid consumption would be high. The difference between the curves is small and lies in the region of lowest precision for the instrument. Overall, these data all point to a poor applicability of anionic (fatty acid) flotation for separation of beryl and quartz.

Addition of acetic acid to the CCFU silicate flotation circuit aided separation of the beryllium silicate from the

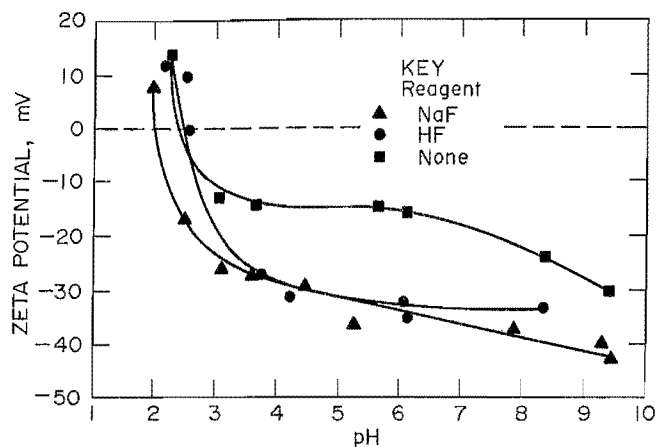


Figure 8.—Zeta potential of beryl with 0.05 lb/st NaF or HF.

silica sand, increasing the rougher concentrate grade from approximately 1.8 pct to 5.0- to 5.5-pct BeO. The increase in selectivity is believed to be due to improved dispersion and decreased amounts of collector attached to particles allowing the column flotation system to reject particles containing iron or silicate middlings. No significant decrease in beryl recoveries was noted, and silicate flotation tailings assays increased by only 0.01 pct BeO or less. Control of froth depth and wash water addition were used to obtain maximum BeO grade and recovery depending on silicate column froth characteristics. This physical process manipulation was not possible in conventional froth flotation.

Increased additions of acetic acid and cleaner flotation stages in the beryl flotation circuit showed minimal improvements, recovering 70.7 pct of the beryl at a grade of 7.36 pct BeO in a second cleaner concentrate with a total addition of 20 lb/st  $\text{CH}_3\text{COOH}$ . The amine attached to the iron stained particles was too tenacious for the wash water and acetic acid to overcome.

## CONCLUSIONS

Based upon results obtained in operating the continuous column flotation unit with Fish Creek ore, it is concluded:

1. An acid-grade fluorite product can be recovered with rougher and a single stage of cleaner flotation using flotation columns with bubble generators developed by the U.S. Bureau of Mines.

2. Muscovite and silica sand byproducts of commercial grade can also be recovered by column flotation of the deslimed rougher fluorite tailings.

3. Column flotation can control flotation grades and recoveries more closely by providing greater flexibility of

- operation parameters including: (1) controllable froth depth; (2) addition of wash water to enhance removal of mineral particles only slightly bound by collector to the bubbles in froth phase; (3) minimization of turbulence that may promote bubble particle detachment; and (4) control of retention time by variation in feed injection location and column height.

4. Water should be kept at room temperature or higher for optimum fluorite grades and recoveries.

5. Acetic acid addition improved selectivity of the amine acetate collector in beryl flotation.



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## APPENDIX A.—GLOSSARY

**Air holdup.**—The volume percent of air present in the flotation column at any given time. This may be measured by filling a chamber (such as a flotation column) with water or slurry and bubbles, closing off the bubble source, and measuring the drop in water-pulp level compared with the level of bubbles present, or through use of a hydrostatic manometer.

**Air injection point.**—The vertical location on the column at which the air or air-water mixture is injected into the column in the form of bubbles.

**Air slugs.**—Large volumes of air formed from either coalescence within the column or flooding at the air injection point. These slugs can be as large as the diameter of the column and lift everything above them as they rise to the top of the column. This greatly disrupts the column and can result in decreased concentrate grades and/or recoveries.

**Bias.**—Tailings flow rate - feed flow rate. If this number is positive, then the system is said to have a positive bias. If this equals zero, then the system is said to have a neutral bias. If this number is negative, then the system is said to have a negative bias.

**Bubble generator.**—Any of a number of devices that makes use of techniques such as electrolysis, aspiration, pressure dissolution, or combinations of these techniques to produce bubbles.

**Bubble generator feed water.**—Clarified (0 pct solids) water used as feed to the bubble generator.

**Bubble injection tip.**—Usually a fine tip with a cross-sectional area on the order of 0.1 mm<sup>2</sup>, where the air-water mixture is released as bubbles into the column or flotation cell.

**Coalescence.**—A phenomenon observed where small bubbles recombine into larger bubbles. The occurrence of this phenomenon is apparently a function of the concentration of frother and bubbles present. The occurrence of coalescence in a flotation column is one of the factors that limits the maximum amount of air, which can be injected into the column.

**Coarse bubbles.**—Bubbles with average diameter of 1.5 mm or greater.

**Cleaning zone.**—The region in the column between the feed injection port and the top of the column.

**Collection zone.**—The region in the column between the feed injection port and the lower air injection port.

**Column flotation.**—The use of a tall containment area having either a square or circular cross section as a cell in which to perform a flotation separation. In flotation columns, no mechanical impellers are used for agitation and suspension of solids. Instead, slurry gravity feeds counter-current to bubbles rising in the column.

**Column mineral profile.**—This is a graph of mineral grades obtained as a result of samples drawn using the column sample valves plotted against the vertical location at which the sample was taken. This provides a profile of the mineral concentration within the column and is a key

to understanding the behavior of flotation columns and the flotation response of different mineral systems.

**Contact media.**—Material such as glass beads or pieces of plastic placed in the bubble generator chamber to improve the contact and mixing of air and water.

**Direct photographic bubble size measurement.**—Measurement of bubble diameters by photographing bubbles against a microgrid of known spacing.

**Electrolytic bubble generation.**—Use of electric current passed through a solution to break water down into gaseous hydrogen and oxygen.

**Feed conditioner.**—A continuously stirred tank in which reagents are added to the slurry and allowed to condition for a prescribed amount of time before being fed into the flotation column.

**Feed injection point.**—The vertical location on the column at which the conditioned slurry is fed into the column.

**Fine bubbles.**—Bubbles with an average diameter of between 0.2 and 1.5 mm.

**Flooding.**—A phenomenon that occurs when the air injection flow rate exceeds a certain limit. During flooding, large slugs of air are injected into the column rather than discrete bubbles. Flooding occurs at the air injection point, while coalescence may occur at any point within the flotation column.

**Fritted glass.**—A piece of glass that has been processed so it is porous. This makes it possible to pass air or water through it as long as the pores do not become plugged off. Pore size may vary from a few microns to a hundred microns depending on the type of frit produced.

**Froth height.**—The distance between the pulp-froth interface and the overflow weir. This represents the vertical depth of the froth bed.

**Froth phase.**—The predominantly air phase within the column containing lesser amounts of solids and water.

**Gravity feeding.**—The gravity used to transport slurry from a high point to a lower point through a pipe or tubing. This type of feeding may be controlled by use of a valve to restrict the flow of material at some point between the source and the destination point.

**Hydrostatic manometer.**—This is a small diameter vertical tube connected to the main body of the column. The water level in the manometer is an indication of the pulp level (not including the air) inside the column. By using two such manometers; one located towards the bottom, and one located towards the top of the column, a quantitative measure of the air holdup within the column may be obtained as: the difference in water levels between each manometer tube divided by the vertical distance between the points where each manometer connects to the column.

**Mechanical air induction.**—Use of mechanical means such as a pump to aspirate or aerate water physically, thus producing bubbles (such as in a conventional flotation cell).

**Micro bubbles.**—Bubbles with an average diameter of less than 0.2 mm.

**Microgrid.**—A photoreduced series of vertical and horizontal lines forming a grid with a known spacing between lines in each direction. At present, a spacing of 0.2 mm is used for fine-bubble measurement at a Bureau facility.

**Overflow weir.**—The sloped bottom launder attached to the top of the column to catch concentrate as it overflows the top of the column.

**Piston pump.**—A pump that operates by having two or more pistons with alternating strokes, thereby compressing the fluid in stages. This type of pump can produce very high pressures; however, water flows in a discontinuous pulsing action rather than a smooth continuous flow.

**Plug-flow.**—Plug flow is a condition in which no appreciable mixing takes place in the direction of flow. This is the case for small diameter columns. Under this condition, average retention times can be accurately assessed based only on flow rates in and out of the column. In large diameter columns, a significant amount of axial mixing takes place and plug-flow conditions do not exist. Therefore, scale-up factors must be used to determine the size of column needed to provide adequate retention time for the mineral in question. Plug flow is the opposite to complete mixing.

**Pressure dissolution.**—The use of pressure to chemically dissolve air or other gases in water according to Henry's Law. (Similar to carbonation of soda pop). This technique is used in waste water treatment plants and is known as dissolved air flotation (DAF).

**Pulp phase.**—The predominantly liquid phase within the column in which the solids are suspended.

**Rinse water.**—Water used for cleaning out the column at the end of or in-between tests.

**Sample valves.**—Solenoid operated valves that have a plunger-type action and are normally closed. These open when a switch is activated and draw back the plunger long enough to gather the desired sample.

**Scale-up factors.**—There are usually a set of numbers or semiempirical relationships, which provide correction factors for sizing of equipment for large-scale production or testwork based on small-scale bench test results.

**Spacer.**—An object such as a piece of plastic used to provide a gap between two areas such as between the bubble generator contact media and the bottom of the bubble generator.

**Sparger.**—A device (usually made of a porous material such as fritted glass or perforated rubber) which is used to disperse air as bubbles in pulp or water.

**Superficial gas velocity.**—The volumetric airflow rate going into the column divided by the cross-sectional area of the column. This provides a normalized measure of the amount of air being used in the column and is also an indirect measure of the air holdup within the column.

**Tailings recirculation point.**—The vertical position on the column to which tailings are recirculated during a batch column flotation test.

**Tailings removal point.**—The vertical location on the column at which tailings are removed. This is usually at the base of the column.

**Tip velocity.**—The velocity achieved by the air-water mixture upon release into the flotation column. Velocities between 100 and 200 ft/s are typical.

**Turbine blade pump.**—A pump that uses a series of blades (similar to a turbine) to push water through, thus producing high pressures and consistent flows.

**Wash water.**—Water added through some type of sprayer or disperser to the froth phase in the column to help wash entrained gangue particles back into the pulp phase. This water may be added above the froth or from within the froth phase.

**Water recirculation-bypass valve.**—A valve that controls the flow of water through a line, which recirculates to the pump feed water holding tank. Bubble generator water pressure and flow rate can be increased or decreased by adjusting this valve.

## APPENDIX B.—COLUMN FLOTATION PARAMETER TESTING

By R. A. Ynchausti,<sup>1</sup> D. G. Foot, Jr.,<sup>2</sup> and J. D. McKay<sup>3</sup>

The batch column flotation results presented here were previously published (14)<sup>4</sup>. These data, however, were important in establishing the conditions for optimum continuous column flotation of the Fish Creek fluorite ore. Batch column flotation tests were conducted on column length, feed injection location, tailing recirculation, froth depth, wash water additions, and particle size fractions.

### COLUMN LENGTH

Since the inception of flotation columns in the early 1960's, column length has been a concern to commercial mineral processing plants anticipating installation and operation of flotation columns. A column flotation cell is free from violent agitation. Feed and tailings slurry flow rates and particle settling rates affect the retention time of the particles in the column.

The collection zone has its upper boundary at the feed injection port and extends downward to the base of the column. This zone must have sufficient length to provide adequate retention time for the settling particles to attach to the rising bubbles. Column length design theory is based on this concept. Additional column length must be included for the upper three column zones as prescribed by the particular mineral system needs. Most work backing this theory has been performed on copper-molybdenum separations.

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<sup>2</sup>Supervisory chemical engineer.

<sup>3</sup>Chemical engineer.

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<sup>4</sup>Italic numbers in parentheses refer to items in the list of references preceding the appendixes.

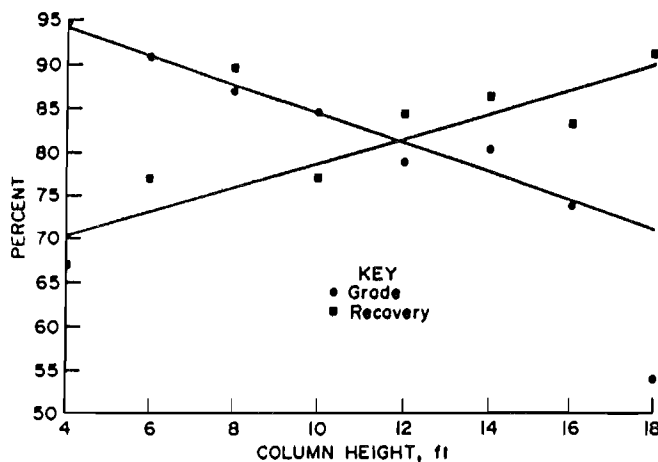


Figure B-1.—Effect of column length on fluorite grades and recoveries.

The effect of column length variations on the fluorite ore was studied by shortening the column, while maintaining a constant ratio of each column zone within physical limitations of the equipment and observing grade and recovery fluctuations. As the column flotation cell was shortened, recoveries of fluorite decreased (fig. B-1). Retention time was calculated for plug flow conditions based on the collection zone volume and tailings flow rate. Fluorite recovery decreased because particle retention time was not sufficient as the collection zone was shortened by decreasing the column length.

Fluorite grades increased with decreasing column length because only the particles with sufficient hydrophobicity to achieve bubble attachment were reported to the concentrate stream. As the column was shortened, the particle retention time decreased causing smaller fractions of the more liberated fluorite to be collected, while also reducing the amount of gangue that was either entrained or collected to the froth (fig. B-2).

### FEED INJECTION LOCATION

Tests were conducted to determine the effect of the vertical location of the feed slurry injection port on fluorite grade and recovery.

Fluorite recovery gradually decreased as the feed injection port was moved closer to the base of the column (fig. B-3). In essence, feed injection location is directly linked to particle retention time in the collection zone of the column. As the feed injection location was moved towards the base of the column, the length of the collection zone decreased, reducing the particle retention time, and decreased fluorite recovery resulted (fig. B-4).

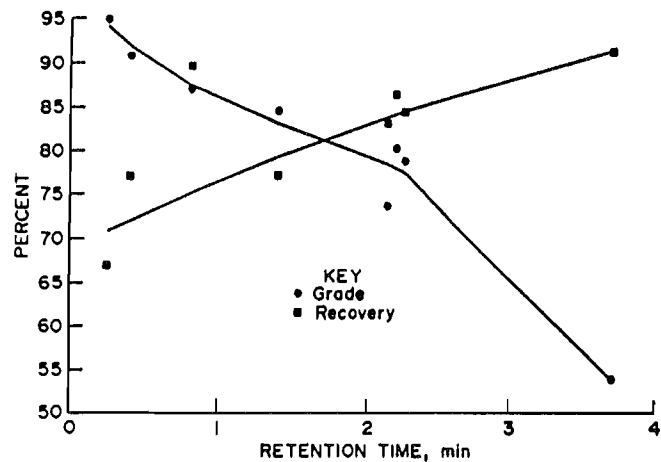


Figure B-2.—Effect of retention time on fluorite grades and recoveries with varying column length.

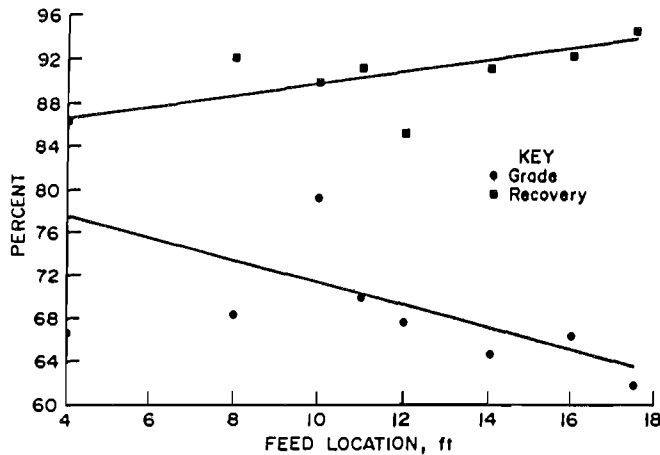


Figure B-3.—Variation of fluorite grades and recoveries with feed injection location.

Fluorite concentrate grades increased as feed injection location approached the base of the column. Lowering the vertical position of the feed slurry injection port is equivalent to shortening the length of the collection zone. This accounts for the similar response of fluorite recovery and grade to variations in either column length or feed slurry injection location. The increased fluorite grade observed as feed injection location moved towards the base of the column were due to the added concentration effects of the upper three column zones. Unlike the column length variation tests, the upper two zones remained at constant length, while the pulp phase cleaning zone increased in length as the feed injection location approached the column base.

#### TAILINGS RECIRCULATION

Fluorite grade and recovery variations were investigated as a portion of the tailings stream was recirculated at different rates during column flotation. The tailings recirculation flow rates were converted to superficial pulp velocities based on plug flow conditions in the collection zone volume. The resultant trend is given in figure B-5.

Fluorite recovery suffered and the grade was enhanced as the recirculation velocity was increased. Since short circuiting of some of the feed slurry, caused by axial mixing, increases as recirculation velocity increases, these trends were expected. Furthermore, trending of data from this investigation versus superficial liquid velocity showed no correlation, therefore, the reduced recoveries and increased grades were concluded to result from the decrease in the particle retention time distribution. Under these conditions, only those particles with the strongest adsorption energies have sufficient time to attach to the rising bubbles.

These results show that the collection zone must be lengthened to provide sufficient particle retention time as

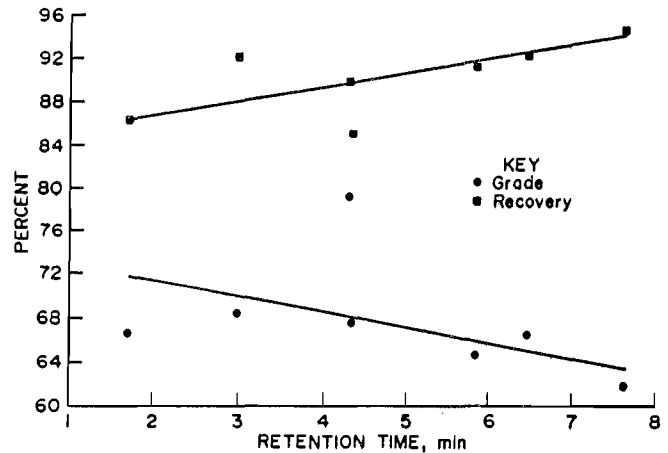


Figure B-4.—Effect of retention time on fluorite grades and recoveries with varying feed injection location.

the particle retention time distribution is broadened and to compensate for increase in pulp mixing.

#### FROTH DEPTH

A series of tests were conducted on the fluorite ore to determine the effect of froth depth on mineral grades and recoveries. Froth depth had a discernible effect on fluorite concentrate grades; as froth depth increased, fluorite grades increased (fig. B-6). Fluorite upgrading occurred in the froth phase cleaning and pulp-froth interfacial zones. The froth phase is more efficient than the pulp phase in upgrading the concentrate. At greater froth depths, more fluorite cleaning took place and higher grade concentrates were obtained.

Fluorite recovery data were scattered. No adequate trend could relate the recovery data with greater accuracy than a line with a slope of nearly zero. Although the source of these fluctuations could not be identified, no direct connection with froth depth variations could be made. It was concluded that froth depth had no primary correlation with fluorite recovery.

Since froth depth enhanced fluorite grades without hindering recoveries, froth depth should be maintained at as great a depth as possible, while maintaining sufficient column length for the collection zones to provide the necessary particle retention time to maintain mineral recoveries.

#### WASH WATER ADDITIONS

The principal reason for using wash water in column flotation systems is to increase the grade of the recovered concentrate by displacing entrained hydrophilic (gangue) particles that report to the froth phase. Wash water additions have also been observed to aid in stabilizing the froth bed. Tests were performed to determine the effect of changes in wash water addition rates on column

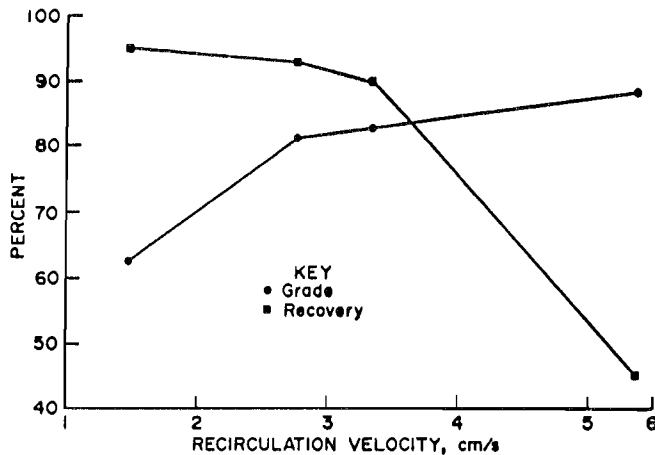


Figure B-5.—Effect of tallings recirculation on fluorite grades and recoveries.

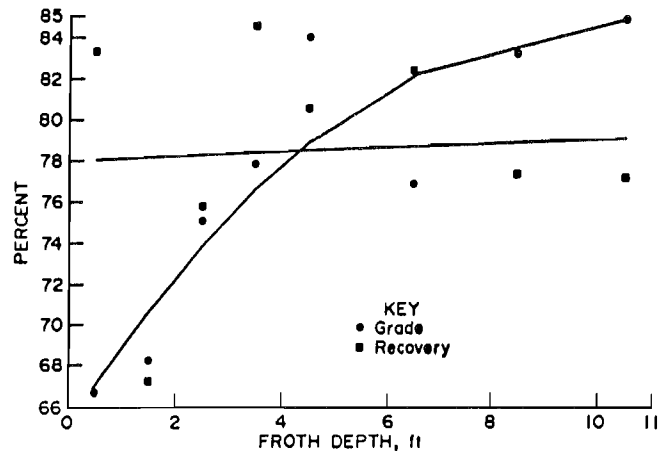


Figure B-6.—Effect of froth depth on fluorite grades and recoveries.

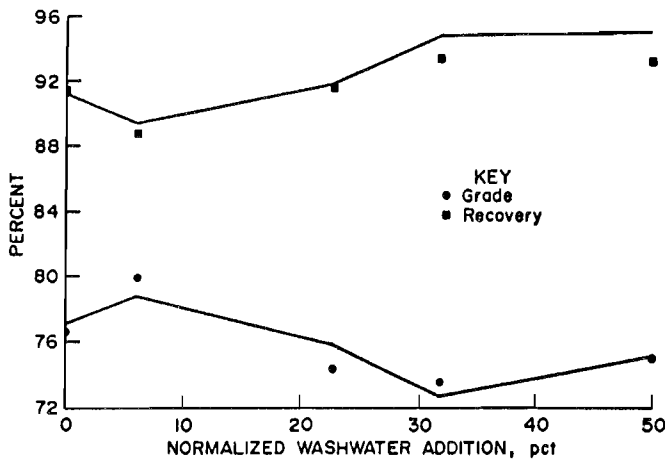


Figure B-7.—Effect of column wash water on fluorite grades and recoveries.

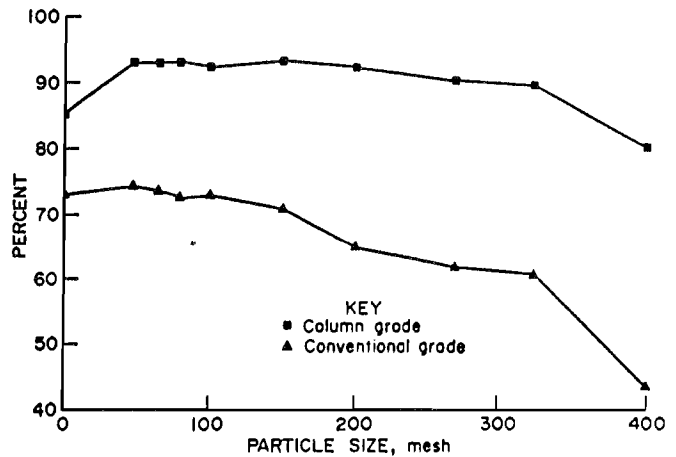


Figure B-8.—Comparison of fluorite grades by particle sizes for column and conventional flotation.

flotation grades and recoveries of the Fish Creek fluorite ore. Wash water was introduced through a spray nozzle located 1 in above the top of the column. The addition rates were normalized by dividing by the volumetric feed slurry flow rates to the column.

Wash water additions affected fluorite grade and recovery during column flotation in a complex manner (fig. B-7). Increasing wash water additions from 0 to 6 pct of the volumetric feed slurry flow rate increased fluorite grade, but decreased recovery. Increasing wash water additions from 6 pct to approximately 35 pct improved fluorite recovery and decreased fluorite grade. Above 35 pct wash water addition, fluorite grade again increased, while recovery remained approximately constant.

The complex effect of wash water additions on fluorite flotation in the column may be attributed to the twofold

nature of column wash water in removing gangue material reporting to the froth, while fluidizing the froth bed to prevent mineral overloading. As detailed in figure B-7, the optimum wash water addition rate was 6 pct. However, wash water flow rates in excess of 40 pct produced grades and recoveries that approached those at 6 pct. These additions may not be feasible due to increased water consumption, system dilution, and downstream materials handling problems.

## PARTICLE SIZE FRACTIONS

To quantify the separation efficiency of column flotation compared with conventional flotation, particle size distribution analyses were performed on products obtained from each method. The study was conducted by extracting a

portion of the conditioned column feed slurry and sending it to a conventional batch flotation process. The column and conventional flotation products were sized using Tyler 48-, 65-, 80-, 100-, 150-, 200-, 270-, 325-, and 400-mesh screens.

Column rougher flotation produced substantially higher grade concentrates than did conventional flotation of the fluorite ore (fig. B-8). Column flotation fluorite grades were greater than those of conventional flotation for all size fractions.

Conventional flotation recoveries of fluorite were slightly higher than those of column flotation, except particles between 65 and 100 mesh (fig. B-9). Although conventional flotation provides a small increase in fluorite recovery for most particle size fractions, column flotation still holds the advantage because conventional fluorite recoveries would fall well below column fluorite recoveries in cleaning stages that would be necessary to meet the fluorite grades achieved in column flotation.

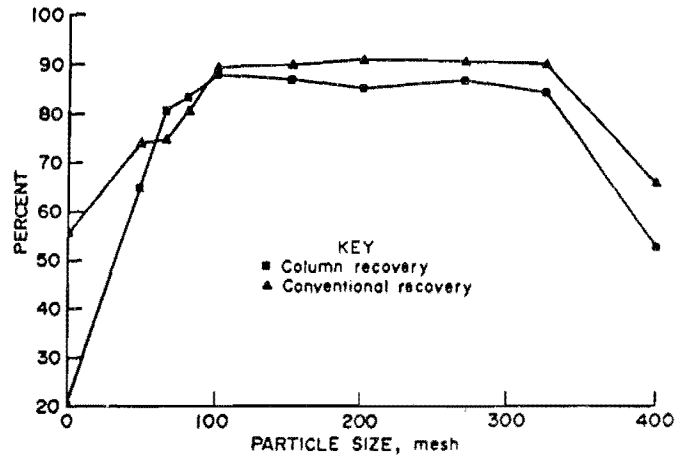


Figure B-9.—Comparison of fluorite recoveries by particle sizes for column and conventional flotation.

## APPENDIX C.—FLUORITE COLUMN FLOTATION ECONOMICS<sup>1</sup>

Capital and operating costs are presented for a column flotation plant designed to process 2,000 st/d of ore from the Fish Creek deposit and to recover about 280 st/d of acid-grade fluorspar product. Assuming 330 days of operation per year, annual production is equal to about 92,400 st of fluorspar.

### CAPITAL COSTS

The capital cost estimate is of the general type called a study estimate by Weaver (21).<sup>2</sup> This type of estimate,

<sup>1</sup>Cost estimates were made by Thomas A. Phillips, Staff Engineer, U.S. Bureau of Mines, Washington DC.

<sup>2</sup>Italic numbers in parentheses refer to items in the list of references preceding the appendixes.

**Table C-1.—Estimated capital cost,<sup>1</sup>  
fluorspar column flotation**

Fixed capital:	
Feed preparation section . . . . .	\$8,221,200
Flotation section . . . . .	2,156,800
Product handling section . . . . .	2,831,100
Water recovery section . . . . .	<u>6,443,000</u>
Subtotal . . . . .	19,652,100
Plant facilities, 10 pct of above subtotal . .	1,965,200
Plant utilities, 12 pct of above subtotal . .	<u>2,358,300</u>
Total plant cost . . . . .	23,975,600
Tailings pond . . . . .	3,881,700
Land cost . . . . .	<u>0</u>
Subtotal . . . . .	27,857,300
Interest during construction period . . . . .	<u>1,957,300</u>
Fixed capital cost . . . . .	<u>29,814,600</u>
Working capital:	
Raw material and supplies . . . . .	270,100
Product and in-process inventory . . . . .	878,900
Accounts receivable . . . . .	878,900
Available cash . . . . .	<u>529,900</u>
Working capital cost . . . . .	2,557,800
Capitalized startup costs . . . . .	<u>298,100</u>
Subtotal . . . . .	<u>2,855,900</u>
Total capital cost . . . . .	<u>32,670,500</u>

<sup>1</sup>Basis: M & S equipment cost index of 897.0.

prepared from a flowsheet and a minimum of equipment data, can be expected to be within 30 pct of the actual cost for the plant described. The estimated fixed capital cost on a third quarter 1989 basis (Marshall and Swift (M & S) index of 897.0) for the column cell flotation process is approximately \$30 million, as shown in table C-1.

Sizes of major items of equipment are shown in table C-2. Equipment costs for the process are based on cost-capacity data and manufacturer's cost quotations. Equipment costs for each section are shown in tables C-3 through C-6. Cost data are brought up to date by the use of inflation indexes. Mild steel is used as the material of construction throughout most of the plant. The main exceptions are where slurry handling will cause abrasion in which case rubber linings are used.

**Table C-2.—Major items of equipment, fluorspar column flotation**

Section and Item	Quantity	Unit size
Feed preparation:		
Jaw crusher . . . . .	1	30 by 42 in
Standard-head cone crusher . .	1	5.5 ft
Short-head cone crushers . . . .	2	7 ft
Ball mill . . . . .	1	6,015 st/d
Flotation:		
Conditioning tank . . . . .	1	3,882 gal
Do . . . . .	1	3,902 gal
Do . . . . .	1	4,704 gal
Rougher flotation columns . . . .	2	6 ft diam by 35 ft high
Cleaner flotation column . . . . .	1	Do.
Reciprocating compressor . . . .	1	300 hp
Product handling:		
Rotary-vacuum drum filter . . . .	1	181 ft <sup>2</sup>
Rotary dryers . . . . .	2	6 ft diam by 60 ft high
Water recovery:		
Thickeners . . . . .	3	191 ft diam
Recycle water tanks . . . . .	6	87,469 gal



**Table C-3.—Equipment cost summary, feed preparation section, and fluorspar column flotation**

Item	Equipment <sup>1</sup>	Labor	Total
Apron feeder	\$25,400	\$3,800	\$29,200
Jaw crusher	112,300	15,700	128,000
Belt conveyor	17,100	3,900	21,000
Vibrating screen	10,800	1,300	12,100
Belt conveyor	15,400	3,400	18,800
Do	10,200	2,600	12,800
Apron feeder	22,000	3,300	25,300
Standard-head cone crusher	320,800	38,500	359,300
Belt conveyor	8,600	2,100	10,700
Do	50,100	12,200	62,300
Hopper	1,100	200	1,300
Apron feeder	18,800	2,800	21,600
Belt conveyor	51,100	12,400	63,500
Storage bin	23,000	2,400	25,400
Apron feeder	18,800	2,800	21,600
Vibrating screen	26,500	3,200	29,700
Belt conveyor	8,700	2,100	10,800
Hoppers	5,000	700	5,700
Belt feeders	19,300	2,900	22,200
Short-head cone crushers	976,800	80,200	1,057,000
Belt conveyor	9,500	2,100	11,600
Do	22,800	5,300	28,100
Apron feeder	14,300	2,100	16,400
Belt conveyor	25,300	2,800	28,100
Vibrating screens	118,300	14,200	132,500
Sump	3,100	1,000	4,100
Pumps	17,600	1,000	18,600
Ball mill	943,300	56,600	999,900
Slurry tank	6,000	1,600	7,600
Pumps	21,400	1,200	22,600
Bag dust collector	57,100	2,700	59,800
Total	<u>2,980,500</u>	<u>287,100</u>	<u>3,267,600</u>
Unloading hopper <sup>2</sup>			21,400
Silo <sup>2</sup>			219,400
Front-end loader			81,500
Total equipment cost × factor indicated:			
Foundations, × 0.126			375,800
Buildings, × 0.043			128,200
Structures, × 0.070			208,600
Instrumentation, × 0.050			149,000
Electrical, × 0.060			179,200
Piping, × 0.300			894,200
Painting, × 0.020			59,600
Miscellaneous, × 0.100			298,100
Total			<u>2,292,700</u>
Total direct cost			5,882,600
Field indirect, 10.0 pct of total direct cost			588,300
Total construction cost			6,470,900
Engineering, 5.0 pct of total construction cost			323,500
Administration and overhead, 5.0 pct of total construction cost			323,500
Subtotal			<u>7,117,900</u>
Contingency, 10.0 pct of above subtotal			711,800
Subtotal			<u>7,829,700</u>
Contractor's fee, 5.0 pct of above subtotal			391,500
Section cost			<u>8,221,200</u>

<sup>1</sup>Basis: M & S equipment cost index of 897.0.

<sup>2</sup>Installed cost.

**Table C-4.—Equipment cost summary, flotation section, and fluorspar column flotation**

Item	Equipment <sup>1</sup>	Labor	Total
Sodium carbonate storage bin	\$600	\$200	\$800
Screw feeder	2,700	400	3,100
Mixing tank	1,300	500	1,800
Pump	1,100	200	1,300
Conditioning tank	4,300	1,200	5,500
Sodium silicate storage bin	700	100	800
Screw feeder	2,700	400	3,100
Mixing tank	1,000	500	1,500
Pump	1,000	200	1,200
Conditioning tank	4,400	1,200	5,600
Pump	1,600	200	1,800
Oleic acid storage tank	400	100	500
Pump	1,600	200	1,800
Mixing tank	1,000	500	1,500
Pump	1,000	200	1,200
Conditioning tank	4,700	1,300	6,000
Pump	2,100	200	2,300
Pumps	27,700	1,400	29,100
Rougher flotation columns	143,100	6,900	150,000
Sump	3,800	1,100	4,900
Pumps	25,800	1,300	27,100
Sump	1,600	600	2,200
Pumps	11,900	300	12,200
Cleaner flotation column	71,600	3,500	75,100
Reciprocating compressor	99,800	1,600	101,400
Pumps	13,700	800	14,500
Sump	600	200	800
Pumps	11,900	300	12,200
Total	<u>443,700</u>	<u>25,600</u>	<u>469,300</u>
Forklift truck			44,200
Total equipment cost × factor indicated:			
Foundations, × 0.282			125,000
Buildings, × 1.150			510,300
Structures, × 0.070			31,100
Instrumentation, × 0.070			31,100
Electrical, × 0.129			57,100
Piping, × 0.500			221,900
Painting, × 0.020			8,900
Miscellaneous, × 0.100			44,400
Total			<u>1,029,800</u>
Total direct cost			1,543,300
Field indirect, 10.0 pct of total direct cost			154,300
Total construction cost			1,697,600
Engineering, 5.0 pct of total construction cost			84,900
Administration and overhead, 5.0 pct of total construction cost			84,900
Subtotal			<u>1,867,400</u>
Contingency, 10.0 pct of above subtotal			186,700
Subtotal			<u>2,054,100</u>
Contractor's fee, 5.0 pct of above subtotal			102,700
Section cost			<u>2,156,800</u>

<sup>1</sup>Basis: M & S equipment cost index of 897.0.

**Table C-5.—Equipment cost summary, product handling section, and fluorspar column flotation**

Item	Equipment <sup>1</sup>	Labor	Total
Horizontal-belt filter . . . . .	\$91,700	\$9,200	\$100,900
Belt conveyor . . . . .	38,600	5,500	44,100
Sump . . . . .	400	100	500
Pump . . . . .	1,300	200	1,500
Hoppers . . . . .	2,000	300	2,300
Screw feeders . . . . .	5,300	800	6,100
Rotary dryers . . . . .	454,800	136,500	591,300
Belt conveyor . . . . .	56,200	8,500	64,700
Storage bins . . . . .	179,200	15,700	194,900
Vibrating feeder . . . . .	2,200	300	2,500
Cyclone dust collectors . . . . .	22,100	2,200	24,300
Bag dust collector . . . . .	42,300	2,000	44,300
Total . . . . .	896,100	181,300	1,077,400
Total equipment cost × factor indicated:			
Foundations, × 0.272 . . . . .			243,700
Buildings, × 0.120 . . . . .			107,500
Structures, × 0.070 . . . . .			62,700
Instrumentation, × 0.050 . . . . .			44,800
Electrical, × 0.027 . . . . .			23,800
Piping, × 0.400 . . . . .			358,400
Painting, × 0.020 . . . . .			17,900
Miscellaneous, × 0.100 . . . . .			89,600
Total . . . . .			948,400
Total direct cost . . . . .			2,025,800
Field indirect, 10.0 pct of total direct cost . . . . .			202,600
Total construction cost . . . . .			2,228,400
Engineering, 5.0 pct of total construction cost . . . . .			111,400
Administration and overhead, 5.0 pct of total construction cost . . . . .			111,400
Subtotal . . . . .			2,451,200
Contingency, 10.0 pct of above subtotal . . . . .			245,100
Subtotal . . . . .			2,696,300
Contractor's fee, 5.0 pct of above subtotal . . . . .			134,800
Section cost . . . . .			2,831,100

<sup>1</sup>Basis: M & S equipment cost index of 897.0.

Factors for piping, etc., except the foundations, buildings, and electrical factors are assigned to each section, using as a basis the effect fluids, solids, or a combination of fluids and solids may have on the process equipment. Foundation costs are estimated for each piece of equipment, and a factor is calculated from these costs for each section. Building costs are estimated from equipment space requirements, and factors calculated from these costs. The electrical factors are based on motor horsepower requirements for each section. A factor of 10 pct, called miscellaneous, is added to each section to cover minor equipment and construction costs that are not shown with the equipment listed.

For each section, the field indirect cost, which covers field supervision, inspection, temporary construction, equipment rental, and payroll overhead, is estimated at 10 pct of the direct cost. Engineering cost is estimated at 5 pct, and administration and overhead cost is estimated at 5 pct of the construction cost. A contingency allowance of 10 pct and a contractor's fee of 5 pct are included in the section cost.

**Table C-6.—Equipment cost summary, water recovery section, and fluorspar column flotation**

Item	Equipment <sup>1</sup>	Labor	Total
Thickeners . . . . .	\$1,523,400	\$238,000	\$1,761,400
Pumps . . . . .	25,800	1,300	27,100
Pump . . . . .	3,500	600	4,100
Recycle water tanks . . . . .	193,700	71,400	265,100
Pump . . . . .	6,400	700	7,100
Total . . . . .	1,752,800	312,000	2,064,800
Total equipment cost × factor indicated:			
Foundations, × 0.718 . . . . .			1,258,400
Structures, × 0.060 . . . . .			105,200
Instrumentation, × 0.050 . . . . .			87,600
Electrical, × 0.014 . . . . .			25,000
Piping, × 0.500 . . . . .			876,400
Painting, × 0.010 . . . . .			17,500
Miscellaneous, × 0.100 . . . . .			175,300
Total . . . . .			2,545,400
Total direct cost . . . . .			4,610,200
Field indirect, 10.0 pct of total direct cost . . . . .			461,000
Total construction cost . . . . .			5,071,200
Engineering, 5.0 pct of total construction cost . . . . .			253,600
Administration and overhead, 5.0 pct of total construction cost . . . . .			253,600
Subtotal . . . . .			5,578,400
Contingency, 10.0 pct of above subtotal . . . . .			557,800
Subtotal . . . . .			6,136,200
Contractor's fee, 5.0 pct of above subtotal . . . . .			306,800
Section cost . . . . .			6,443,000

<sup>1</sup>Basis: M & S equipment cost index of 897.0.

The costs of plant facilities and plant utilities are estimated as 10 and 12 pct, respectively of the total process section costs and include the same field indirect costs, engineering, administration and overhead, contingency allowance, and contractor's fee as are included in the section costs. Included under plant facilities are the costs of material and labor for auxiliary buildings such as offices, shops, laboratories, and cafeterias, and the cost of non-process equipment such as office furniture, together with safety, shop, and laboratory equipment. Also included are labor and material costs for site preparation such as site clearing, grading, drainage, roads, and fences. The cost of water, power, and steam distribution systems are included under plant utilities.

The cost for interest on the capital borrowed for construction is included as interest during construction. A simple annual interest rate of 8 pct has been assumed. An expenditure schedule, based on a construction period of 24 months, is used to calculate the interest to be paid over the construction period. Land investment is not included

in this estimate. Cost for the plant owner's supervision is not included in the capital cost of the proposed plants.

Working capital is defined as the funds in addition to fixed capital, land investment, and startup costs that must be provided to operate the plant. Working capital, also shown in table C-1, is estimated from the following items: (1) Raw material and supplies inventory (cost of raw material and operating supplies for 30 days), (2) product and in-process inventory (total operating cost for 30 days), (3) accounts receivable (total operating cost for 30 days), and (4) available cash (direct expenses for 30 days).

Capitalized startup costs are estimated as 1 pct of the fixed capital, which is shown in table C-1.

### OPERATING COSTS

The estimated operating costs are based on 330 d/yr operation over the life of the plant. This allows 35 d/yr downtime for inspection, maintenance, and unscheduled interruptions. The operating costs are divided into direct, indirect, and fixed costs.

Direct costs include raw materials, utilities, direct labor, plant maintenance, payroll overhead, and operating supplies. The raw material costs include an extra 10 pct charge to cover transportation costs to the plant site. Electricity, water, and natural gas are purchased utilities. Raw material and utility requirements per short ton of fluorspar are shown in table C-7.

Table C-7.—Raw material and utility requirements, column flotation

	Quantity per short ton fluorspar concentrate
<b>Raw materials:</b>	
Fluorspar ore . . . . . st ..	7.143
Sodium carbonate . . . . . lb ..	21.429
Sodium silicate . . . . . lb ..	14.286
Oleic acid . . . . . lb ..	8.143
Hydrochloric acid . . . . . lb ..	0.714
Replacement balls for grinding . . . lb ..	2.086
<b>Utilities:</b>	
Electric power . . . . . kW•h ..	93.257
Process water . . . . . Mgal ..	0.903
Raw water . . . . . Mgal ..	0.192
Natural gas . . . . . MMBtu ..	0.768

Direct labor costs are estimated on the basis of assigning 4.2 employees to each position that operates 24 h/d, 7 days per week, and 1.4 employees to each position that operates 8 h/d, 7 days per week. The cost of labor supervision is estimated as 15 pct of the labor cost.

Plant maintenance is separately estimated for each piece of equipment and for the buildings, electrical system, piping, plant utility distribution systems, and plant facilities.

Payroll overhead, estimated as 35 pct of direct labor and maintenance labor, includes vacation, sick leave, social security, and fringe benefits. The cost of operating supplies is estimated as 20 pct of the cost of plant maintenance.

Indirect costs are estimated as 40 pct of the direct labor and maintenance costs. The indirect costs include the expenses of control laboratories, accounting, plant protection and safety, plant administration, marketing, and company overhead. Research and overall company administrative costs outside the plant are not included.

Fixed costs include the cost of taxes (excluding income taxes), insurance, and depreciation. The annual costs of both taxes and insurance are each estimated as 1 pct of the plant construction cost. Depreciation is based on a straight-line, 10-yr period.

The estimated annual operating cost for the column flotation process (table C-8), is about \$10.7 MM or \$116/st of fluorspar, based on an annual production of 92,400 st of fluorspar.

Table C-8.—Estimated annual operating cost, fluorspar column flotation

	Annual cost	Cost per short ton fluorspar concentrate
<b>Direct cost:</b>		
<b>Raw materials:</b>		
Fluorspar ore at \$3.00/st . . . . .	\$1,980,000	\$21.43
Sodium carbonate at \$0.07/lb . . . .	139,600	1.51
Sodium silicate at \$0.31/lb . . . . .	409,200	4.43
Oleic acid at \$0.65/lb . . . . .	489,100	5.29
Hydrochloric acid at \$0.09/lb . . . .	5,900	0.06
Replacement balls for grinding at \$0.27/lb . . . . .	52,000	0.56
Total . . . . .	<u>3,075,800</u>	<u>33.28</u>
<b>Utilities:</b>		
Electric power at \$0.072/kW•h . . . .	620,400	6.71
Process water at \$0.25/Mgal . . . . .	20,900	0.23
Raw water at \$0.05/Mgal . . . . .	900	0.01
Natural gas at \$4.20/MMBtu . . . . .	298,000	3.23
Total . . . . .	<u>940,200</u>	<u>10.18</u>
<b>Direct labor:</b>		
Labor at \$11.50/h . . . . .	621,900	6.73
Supervision at 15 pct of labor . . . .	93,300	1.01
Total . . . . .	<u>715,200</u>	<u>7.74</u>
<b>Plant maintenance:</b>		
Labor . . . . .	478,800	5.18
Supervision, 20 pct of maintenance labor . . . . .	95,800	1.04
Materials . . . . .	478,800	5.18
Total . . . . .	<u>1,053,400</u>	<u>11.40</u>
<b>Payroll overhead, 35 pct of above payroll . . . . .</b>		
Operating supplies, 20 pct of plant maintenance . . . . .	210,700	2.28
Total direct cost . . . . .	<u>6,446,700</u>	<u>69.77</u>
Indirect cost, 40 pct of direct labor and maintenance . . . . .	707,400	7.66
<b>Fixed cost:</b>		
Taxes, 1.0 pct of total plant cost . . . .	278,600	3.02
Insurance, 1.0 pct of total plant cost . .	278,600	3.02
Depreciation, 10-yr life . . . . .	<u>2,981,500</u>	<u>32.27</u>
Total operating cost . . . . .	<u>10,692,800</u>	<u>115.74</u>

### PROFITABILITY ANALYSIS

Column flotation recovers about 95 pct of the fluorspar in the feed to produce 92,400 st of acid grade (97 pct  $\text{CaF}_2$ ) product. The published price of this grade fluorspar produced in the U.S. is \$168 to \$173/st (Nov. 1989). With the estimated operating cost of \$116/st, this process was expected to yield a profit of about \$55/st. The interest rate of return on investment after taxes (estimated at 40 pct) is calculated to be about 12 pct over the 10-yr depreciation period, which is less than the 15 pct normally deemed necessary for an investment. To obtain an interest rate of return of 15 pct, a selling price of about \$185/st of product would be needed.

### BYPRODUCT RECOVERY

The Fish Creek deposit contains beryllium and mica, which could be potentially recovered as byproducts from the process tailings. Current markets for these commodities are small and, therefore, were not considered in this economic evaluation. Additional process development would be required to develop byproduct recovery steps. This is especially true for the beryllium recovery where the flotation studies resulted in a concentrate lower in beryllium than that being treated for metal recovery.