CIRCULAR 70

RECOVERY OF VALUABLE MINERALS FROM PEGMATITIC ORES

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NEW MEXICO INSTITUTE OF MINING AND TECHNOLOGY
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STATE BUREAU OF MINES AND MINERAL RESOURCES
Alvin J. Thompson, *Director*

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Abstract

The recovery of valuable minerals, such as beryl, spodumene, lepidolite, and microlite from pegmatitic ores has presented a challenge to mineral beneficiation engineers in the past. This difficult problem has been solved through a systematic study of the response of these minerals, individually and in combination with each other, to various collectors, pH, activators, and depressants. It has been shown that lepidolite and other micaceous minerals can be removed by a conventional amine float at pH .= 2. 5. Beryl, spodumene, and microlite are recovered with a sulfonate float at pH = 2. 7. Quartz and feldspars are depressed at this pH by sulfate ions introduced into the system from the sulfuric acid used for pH adjustment. Finally, the selective flotation among spodumene, beryl, and microlite is accomplished through the use of fluoride or hydrosulfide ions and gravity separation. Both the theoretical and practical aspects of the flotation procedures are discussed, and a workable flowsheet is presented for the economic exploitation of these difficult-to-treat ores.

Introduction

Granitic pegmatite deposits are the chief source of commercial feldspars, micas, lithium minerals, tantalum-columbium, beryl, and certain types of kaolin. The output from pegmatite mines in the United States is small compared to other mineral products in terms of bulk or value, and much of it comprises only minor metals and nonmetals. Nevertheless, pegmatite minerals play a vital part in our domestic industrial economy, particularly in the ceramic and electrical industries. Numerous special purpose uses also are important, even though they require small quantities of raw materials.

In recent years, realizing the potential importance of the pegmatite minerals, the U. S. Geological Survey, the United States Bureau of Mines, several state Bureaus of Mines, and a few private companies have carried on extensive programs of pegmatite studies in the New England and southeastern states, in the Black Hills area of South Dakota, in the Rocky Mountain region of Wyoming, Colorado, and New Mexico, and in various parts of Washington, Idaho, Utah, Nevada, Arizona, and California.

Nature has endowed New Mexico with several pegmatite-containing areas indicating fair potential for minerals such as micas, spodumene, lepidolite, beryl, and microlite. Most of these are located in the northern part of the state in Taos, Rio Arriba, Santa Fe, Mora, and San Miguel counties. Previous investigations *(see* References) have shown that potential reserves of micas, beryl, lithium minerals, and columbium-tantalum minerals are available in the above pegmatites, but very little active mining or exploration has taken place. This is due to (a) lack of demand for nonmetallic minerals, (b) remoteness from consumer markets, and (c) lack of a reliable concentration procedure to recover several of these valuable minerals economically from the pegmatitic ores.

Although some success has been achieved in separating micas, beryl, spodumene, or feldspar from their individual ores, selective separation of such minerals from a complex pegmatite containing all these minerals has not been achieved. Since the exploitation of some of these pegmatites in the northern part of New Mexico would improve the economy of this area, the current investigation concerning selective recovery of the pegmatitic minerals was undertaken with the hope of finding a solution to this important problem.

The basis of the current study has been the pertinent data collected by Fuerstenau and Bhappu (1963) and by Fuerstenau, Martin, and Bhappu (1963). Initial experiments were conducted on relatively pure samples of micas (muscovite, biotite, and schists), beryl, spodumene, lepidolite, microlite, quartz, and feldspars (microcline and albite). The results of these experiments were then applied to the selective flotation of the minerals from natural ores obtained from various pegmatites in New Mexico, Arizona, and Colorado.

Although micas (muscovite, biotite, and schist) constitute one of the major valuable minerals in the pegmatite, their concentration as a principal marketable product will not be considered in detail in the current investigation. The beneficiation of micas has been investigated thoroughly (Browning and McVay, 1963), and their concentrations do not present serious problems in practice. The micas will be treated here as gangue constituents of the three pegmatite ores investigated in detail, namely, low-grade beryl ore, lithium-tantalum ore, and spodumene-beryl ore.

ACKNOWLEDGMENTS

The authors wish to thank Mr. Alvin J. Thompson, director of the State Bureau of Mines and Mineral Resources, for helpful advice and encouragement through the investigation. Thanks are especially due Dr. Dexter H. Reynolds of the Bureau staff for the analyses of various flotation products and his helpful comments. The authors extend their gratitude to the owners and the managements of various pegmatitic deposits and mines for providing the high-purity mineral samples and natural ores for the experimental work.

Acknowledgment is made of the assistance of Mrs. Lois Devlin and Mrs. Helen Waxler in typing, Mr. William Arnold in drafting of figures, and Miss Teri Ray in editing the manuscript. All are of the Bureau staff.

GEOLOGICAL ASPECTS OF PEGMATITES

In the current investigation, close attention was given to the geological features of the pegmatitic ores investigated in order to help determine the treatment procedures for the recovery of valuable minerals. The salient geological and mineralogical features of the pegmatites have been adequately described by Jahns (1951), Just (1937), and Cameron et al. (1949). In this report, only those geological and mineralogical aspects having a direct bearing on the metallurgical problem will be discussed.

According to Jahns (1951), studies in all parts of the United States have shown that, despite numerous complexities of detail, most pegmatite bodies of commercial interest are rather regular in general structure. Plunging bodies are especially common, and many pegmatites are shaped in three dimensions more like laths or flattened cigars, with long axes that plunge gently to moderately. In general, pegmatites that are lithologically and structurally more complex contain rare minerals, as well as most of those with minable concentrations of feldspar, mica, beryl, spodumene, and other minerals. A general systematic arrangement of lithologic units in such pegmatites has long been recognized, and *bands, barrels, columns, layers, lenses, pipes, pods,* ribs, *shoots, streaks,* veins, and *zones* are terms commonly used by miners and referred to in geologic literature.

According to a recently proposed classification (Cameron et al.), the internal units of pegmatites comprise three fundamental types: (a) fracture fillings, (b) replacement bodies, and (c) zones. Typical pegmatite units illustrated by Jahns are shown in Figure 1. Such units range widely in size, shape, and texture. The smallest are tiny fracture-filling veinlets and the thin outermost zones of many pegmatite bodies. The largest are masses several hundred feet long and more than fifty feet in minimum dimension. Many units are easily distinguished and sharply marked from adjacent units, especially where they differ noticeably from them in composition or texture.

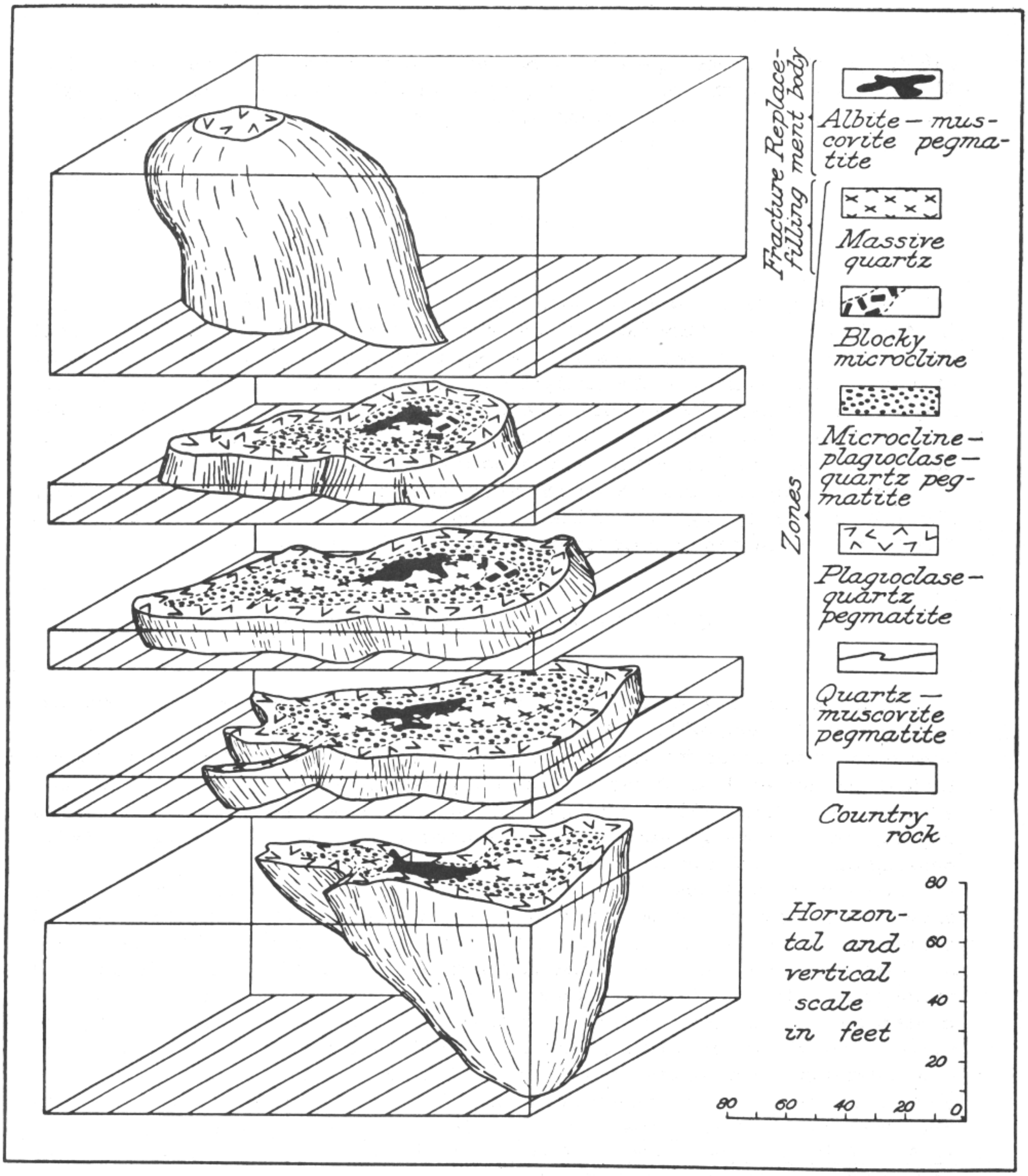

Figure I. Isometric block diagram of a typical steeply plunging podlike pegmatite body showing distribution of zones and other units at different levels (after Jahns)

The above-mentioned zones are important from a beneficiation viewpoint, since each zone may exhibit characteristic mineralization which may significantly influence the treatment procedure for recovery of valuable mineral or minerals. It is true that pegmatites, in general, characteristically exhibit extreme irregularity as to their component minerals. However, the deposits of economic importance investigated usually appear to show distinct zones with this characteristic mineralization. Because of their distinct nature, the zones are described as spod-lath, spotted rock, quartz-albite band, and so on, depending upon the mode of occurrence of the valuable and gangue minerals. These characteristics of zone will become clear as the mineralogical aspects of pegmatitic ores are discussed.

Mineralogy

The general succession of mineral crystallization in some of the pegmatite deposits investigated is as follows:

Zones	Minerals Present
1. Quartz	Quartz
2. Quartz -feldspar	Microcline-albite, quartz, muscovite, beryl, (most of the beryl found in this zone)
3. Feldspar (Li-Ta-rich) "Spotted rock"	Albite, quartz, spodumene, lepidolite, microlite, and tantalite
4. Feldspar-quartz "Spod-lath"	Quartz, spodumene, lepidolite, albite, microlite, tantalite, and beryl (some beryl found in this zone)

For purposes of beneficiation these zones can be conveniently grouped into four types of ores which might be encountered in practice:

Group 1. Mica ore, containing muscovite along with quartz, feldspars, and a minor amount of tantalum minerals;

Group 2. Low-grade beryl ore, comprising the first two zones and containing quartz, feldspars, mica, and beryl;

Group 3. Lithium-tantalum ore (spotted rock), comprising the third zone and containing feldspars, quartz, spodumene, lepidolite, and tantalum minerals;

Group 4. Spodumene-beryl ore, comprising the fourth zone and containing quartz, feldspars, spodumene, lepidolite, beryl, and tantalum minerals.

The beneficiation of Group 1 ores will not be included in this report.

Representative samples of the other three groups were obtained from New Mexico, Arizona, and Colorado. These samples were subjected to complete mineralogical examinations to determine the relative abundance and the liberation of valuable minerals, such as spodumene, beryl, lepidolite, microlite, from the gangue minerals and from each other. The desired information was collected through microscopic, spectrographic, X-ray, and chemical analyses on various sized fractions.

The results of the mineralogical examination of different types of ore samples are summarized below.

Low-grade Beryl Ore

The low-grade beryl ore samples were collected from the existing dumps of a well-known pegmatite deposit in New Mexico. The beryl content of these samples was about 0.5 per cent BeO. The major gangue minerals were mica-schist, feldspars (microcline and albite), quartz, fluorapatite, and various iron-bearing silicate minerals including garnet, pyroxene, and tourmaline. Such a material would represent mined ore and dumps from which most of the beryl would be recovered. If the overburden of schist were removed prior to the mining of fresh ore, the amount of mica-schist and iron-bearing silicates would be kept to a minimum.

Low-grade beryl ore samples from pegmatite deposits in Colorado, Arizona, and Canada were also tested to confirm these results.

Beryl /$Be_3Al2(SiO_3)6$; BeO = 12 to 13%; specific gravity 2.63 to 2. 80; hardness 7.5 to 8.07 in these ore samples is white, pale yellowish green, and pinkish in color (color shown by coarse grains only) and generally lacks crystal form. The microscopic examination revealed that although some free beryl is found in coarser sizes, the complete liberation of beryl occurs at minus 60 mesh, However, the economic point of liberation for flotation testing (the best concentration procedure applicable to this ore) was determined to be 48 mesh. Most of the quartz and feldspars were also free at this size.

It should be mentioned that beryl was the only valuable mineral present in these ores. Spectrographic analysis of various fractions failed to show any valuable elements besides beryl, germanium, and cesium. Germanium and cesium are known to be present in beryls from these pegmatites.

Lithium-Tantalum Ore (Spotted Rock)

The ore samples representing this zone of the pegmatite were obtained from deposits in Taos and Mora counties.

This type of ore contains, on a weight basis, about 7 per cent lepidolite, 20 per cent spodumene and 0.15 per cent microlite. The major gangue minerals are quartz, microcline, and albite. Such ores would constitute a major source of lepidolite, spodumene, and tantalum-rich microlite.

Spodumene /$LiAl(SiO_3)_2$; Li_2O = 6.0 to 7.0%; specific gravity 3.13 to 3.20; hardness 6. 5 to 7.0; breaks into small elongated laths/ in this type of ore occurs as disseminated small rounded crystals up to one inch in diameter, weathered and usually white to grayish white in color. Elongated crystals of spodumene are readily discernible under a microscope.

Lepidolite tLiK Al_t F2 (SiO3)3; Li2O = 1.9 to 4.6%; specific gravity 2.8 to 2, 9; hardness 2.5 to 4. 0; slightly magnetic; color light gray to pink and deep lilac/ occurs chiefly as scattered masses of small disseminated micaceous scales replacing microcline and showing all stages of replacement. The degree of replacement increases with increasing Li2O content. The purest lepidolite is very micaceous and lilac in color, while the less pure varieties are either light gray or pink and show a coarser texture.

Microlite /essentially Ca2Ta2O7; Ta2O5 = 60 to 80%; specific gravity 6. 0 to 6. 3; hardness 5.5/ in the ore occurs disseminated as yellowish to light and dark brown crystals in spodumene, cleavendite, and lepidolite. The yellowish variety is decidedly predominant and contains 70 to 80 per cent Ta2O5. Minor amounts of tantalite-columbite and hatchettolite are also found in this type of ore; although medium hard, microlite tends to be friable and there is always a danger of sliming it during crushing and grinding. For this reason, care should be taken to avoid overgrinding and sliming of microlite by stage crushing, screening, and classification.

From the viewpoint of beneficiation, microscopic examination revealed that a 48-mesh grind was necessary to liberate spodumene and lepidolite from the gangue. For microlite, however, it was noticed that a substantial amount of microlite was liberated at coarser sizes (about 50 per cent at 20 mesh, 70 per cent at 28 mesh, and nearly complete liberation at 35 mesh). The nature and the mode of occurrence of microlite in this ore suggests strongly the possibility of recovering such values at a coarser size using some type of gravity-concentration procedure, such as spiraling or tabling.

Spodumene -Beryl Ore

This type of ore contains about 20 to 30 per cent spodumene on weight basis and 1 to 2 per cent beryl along with quartz, microcline, and albite as the major gangue constituents. Here again the spodumene occurs as coarse and fine laths while the beryl is finely disseminated throughout the ore. Also, a 48-mesh grind was found to be sufficient for liberating spodumene and beryl from the gangue.

Although this type of ore usually constitutes only a small portion of the pegmatite, the treatment of such ores is most important, because it is contemplated that during the life of the mine it would be necessary to separate beryl and spodumene from these ores and from the mine-dump material. Heretofore, a consistently successful selective separation of beryl and spodumene from their mixtures has not been achieved. Therefore, a technique for such a separation would be of considerable practical importance.

FLOTATION EXPERIMENTS ON PURE MINERALS

The mineralogical studies on the various pegmatite ores indicated that although it should be possible to use gravity-separation methods in one or two instances (partial recovery of spodumene and microlite at coarser sizes), all the ores would need to be subjected to flotation procedures to recover the valuable minerals.

To develop such flotation procedures and to obtain a fuller understanding of silicate flotation in general, a research program was initiated to investigate the flotation characteristics of selected pure mineral systems. The pur minerals selected for this study were beryl, spodumene, quartz, microclim albite, muscovite, lepidolite, and microlite.

The previously reported investigations on froth flotation of silicate minerals have centered around fatty acids, alkyl sulfonates, and amines as collectors in the presence of various activators and depressants. Low recover and lack of selectivity were problems encountered in most of the wor reported. Since alkyl sulfonates appeared to be the most promising collector for the majority of pegmatitic minerals, an alkyl sulfonate containing relatively long hydrocarbon chain (Shell Chemical Company's sodium all() aryl sulfonate, 97 per cent pure) was selected to study the flotation behavior c all the minerals investigated except the micas (including lepidolite), for whic an amine type of collector was employed. The primary aim of the experimental program was to examine the flotation response of all the silicate minerals (both valuable and gangue) under similar environments to determine th optimum experimental conditions for selective separation of valuable mineral from the gangue.

The results of the investigation for the beryl and the quartz systems hav been reported by Fuerstenau and Bhappu (1963) and Fuerstenau, Martin, an Bhappu (1963). The published results will be briefly referred to, wheneve applicable, in order to give a comprehensive picture of the problem at hanc

The pure minerals used in the research were obtained from well-know pegmatite deposits, mine workings, and dumps. All mineral samples wer hand-picked lumps of nearly pure mineral. These lumps were crushed in th laboratory and hand-picked once again to obtain as pure a mineral sample a possible. All the experiments were conducted on these unleached minerz samples. <u>Unleashed mineral</u> simply designates naturally pure hand-picke mineral that was wet-ground in a mild steel rod mill, sized to 48 x 150 me sI and dried. Results of experiments with high-purity minerals (obtained b leaching) using conductivity water, although giving very valuable information were not considered for the present study.

All experiments were conducted at room temperature in a Fagergre flotation cell with 100 grams of dry solids (48 x 150 mesh) using 2. 6 liters c tap water or in some instances mine water if available.

Results

Beryl

A number of interesting and important observations were made during th study of the response of beryl to sulfonate flotation (Fuerstenau and Bhappt 1963); namely, (1) at moderate concentrations of sulfonate, beryl respond readily to flotation, (2) the optimum pH for flotation is in the vicinity of pH 4, (3) certain cations affect flotation response significantly, and (4) ferri sulfonate, $Fe(RSO_3)_2 OH$, appeared to be the only cation-sulfonate possessin collector properties for beryl in the pH range 2. 5 to 4.5.

Experimental results with unleached beryl in tap water with variou amounts of sulfonate present in acid media are shown in Figure 2. They reveal that high recoveries are possible at relatively low concentrations c sulfonate; that is, a recovery of 95 per cent was achieved at a sulfonate concentration of 1.28×10^{-5} mole per liter (0. 3 lb/ton).

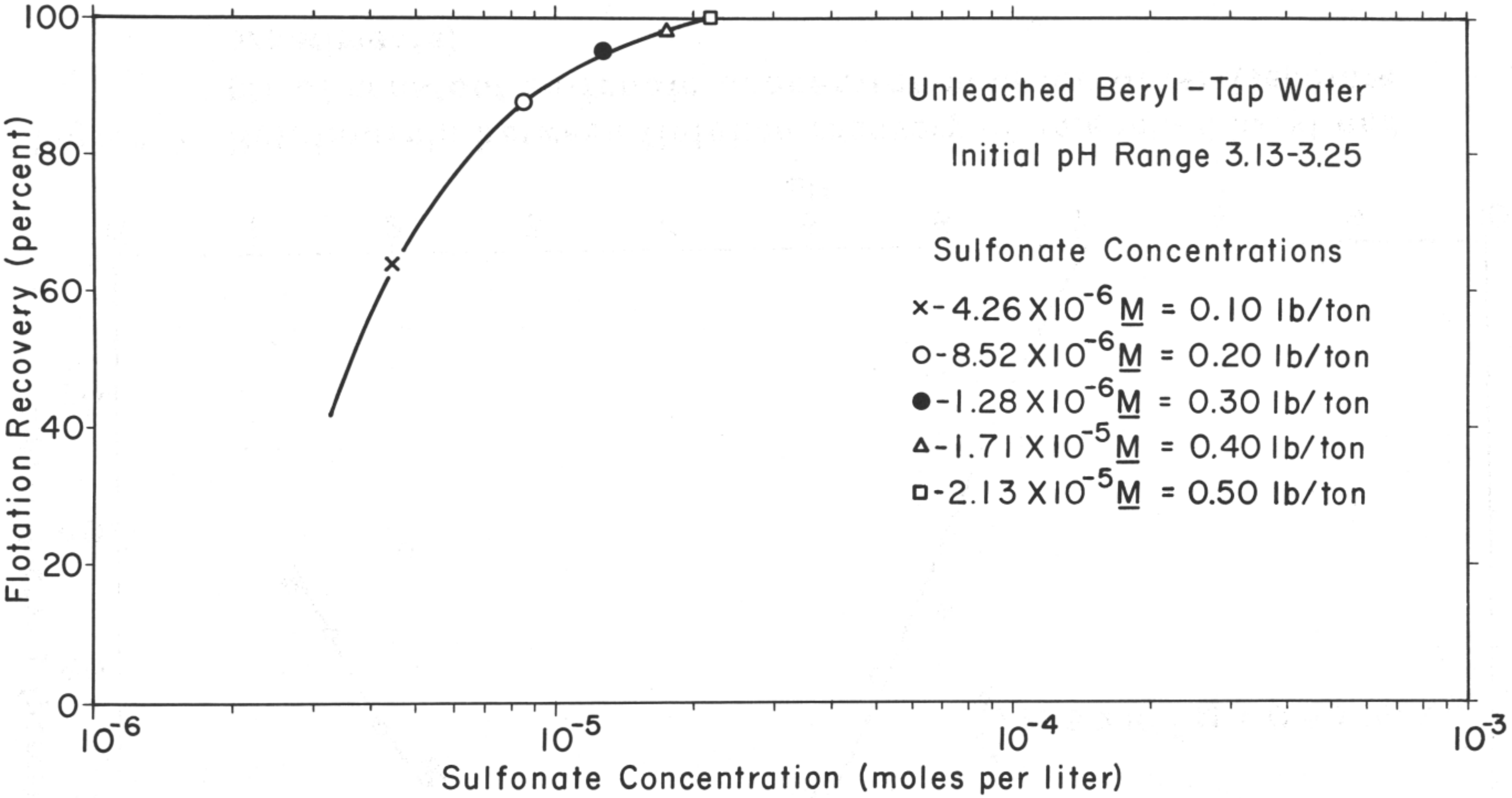

Figure 2. Relationship between flotation recovery of unleached beryl and sulfonate concentration in tap water

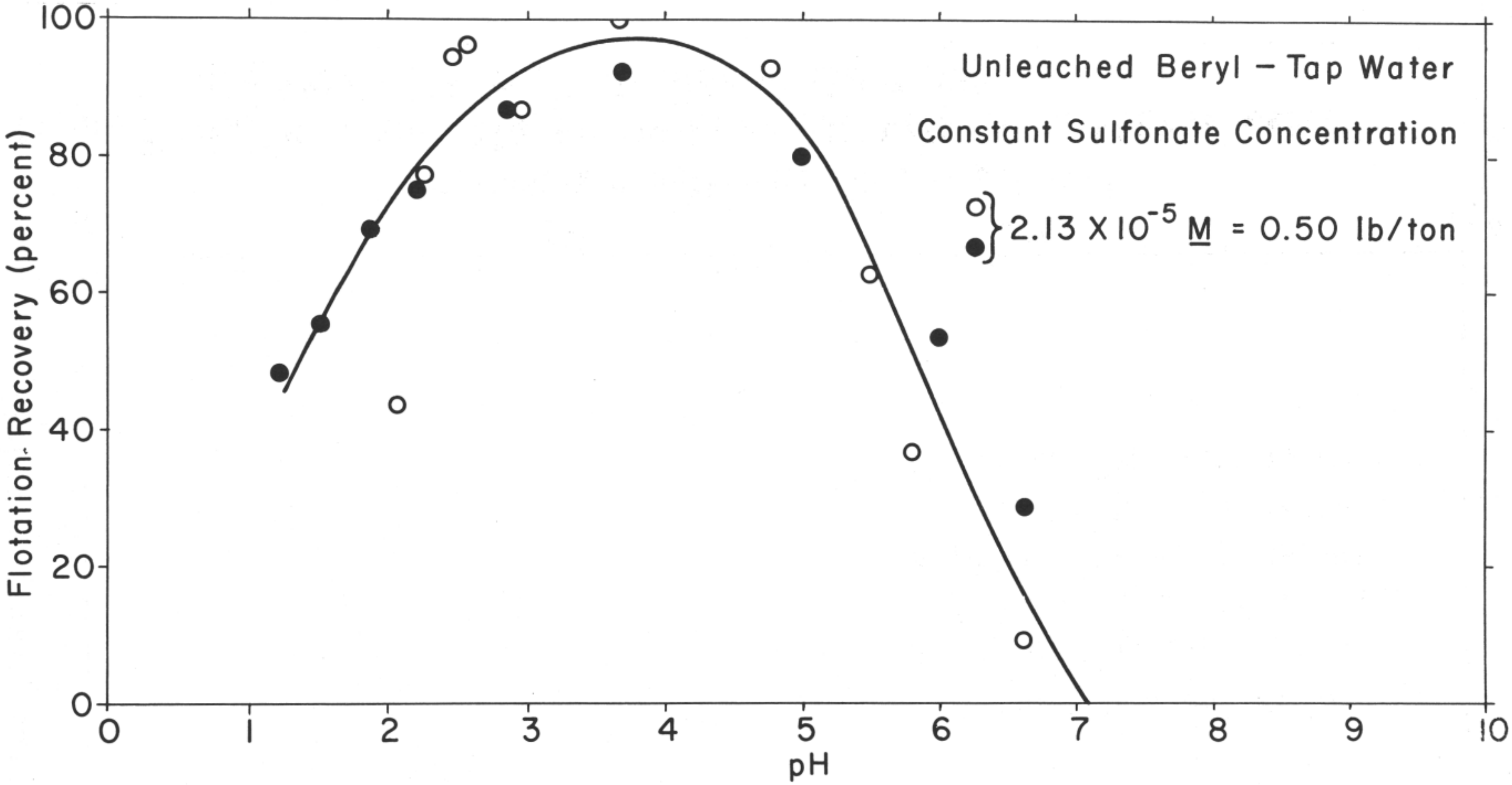

Figure 3. Relationship between flotation recovery of unleached beryl and pH at constant sulfonate concentration in tap water (separate experiments)

Experiments were also conducted with unleached beryl in tap water at constant sulfonate concentration but variable pH to determine the optimum hydrogen ion concentration for maximum flotation recovery in the presence of various ionic impurities. The results of this series of separate experiments are illustrated in Figure 3 in which flotation recovery is plotted as a function of pH at a constant sulfonate concentration of 2.13 x 10^{-5} mole per liter (0.5 lb/ton). The circles and dark circles represent data that were determined with samples of beryl ground at two different times and also used in the experiments at two different times. As shown, complete flotation is effected at pH 3.7. However, there is a fairly broad range of pH (2.5 to 5. 0) in which high recoveries are possible. At lower pH (approaching 2. 0), recovery drops rapidly because of the high total sulfate concentration. A similar drop in recovery occurs above pH 5, apparently due to electrical phenomena at the surface.

Spodumene

Experiments with unleached (naturally pure) spodumene in tap water revealed interesting facts about the flotation behavior of the mineral. It was observed that spodumene behaves very much like beryl except for the amount of sulfonate required to obtain high recoveries, as shown by the critical sulfonate concentration curve in Figure 4. Spodumene requires nearly 1.71 x 10^{-4} mole per liter (4.0 lb/ton) to obtain a recovery of 95 per cent, whereas only 1.28 x 10^{-5} mole per liter (0.3 lb/ton) is required to float beryl at the same pH value. From these results, one may conclude that spodumene is less floatable than beryl, at least in their natural states, and some flotation selectivity in favor of beryl may be possible by using small amounts of sulfonate collector. In practice, however, because of the presence of substantial amounts of Fe+++ ions (activating ions for both beryl and spodumene in pH range 2.5 to 4.5 (Fuerstenau and Bhappu, 1963)) in the natural ores, as well as those contributed during the grinding operation, it is doubtful if any selectivity between the two minerals would be possible.

The similarity of spodumene flotation behavior to that of beryl is clearly illustrated in Figure 5, in which flotation recoveries are shown as a function of pH at a constant collector concentration of 1.28 x 10^{-4} mole per liter (3.0 lb/ton). Nearly complete flotation is effected at pH 3. 6; however, there is a fairly broad range of pH (2. 25 to 4. 25) in which high recoveries are possible. These results are in accord with those shown in the critical pH curve for beryl.

Micas (Muscovite and Biotite)

Flotation of micas has been studied extensively not only for their recovery as a valuable mineral from pegmatitic ores but also for their removal as a primary gangue constituent of the ores to facilitate the flotation of other valuable minerals.

In general, micas are best recovered through the use of amines in an acid circuit at about pH = 2.5. Since sufficient information was available on this treatment, no additional flotation research was conducted on the pure mineral

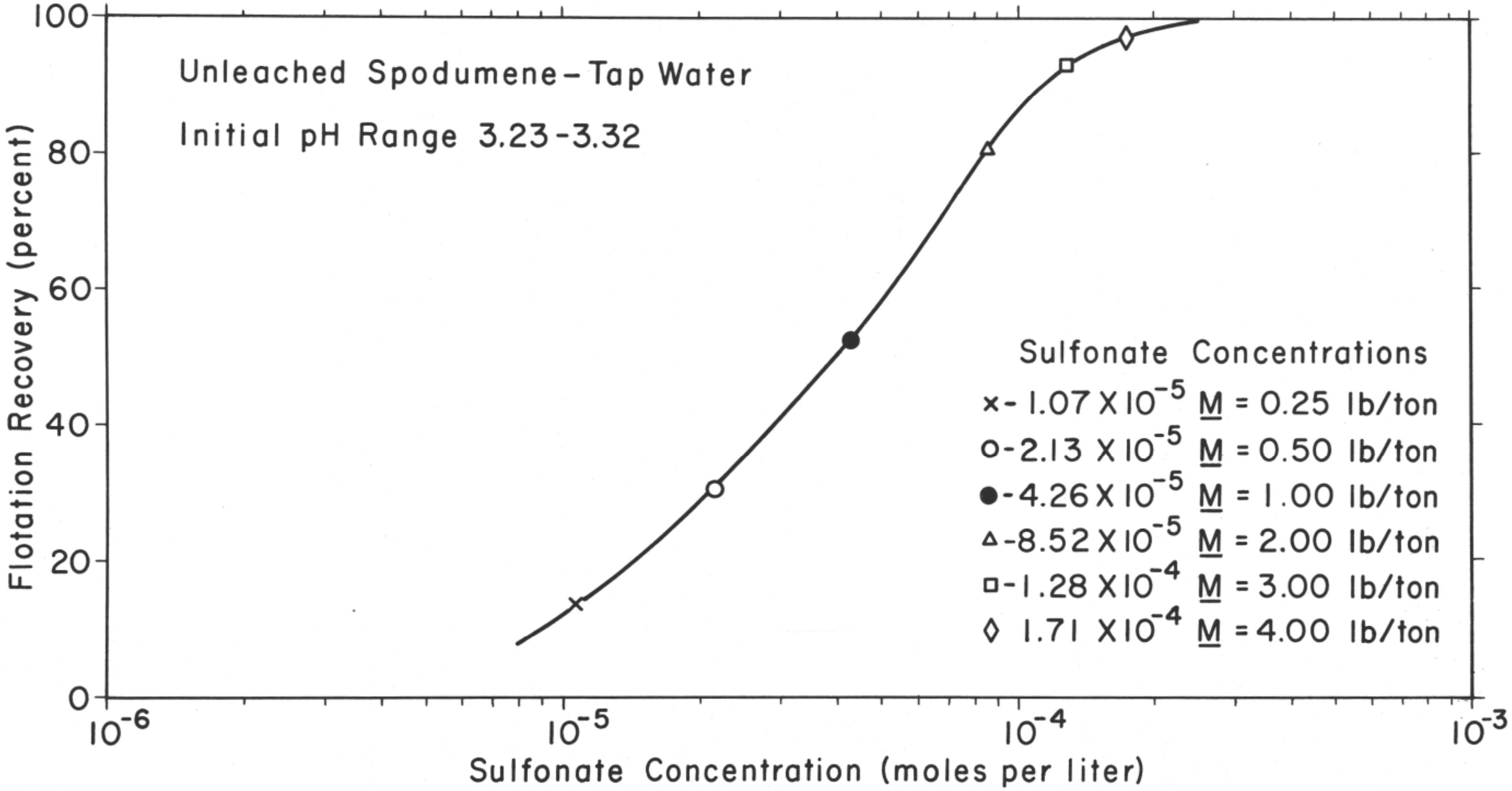

Figure 4. Relationship between flotation recovery of unleached Spodumene and sulfonate concentration in tap water

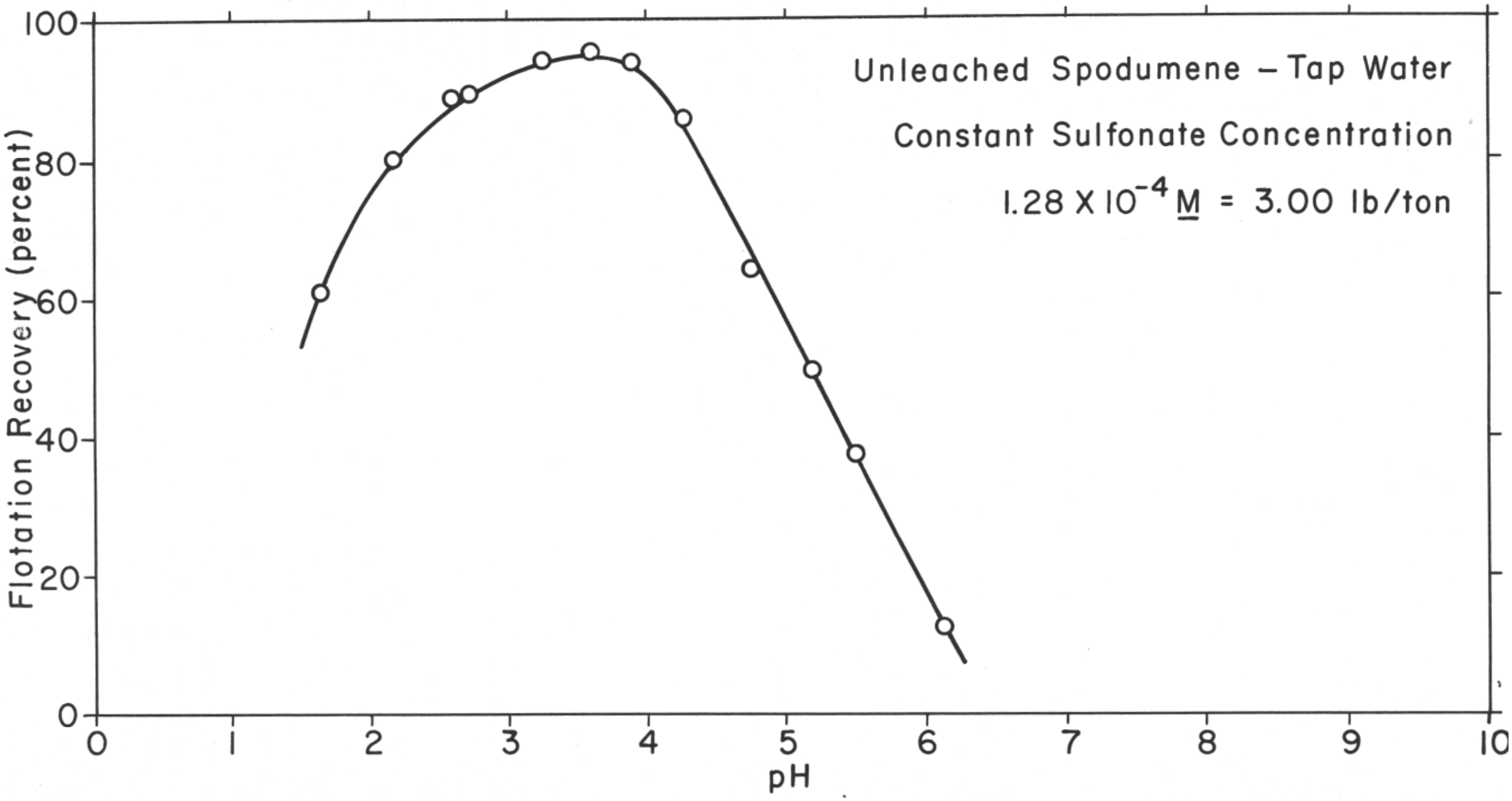

Figure 5. Relationship between flotation recovery of unleached spodumene and pH at constant sulfonate concentration in tap water

samples of muscovite and biotite, except to determine their behavior with the sulfonate collector at low pH values about 2.0 to 5.0. The results of these tests revealed that micas (both muscovite and biotite) showed a weak to medium tendency to float at moderate sulfonate concentration of 2.13 x 10^{-5} mole per liter (0.5 lb/ton). Of the two, the biotite floated the best, apparently because of the presence of ferric ions in its structure.

In view of these results, it was decided to remove the micaceous minerals from the pegmatitic ores prior to the recovery of the valuable minerals beryl, spodumene, and microlite.

Lepidolite

Lepidolite is truly a micaceous mineral and hence shows all the characteristics of muscovite and biotite. Initial tests with pure (high-grade lepidolite rock) mineral indicated that the response of lepidolite to an amine float (primary tallow amine acetate) in a pH range 2.0 to 3.0 was excellent. Amine concentration of 3.2 x 10^{-5} mole per liter (0.75 lb/ton) was sufficient to obtain a recovery of nearly 100 per cent of the mineral. Moreover, no additional activator was necessary for lepidolite flotation. On the other hand, the response of lepidolite to sulfonate flotation at low pH values (2. 0 to 5. 0) was weak even at a sulfonate concentration of 2.13 x 10^{-5} mole per liter (0.5 lb/ton).

These results indicate that from the viewpoint of plant practice, it would be advantageous to remove the lepidolite from the pegmatitic ores by an amine float prior to the recovery of other valuable minerals.

Quartz

The results of investigation concerning sulfonate flotation of quartz indicated that it responds well at moderate concentrations of sulfonate; for example, 6. 37 x 10^{-5} mole per liter (1.5 lb/ton) at pH about 3.0. From these results it would appear that the separation of beryl or spodumene from quartz is not possible. However, the critical pH curve for quartz with sulfonate, as shown in Figure 6, reveals that the flotation of quartz can be prevented by using lower pH. The data presented are for constant sulfonate concentrations of 6.37 x 10^{-5} mole per liter (1.5 lb/ton) and 1.68 x 10^{-4} mole per liter (4.0 lb/ton). The circles and darkened circles represent data that were determined using samples of quartz ground at two different times. The maximum recovery is obtained in the vicinity of pH 4. Above and below pH 4, the curve of recovery as a function of pH drops very steeply. The decrease in recovery above pH 4 may be directly attributable to electrical phenomena at the surface, while the decrease in recovery below pH 4 may be attributed to the competition of the increasing number of sulfate ions (contributed by H2SO4 used for pH adjustment) with the sulfonate ions for the quartz surface. With excess of collector (1.68 x 10^{-4}M = 4.0 lb/ton), however, about• 25 per cent of the material can be floated at the relatively low pH of 2.5.

Should it be desirable to recover previously depressed quartz after the removal of beryl or spodumene, all that is necessary is to raise the pH to 4.0. Addition of ferric salt as an activator at the same pH or somewhat lower will also help in recovering the quartz.

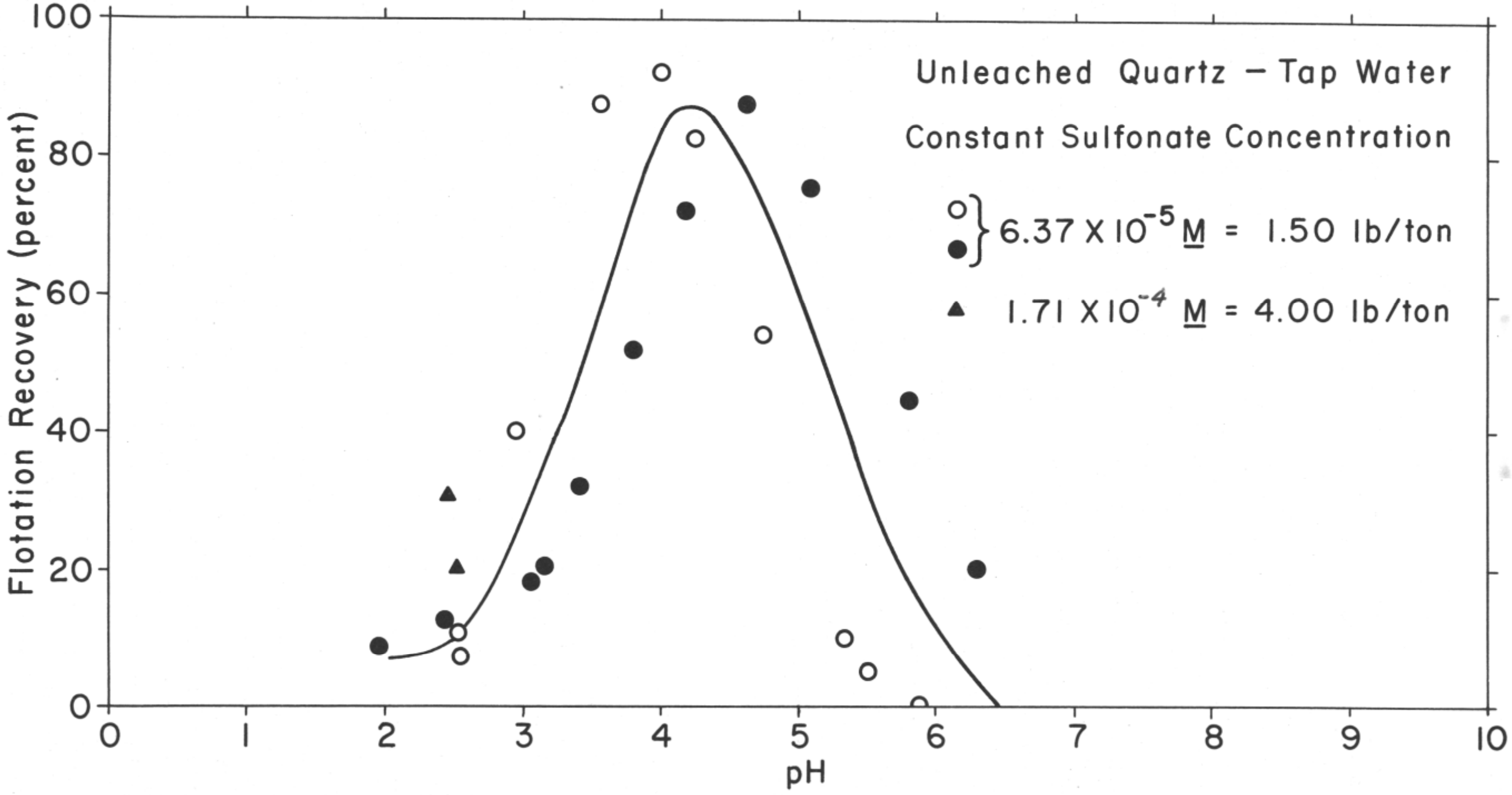

Figure 6. Relationship between flotation recovery of unleached quartz and pH for two different constant sulfonate concentrations in tap water (separate experiments)

Feldspars

The behavior of feldspars was studied with pure samples of microcline and albite, using the sulfonate collector in an acid circuit.

Results of the recovery of unleached microcline ($KA1Si_30_8$) in tap water at various pH values are illustrated in Figure 7. Virtually no flotation could be effected at any pH with 6.37 x 10^{-5} mole per liter (1.5 lb/ton) sulfonate. From a practical viewpoint, this negative response of microcline to sulfonate flotation is advantageous, since good flotation is effected with beryl, spodumene, and quartz in the vicinity of pH 4.0 at the same sulfonate concentration (1, 5 lb/ton). It appears that the complete depression of microcline may be directly attributed to the relatively high sulfate ion concentration (H_2SO_4 used for pH adjustment) at lower pH.

The results of similar experiments with unleached albite ($NaA1Si_30_8$) in tap water revealed that albite behaves more or less like microcline in flotation. For this reason, it would be possible to float beryl, spodumene, or quartz selectively away from albite as well as microcline.

If it is desirable to recover feldspar as a high-grade product from a mixture of other pegmatite minerals, it would be advantageous to float the micas, beryl, spodumene, and quartz selectively as outlined and leave the feldspars as a high-grade product in the tailings. It would also be possible to float feldspars with an amine collector at pH above 3.0. Thus, they can be removed after the mica float with an amine (pH = 2. 5) by raising the pH to 3.0 or above.

Microlite

The flotation of microlite ($Ca_2Ta_2O_7$) has not been reported in the literature. Since microlite occurs in small but economically appreciable quantities in some of the pegmatitic ores, it would be desirable to perfect a treatment for its recovery. In the past, microlite, because of its relatively high specific gravity (6 to 6. 3), has been concentrated by gravity separation (Wood, 1946), and logical plant practice dictates that such a method should be used whenever possible in the flowsheet to recover microlite in coarser sizes. This is not true for the recovery of microlite in sizes finer than 150 mesh. Microlite is known to be a friable mineral and tends to slime badly during crushing and grinding stages. Such fine microlite reports in the tailings of gravity concentration procedures and is lost. A flotation procedure, if available, would be an ideal solution to this problem.

While experimenting with "spotted rock" type of ore containing lepidolite, spodumene, and microlite as the valuable minerals, it was noticed that the microlite particles had a tendency to float along with spodumene, using the sulfonate collector. This observation clearly indicated the flotation response of microlite under the conditions required for spodumene flotation. This fact was further confirmed through flotation experiments on high-grade microlite samples from pegmatites known to contain this mineral, which indicated that more than 85 per cent recovery of microlite was possible with a moderate amount (8.52 x 10^{-5}M = 2.00 lb/ton) of sulfonate in a pH range 2.5 to 4.5. Moreover, it was possible to obtain marketable grades of concentrates containing better than 50 per cent Ta_20_5 after two or three cleanings.

Naturally, when both spodumene and microlite are present in the feed, a

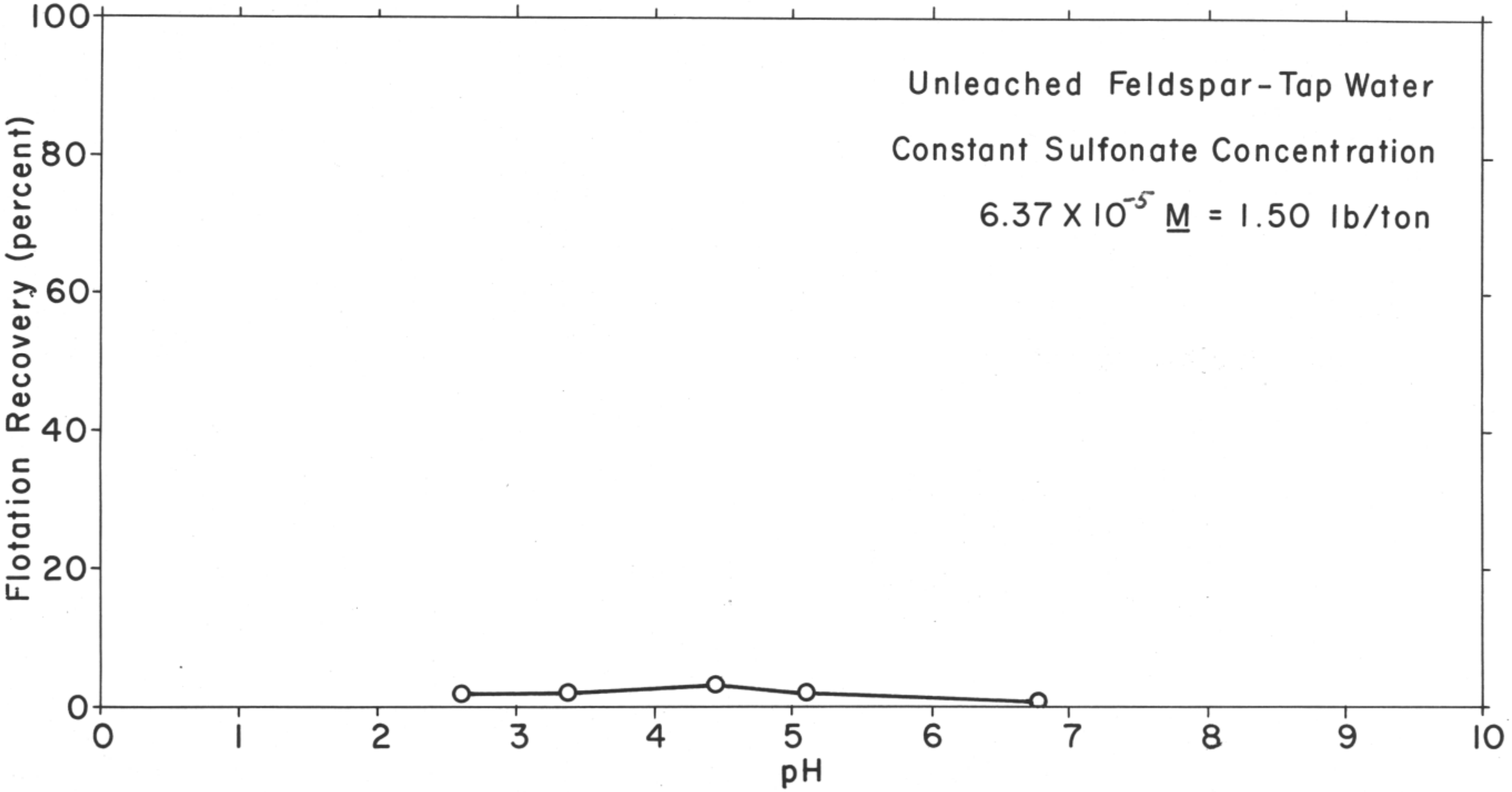

Figure 7. Relationship between flotation recovery of unleached feldspar and pH at constant sulfonate concentration in tap water (separate experiments)

bulk concentrate of spodumene-microlite would be obtained. The microlite from the bulk concentrate can then be selectively separated by a simple gravity-concentration technique, such as tabling or spiraling. Some efforts have also been made to separate microlite selectively from its bulk concentrate by using fluoride ion which is found to be an effective depressant for spodumene. The mechanism of this technique is not well understood at present.

With the current knowledge of flotation for the recovery of microlite, it should be possible to obtain high recoveries of this valuable mineral from pegmatite ores. The fear of overgrinding and microlite-sliming, a serious problem in gravity separation flowsheets, is not so serious with flotation. Alternately, to ensure the optimum recovery of microlite from both coarse and fine sizes, a combination of gravity and flotation procedures could be incorporated in the flowsheet. In this instance, the classifier overflow would be subjected to a suitable gravity-concentration step, such as spiraling or tabling, to recover coarse microlite and also columbite-tantalite, if such minerals are present in the feed. The tailings from this operation then would constitute the feed to a flotation section where the fine microlite would be recovered along with the spodumene concentrate. This.bulk concentrate could then be subjected to a suitable gravity or flotation treatment to obtain a high-grade microlite concentrate and spodumene-rich tailings.

Beryl-Spodumene Separation

Experimental work on the flotation characteristics of various pegmatitic minerals using sulfonate as collector indicates that it should be possible to recover all the minerals selectively. A problem might arise, however, if beryl and spodumene both are contained in the pegmatite ore.

It has been shown that beryl and spodumene both float well with sulfonate collector in the pH range 2.5 to 4.5. This difficult problem of beryl-spodumene separation was successfully solved in experiments with pure mineral systems and mixtures of the pure minerals. The results of this phase of the investigation will be reported in detail in a forthcoming publication by the authors.

Briefly, the selective separation of beryl from a pegmatitic ore containing spodumene is achieved through the use of moderate concentration of fluoride ions ($4.26 \times 10^{-5}M$ = 1.0 lb/ton) and sulfonate collector ($3.2 \times 10^{-5}M$ = 0.75 lb/ton) at a pH of 2.6 to 2.8. The mechanism of depression appears to be selective adsorption of HF_2 ions on the surface of spodumene, thereby preventing its collection by the sulfonate. The complexing property of fluoride ions may also play an important role in this phenomenon. After selective flotation of beryl, spodumene can be activated and floated selectively from the gangue minerals with the help of small amounts of ferric ions ($6.8 \times 10^{-6}M$ = 0.16 lb/ton) and additional sulfonate collector ($3.2 \times 10^{-5}M$ = 0.75 lb/ton) at pH 2.7.

From the view of plant practice, it may be advisable to make a bulk beryl-spodumene concentrate from the pegmatite ore and then obtain a selective separation of one mineral from the other. Such selective separation might be brought about by using hydrosulfide ions to reduce ferric-sulfonate / $Fe(RSO3_2)OH$/, the actual collector, thereby depressing both the minerals.

In addition, the hydrosulfide ions somehow appear to depress the surface of beryl selectively so that on lowering the pH to 3.5 to 4.0 and adding extra sulfonate collector, spodumene is selectively floated away from beryl. Decomposition of ferric-sulfonate collector coating and subsequent selective flotation of spodumene from beryl can also be accomplished by employing iron-complexing (chelating) agents, such as versenes, oxalates, citrates, tartarates, and the like.

As yet, the mechanism of the techniques for selective separation of beryl and spodumene from their bulk concentrate is not clearly understood. From experimental evidence, it appears that the complexing property of hydrogen-sulfide ions and the chelating reagents may somehow be responsible for obtaining the selectivity. Regardless of the mechanisms involved, it is now possible to obtain selective separation of beryl and spodumene either from their ores or from their bulk concentrate. This flotation technique should help considerably in obtaining fair selectivity between pegmatitic minerals.

FLOTATION TESTS ON NATURAL ORES

Experiments performed with separate systems of the pure minerals usually contained in pegmatite ores showed that good selective separation of valuable minerals, such as beryl, spodumene, lepidolite, and microlite from the principal gangue constituents should be effected by proper control of pH using appropriate collectors.

The results with pure mineral systems are summarized in Table 1.

TABLE 1. SUMMARY OF RESULTS WITH PURE MINERAL SYSTEMS

Mineral	Conditions for Selective Flotation			
	pH	Reagent	Amount (lb/ton)	Remarks
Beryl	2.5-4.5	Sulfonate	0.50	Good flotation
Spodumene	2.5-4.5	Sulfonate	3.00	Good flotation
Microlite	2.5-4.5	Sulfonate	3.00	Good flotation
Quartz	4.0	Sulfonate	1.50	Good flotation
Feldspars	2.5-5.0	Sulfonate	0.5-3.0	No flotation
Feldspars	3.0 or more	Amine	0.75	Good flotation
Micas and Lepidolite	2.5-4.5	Sulfonate	0.5-3.0	Mediocre flotation
Micas and Lepidolite	2.5	Amine	0.75	Good flotation
Beryl	2.7-9.0	Sodium sulfide	1.5	Depression
Spodumene	2.7-4.5	HF acid	1.0	Depression

The pertinent experimental data shown in Table 1 serve as guideposts for selective separation of one or more minerals from the pegmatite ores. Thus, beryl can be separated from quartz and feldspars at pH 2.5 with sulfonate as a collector. On the other hand, lepidolite can be separated from beryl, spodumene, quartz, and feldspars at pH 2.5 with an amine as a collector. Experiments were conducted with natural ores to confirm these results.

Low-Grade Beryl Ore

A sample of low-grade beryl ore was obtained from the mine dump of a well-known pegmatite deposit in New Mexico. The ore assayed 0.55 per cent BeO and contained quartz, feldspar, apatite, mica-schist, and a substantial amount of iron-bearing silicates. The optimum experimental procedure employed in selectively separating beryl from the gangue was as follows:

1. A 500-gram charge of ore was ground through 48 mesh;
2. the charge was deslimed by decantation at 20 microns;
3. after repulping with tap water, the pH was adjusted to 2.40 with sulfuric acid;
4. the pulp was conditioned with 0.73 lb/ton primary tallow amine acetate and two drops (0.12 lb/ton) of methyl isobutyl carbinol for three minutes;
5. the mica-schist was removed during three minutes of flotation;
6. the pulp was thickened to remove the supernatent solution;
7. after repulping, the pH was adjusted to 2.60 with sulfuric acid;
8. the pulp was stage-conditioned with 0.98 lb/ton sodium alkyl aryl sulfonate and one drop of methyl isobutyl carbinol for three minutes;
9. a beryl concentrate was obtained in three minutes of flotation (the final pH 2. 65);
10. the rougher beryl concentrate was cleaned and recleaned at pH 2.7 with an addition of only one drop of frother; and
11. the recleaner concentrate was then dried and passed through a high-intensity magnetic separator to remove apatite and iron-bearing gangue minerals.

Typical results obtained from one of several experiments using this technique are given in Table 2. As shown in the table, a recleaner concentration amounting to a recovery of about 70 per cent of the beryl at a grade of 8.00 per cent BeO was obtained by this procedure. The recovery in the rougher concentrate amounted to about 90 per cent of the beryl. The data also revealed that a relatively large amount of material (127.2 grams out of 400 grams) had to be floated to obtain a good rougher tailing (0. 016 per cent BeO). Although experimentation on systems of pure quartz indicated that the flotation response of quartz should be "sluggish" at pH 2.5, a sufficient amount floated from the ore to require two cleanings to raise the grade of the final beryl concentrate.

As anticipated, feldspar did not float at this pH and thus did not contaminate the beryl concentrate.

The other principal contaminates of the recleaner concentrate were apatite and iron-bearing silicates. Their response to flotation was such that the only method available for separating them from the beryl was by high-intensity magnetic separation. The iron-bearing silicates will always present problems of concentrate contamination with this technique because of their amenability to sulfonate flotation. In this instance, the ore contained an appreciable amount of schist and amphiboles. For freshly mined ore, should the overburden of schist be removed prior to mining, the feed to the beryl flotation section would be reasonably free of these iron-bearing impurities, and it should be possible to obtain a higher grade of beryl concentrate without magnetic separation.

TABLE 2. SELECTIVE FLOTATION OF BERYL FROM LOW-GRADE ORE

Product	Weight (%)	Per cent BeO	BeO content	Per cent distribution
Recleaner concentrate (nonmagnetic)	4.86	8.00	0.389	67.83
Recleaner concentrate (magnetic)	2.74	0.290	0.008	1.39
Recleaner tails	6.28	1.360	0.086	15.00
Cleaner tails	12.56	0.298	0.037	6.45
Final tails	53.38	0.016	0.009	1.57
Mica concentrate	15.40	0.122	0.019	3.32
Slimes	4.78	0,542	0.026	4.54
Calculated heads	100.00		0.574	100.10
Heads assay		0.550		

Similar flotation tests were conducted on low-grade beryl ores from Arizona, Colorado, and Canada. In each instance, excellent recovery (above 90 per cent) of the beryl was obtained with a recleaner concentrate assaying in excess of 8.0 per cent BeO.

This procedure for recovering beryl is comparatively simple and the reagents used are inexpensive and easily available. Moreover, it should be easy to adapt this procedure to plant practice.

Lithium-Tantalum Ore (Spotted Rock)

The flotation tests conducted on the lithium-tantalum ore, also obtained from a well-known pegmatite body in the northern part of New Mexico, resulted in the development of a procedure similar to that established for floating beryl. The ore contained 1.7 per cent Li_2O and 0.15 per cent Ta_20_5. Of the total lithium content present, about 90 per cent is in spodumene and the remaining 10 per cent in lepidolite. The optimum experimental procedure employed in selectively separating lithium and tantalum minerals from the gangue was as follows:

1. A 500-gram charge of ore was ground through 48 mesh in three stages;
2. the ground product was tabled to obtain a heavy fraction containing coarse microlite;
3. the charge was deslimed by decantation at pH 2. 0;
4. after repulping with tap water, the pH was adjusted to 2.40 with sulfuric acid;
5. the pulp was conditioned with 0.75 pound a ton primary tallow amine acetate and two drops (0. 12 lb/ton) of methyl isobutyl carbinol for three minutes;
6. the lepidolite concentrate was removed during three minutes of flotation (no effort was made to reclean lepidolite concentrate);

7. the pulp was thickened to remove the supernatent solution;
8. after repulping, the pH was adjusted to 2.60 with sulfuric acid;
9. the pulp was stage-conditioned with 1.50 pounds a ton sodium alkyl aryl sulfonate and one drop (0. 06 lb/ton) of methyl isobutyl carbinol for three minutes;
10. a spodumene concentrate was obtained in three minutes of flotation;
11. the rougher spodumene concentrate was cleaned and recleaned at pH 2.7 with additional sulfonate collector (0. 16 lb/ton in each cleaning);
12. the recleaner concentrate was conditioned with 1.5 pounds a ton sodium sulfide to remove the collector coating; and
13. the surface-cleaned lithium-tantalum concentrate was then manually panned to obtain a heavy microlite concentrate.

Typical results obtained from one of several experiments are given in Table 3. As shown, in excess of 90 per cent of the spodumene contained in the ore was recovered in a rougher concentrate averaging 5.0 per cent Li_2O. The cleaner concentrate amounted to 6.32 per cent Li_2O and accounted for more than 65 per cent of the total spodumene, while the recleaner concentrate assayed 6.49 per cent Li2O and contained about 50 per cent of the total spodumene. The lepidolite rougher concentrate also represents a high recovery of this mineral in the rougher. Such a product was cleaned and recleanedto obtain a marketable product assaying better than 4.0 per cent Li_2O in the final concentrate.

The results indicate that about 39 per cent of the total tantalum values was recovered in the coarse gravity concentration (tabling step), while an additional 46 per cent of the total tantalum content was recovered in the flotation step, yielding a total recovery of about 85 per cent of the tantalum content contained in the ore. This tantalum recovery represents a substantial improvement over past practice in which only coarse values were recoverable by gravity concentration.

These results clearly indicate the simplicity and selectivity possible by employing a sulfonate float for both the spodumene and microlite rather than the usual fatty-acid float which necessitates stringent conditioning with numerous reagents and other specific requirements for successful flotation.

Spodumene-Beryl Ore

Experiments performed with separate systems of the pure minerals beryl and spodumene showed that a good selective separation of beryl or spodumene from their mixture should be effected with appropriate depressants. Experiments were conducted with a natural ore to confirm these observations.

The ore investigated was obtained from the same pegmatite deposit from which the other ore samples were collected. The ore assayed 0.819 per cent BeO and 2.38 per cent Li_2O and contained quartz and feldspar (both albite and microcline) as the major gangue constituents. The optimum experimental procedures employed in selectively separating beryl and spodumene from the gangue were two. One was the selective separation directly from the ore, as follows:

TABLE 3. RECOVERY OF SPODUMENE AND MICROLITE FROM LITHIUM--TANTALUM ORE

Product	Weight (%)	Li2O (%)	Content (Li2O)	Distribution (%)
Recleaner concentrate	13. 15	6. 49	0. 8545	48. 05
Recleaner tails	4. 64	5. 78	O. 2680	15. 09
Cleaner tails	13. 00	3. 14	0. 4080	23. 00
Final tails	52. 02	0. 11	0. 0572	3. 28
Lepidolite concentrate	9. 74	1. 24	0. 1208	6.81
Slimes	7. 12	0. 94	0. 0670	3. 77
Calculated heads	99. 67		1. 7755	100. 00
Heads assay (Li2O)		1. 68		
Metallurgical balance (Ta2O5)		Ta205 (%)	Content (Ta205)	
Coarse concentrate (tabling section)	0. 16	36. 1	0. 0577	38. 5
Fine concentrate (flotation section)	0. 17	40. 8	0. 0695	46. 3
Loss in other products (by difference)	99. 67	----	0. 0228	15. 2
Heads assay (Ta205)	100. 00		0. 1500	100. 00

1. A 500-gram charge of ore was ground through 48 mesh;
2. the charge was deslimed by decantation at 20 microns;
3. after repulping with tap water, the pH was adjusted to 2. 65 with sulfuric acid;
4. the pulp was conditioned with 1. 00 pound a ton sodium fluoride for ten minutes;
5. the pulp was next conditioned with 0. 75 pound a ton sodium alkyl aryl sulfonate and two drops (1. 12 lb/ton) of methyl isobutyl carbinol for three minutes;
6. a beryl concentrate was obtained in three minutes of flotation (the final pH = 2. 78);
7. the rougher beryl concentrate was cleaned and recleaned at pH 2. 7 with an addition of only one drop of frother;
8. the first cleaner tailing was combined with the rougher tailing to constitute the final tails;
9. the combined tailings (rougher and first cleaner) pH were adjusted to 2. 65 with sulfuric acid;
10. the pulp was conditioned with 0. 16 pound a ton ferric chloride and 0. 75 pound a ton sodium alkyl aryl sulfonate and one drop of methyl isobutyl carbinol for three minutes;
11. a spodumene concentrate was obtained in three minutes of flotation (the final pH 2. 8); and
12. the rougher spodumene concentrate was cleaned and recleaned at pH 2. 65 without additional reagents.

Typical results obtained from one of several experiments are given in Table 4. As shown in the table, a beryl recleaner concentrate assaying 8.30 per cent Be0 with a recovery of about 65 per cent was obtained by this procedure, while the spodumene recleaner concentrate assayed 6.26 per cent LiO_2 with a recovery of about 78 per cent. These results indicate the effectiveness of selective flotation of beryl and spodumene away from the gangue and from each other. The results presented here do not necessarily represent the ultimate recoveries or grades possible with this procedure. With further improvement in the technique and additional cleanings, it should be possible to obtain better recoveries as well as grades.

TABLE 4. SELECTIVE FLOTATION OF BERYL AND SPODUMENE DIRECTLY FROM THE ORE

Product	Weight (%)	Assay (%)		Content		Distribution(%)	
		BeO	Li_2O	BeO	Li_2O	BeO	Li_2O
Beryl recleaner conc.	6.32	8.30	2.59	0.525	0.164	65.00	6.87
Beryl recleaner tails	2.08	3.17	3.83	0.066	0.080	8.18	3.35
Spodumene recleaner conc.	30.00	0.34	6.26	0.102	1.880	12.67	78.70
Spodumene recleaner tails	5.45	0.50	1.07	0.022	0.048	2.73	2.01
Final tails	52.26	0.07	0.24	0.037	0.126	4.59	5.27
Slimes	3.86	1.39	2.33	0.054	0.090	6.83	3.80
Calculated heads	100.00			0.806	2.388	100.00	100.00
Heads assay		0.819	2.38				

The procedure for the selective flotation of beryl and spodumene from bulk concentrate was as follows:

1. A 500-gram charge of ore was ground through 48 mesh;
2. the charge was deslimed by decantation at 20 microns;
3. after repulping with tap water, the pH was adjusted to 2.65 with sulfuric acid;
4. the pulp was conditioned with 1.50 pounds a ton sodium alkyl aryl sulfonate and two drops (0.12 lb/ton) of methyl isobutyl carbinol for three minutes;
5. a bulk beryl-spodumene concentrate was obtained in three minutes of flotation (the final pH 2.76);
6. the rougher bulk concentrate was cleaned once at pH 2.7 without additional reagents;
7. the cleaner tailing was combined with the rougher tailing to constitute the final tails;
8. the cleaned bulk concentrate was repulped with tap water and conditioned with 1.5 pounds a ton sodium sulfide (Na2S•9H20) for five minutes (conditioning pH 9.3);

9. the pH of the pulp was adjusted to 3. 5 with sulfuric acid;
10. the pulp was stage-conditioned with 0. 4 pound a ton sodium alkyl aryl sulfonate and one drop of methyl isobutyl carbinol for three minutes; and
11. a selective spodurnene concentrate was then floated for three minutes, leaving behind a concentrated beryl product in the tailing.

Typical results obtained from one of several experiments are given ir Table 5. It was possible to obtain a selective flotation separation of spodumene away from the beryl and from their bulk concentrate. A spodurnene-rich product assaying 5. 28 per cent Liz() with a recovery of about 85 per cen and a beryl-rich product assaying 6. 22 per cent Be0 with a recovery of abou 71 per cent were obtained by this procedure. The grades of Li20 and Be0 ir their respective concentrates appear to be rather low (5. 27 per cent Liz() ir spodurnene concentrate and 6. 22 per cent Be0 in the beryl concentrate); however, it should be remembered that these grades only represent rougher concentrates, and, as clearly shown in the results of previous tests, it shoulc be possible to obtain acceptable grades with further cleanings of the rougher concentrates and improvement in technique.

TABLE 5. SELECTIVE SEPARATION OF SPODUMENE AND BERYL FROM BULK CONCENTRATE

Product	Weight (%)	Assay (%) BeO	Assay (%) Li_2O	Content BeO	Content Li_2O	Distribution (%) BeO	Distribution (%) Li_2O
Spodumene conc.	37. 10	0. 39	5. 28	0. 145	1. 960	17. 95	84. 60
Spodumene tails or beryl conc.	9. 26	6. 22	1. 67	0. 576	0. 155	71. 30	6. 71
Final tails	49. 90	0. 07	0. 26	0. 035	0. 130	4. 34	5. 62
Slimes	3. 74	1. 36	2. 02	0. 052	0. 071	6. 41	3. 07
Calculated heads	100. 00			0. 808	2. 316	100. 00	100. 00
Heads assay		0. 819	2. 38				

Experiments with natural ores were also conducted by conditioning the bulk beryl-spodumene concentrate with iron-complexing (chelating) agents such as Versenes (Regular and Fe-3), citric acid, tartaric acid, and oxalic acid instead of sodium sulfide. In every instance, it was possible to decompose the ferric-sulfonate coating and, after conditioning, to obtain a more or less selective spodumene float. Of the complexing agents investigated, Versenes gave the best selectivity and results. For the sake of brevity, the results of these tests are not included in this report.

Conclusions

This investigation shows that it is possible to recover beryl, spodumene, lepidolite, microlite, and even some of the gangue minerals such as micas, feldspars, and quartz from typical pegmatitic ores by flotation techniques.

The treatment procedures developed for the recovery of various valuable minerals are more or less identical, allowing the use of a common flowsheet for the treatment of various pegmatitic ores, as shown in Table 6.

A critical study of the table reveals that for low-grade beryl ore, the amine float removes the micaceous impurities while the sulfonate recovers the beryl. For lithium-tantalum ore, the same amine float recovers micaceous lepidolite as a concentrate while the sulfonate float recovers spodumene and microlite. This bulk concentrate is then treated separately to obtain a microlite concentrate and a spodumene concentrate. If a pegmatite ore contains both beryl and spodumene (spodumene-beryl ore) as the valuable minerals, the sulfonate float recovers both of these in the form of a bulk concentrate which is then appropriately treated to obtain a beryl concentrate and a spodumene concentrate. If this ore contains micaceous impurities or lepidolite values, they can be removed or recovered by an amine float prior to the sulfonate float.

The inherent advantages of the flowsheet include adaptability and flexibility for treating various pegmatitic ores encountered in practice, use of common flotation and gravity circuits, employment of common reagents, and over-all economy obtained through lower initial milling cost and plant operation.

It is recommended that the proposed flotation procedures for the recovery of valuable pegmatitic minerals and the common flowsheet for the treatment of pegmatitic ores be critically evaluated against known procedures and flowsheets to determine the optimum treatment for valuable minerals contained in pegmatitic ores.

Table 6. Development of a Common Flowsheet for Recovery of Valuable Minerals from Pegmatitic Ores

	LOW-GRADE BERYL ORE		LITHIUM-TANTALUM ORE		SPODUMENE-BERYL ORE	
	Minerals		Minerals		Minerals	
	Valuable	Gangue	Valuable	Gangue	Valuable	Gangue
	Beryl	Micas Fe-Silicates Quartz Feldspars	Lepidolite Spodumene Microlite	Quartz Feldspars	Beryl Spodumene	Quartz Feldspars

COMMON FLOWSHEET	RESULTS	RESULTS	RESULTS	REMARKS
Crushing and Grinding	48-mesh grind	48-mesh grind	48-mesh grind	Rod-mill grind in stages.
Gravity - Concentration	Recovery of heavy minerals	Recovery of coarse microlite	Recovery of heavy minerals	Spiraling or tabling.
Desliming (-15)	Removal of -15 slimes	Removal of -15 slimes	Removal of -15 slimes	Cyclone or classifier may be used.
Amine Flotation	Removal of micas	Recovery of lepidolite	Removal of micas	Primary tallow amine acetate and MIBC used for all ores.
Thickening	Removal of amine solution	Removal of amine solution	Removal of amine solution	Amine solution reused.
Sulfonate Flotation				
Final Tailing Rougher Conc.	Rougher beryl conc.	Rougher spod.-Microlite conc.	Rougher spod.-beryl conc.	Sodium alkyl aryl sulfonate used for all ores.
Cleaning & Recleaning				*Beryl can be selectively floated first.
Middlings Recleaner conc.	Recleaner beryl conc.	Recleaner spod.-microlite conc.	Recleaner spod.-beryl conc.	Selective cleaning by pH control.
Selective Separation				
(a) Magnetic	Magnetic fraction (discard) Final beryl conc.			May or may not be necessary in case of beryl ores.
(b) Gravity or Other		Final microlite conc. Final spodumene conc.		Spiraling or slime tabling after removing collector coating.
(c) Selective Flotation			Final spodumene conc. Final beryl conc.	Removal of collector coating by conditioning with sodium sulfide or iron-complexing reagent followed by selective flotation of spodumene.
*Selective Flotation of Beryl			Beryl conc. Spodumene conc.	Beryl can be selectively floated first by depressing spodumene with HF. Spod. can be then reactivated with Fe^{+++} and sulfonate.

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