Bureau of Mines
Report of Investigations 5105

# BENEFICIATION STUDIES OF COLUMBIUM-TANTALUM-BEARING MINERALS IN ALLUVIAL BLACK-SAND DEPOSITS

BY J. E. SHELTON AND W. A. STICKNEY



United States Department of the Interior — February 1955

# BENEFICIATION STUDIES OF COLUMBIUM-TANTALUM-BEARING MINERALS IN ALLUVIAL BLACK-SAND DEPOSITS

BY J. E. SHELTON AND W. A. STICKNEY

* * * * * * * * * Report of Investigations 5105

UNITED STATES DEPARTMENT OF THE INTERIOR
Douglas McKay, Secretary
BUREAU OF MINES
J. J. Forbes, Director

---



February 1955

TABLES (Con.)



ILLUSTRATIONS

## INTRODUCTION

This presentation covers the results of laboratory studies conducted by the Bureau of Mines Mineral Dressing Section, Northwest Electrodevelopment Laboratory, Albany, Oreg., on alluvial sands from Dismal Swamp, Bear Valley, and Cascade, Idaho.

The search for radioactive minerals has led to the reexamination of alluvial deposits; in the process of this reappraisal the frequent presence of minerals bearing columbium and/or tantalum was noted. Because of increased demand for these metals by the defense industries and because these industries depend upon foreign sources for their supply the domestic alluvial black sands are being considered as a possible domestic source. However, utilization of these alluvial deposits depends upon development of methods of concentrating these valuable minerals.

The primary objective of the test work was the study of the effect of attrition scrubbing, sizing, magnetic, electrostatic, and gravity separation as methods for concentrating and recovering the columbium and tantalum-bearing minerals, samarskite, euxenite, ilmenorutile, and columbite.

## DISMAL SWAMP

### Summary

Laboratory test work was conducted on a sample of columbite-bearing black-sand concentrate from Dismal Swamp, Elmore County, Idaho, with the object of producing, by physical methods, columbite and other valuable mineral concentrates.

Laboratory tests indicated that, by sizing and magnetic separation, 88.0 percent of the $(Cb, Ta)_2O_5$ could be recovered in a columbite concentrate representing 67.47 percent of the weight, which had an analysis of 60.0 percent $Cb_2O_5$ and 6.0 percent $Ta_2O_5$.

Additional magnetic separations recovered 3.07 percent $(Cb, Ta)_2O_5$ in a samarskite concentrate that represented 6.14 percent of the weight and had an analysis of 23.0 percent $Cb_2O_5$ and 2.3 percent $Ta_2O_5$.

### Beneficiation of the Dismal Swamp Black Sands

#### Nature of Ore

This sample of columbite black sand was submitted by Mr. O. E. Pothier, mining engineer for the J. R. Simplot Co., Continental Bank Building, Boise, Idaho.

Representative portions of the head sample was prepared and submitted for chemical and spectographic analyses, with the results as shown in tables 1 and 2.

TABLE 1. - Chemical analyses

| Fe | $TiO_2$ | REO+Th | $U_3O_8$ |
|---|---|---|---|
| 5.85 .................. | 3.67 | 4.00 | 0.34 |

TABLE 2. - Spectrographic analyses

| Constituent | Percent |
|---|---|
| Fe, Cb .................. | Over 10 |
| Ta, Si .................. | 5 to 10 |
| Mn, Zr, Ti .............. | 1 to 5 |
| Al ...................... | 0.1 to 1 |
| Sn, Ca, Mg, Mo | 0.01 to 0.1 |
| Be, Cu .................. | 0.001 to 0.001 |
| La, Y, Nd ............... | Also indicated |
| $Cb_2O_5$ ................ | 46.0[1] |
| $Ta_2O_5$ ................ | 4.6[1] |

1/ Quantitative X-ray fluorescent analysis.

A representative sample of the sands was examined in the petrographic laboratory and was reported to contain essentially columbite, some quartz and feldspar, less garnet and monazite, and relatively small amounts of the following minerals: Ilmenite, altered zircon, cassiterite, sericite, less topaz, rutile, xenotime, and octahedrite.

The sample also contains a small amount of black radioactive material, a part of which is samarskite. The remainder of this material has been identified as fergusonite, according to Dana's Textbook of Mineralogy essentially a metacolumbate (and tantalate) of yttrium with erbium, cerium, and uranium. X-ray analyses of a pure concentrate of the recrystallized material confirmed the identification.

Studies and analyses of a pure concentrate of the altered zircon indicate that it can be classed as the variety cyrtolite. Examination further showed that the mineral contains 2.5 percent uranium and 5.5 percent thorium and had a $\frac{Hf}{Zr+Hf} \times 100$ ratio of 4.6.

## Experimental Procedure

Representative samples of the columbite-bearing sands were sized on a 28-mesh screen and both the oversize and undersize were separately subjected to magnetic separations on the Stearns ring-type number 0 magnetic separator set at 1/4-inch air-gap spacing.

The ilmenite (1)[4] and some of the garnet were removed at an intensity of 1.2 amperes. A clean garnet concentrate (2) was removed at 2.2 amperes. Although all of the garnet was not susceptible, increasing the intensity to completely separate the garnet resulted in attracting more columbite than

4/ Numbers in parentheses refer to operations so designated on flowsheet, fig. 1.

garnet; this would appreciably lower the recovery of $(Cb, Ta)_2O_5$. A setting of 3.2 amperes on the ring-type separator was not enough to completely remove the columbite (3). Therefore, it was necessary to pass the remaining nonmagnetic fraction across the Stearns-Wetherill crossbelt type R number 1-B magnetic separator set at 1/4-inch air-gap spacing and 3.0 amperes for complete recovery of the columbite (3). High-grade samarskite concentrates (4) were produced by passing the nonmagnetic fraction over the crossbelt separator at a setting of 3.8 amperes. A final separation at 4.2 amperes removed monazite (5) from the nonmagnetic quartz, feldspar, and zircon. The nonmagnetic products were also observed to contain cassiterite, topaz, rutile, xenotime, and octahedrite. The nonmagnetic fractions were hydraulically classified, and the sized fractions were separately passed across the Deister-Overstrom one-quarter deck shaking table to produce a zircon-cassiterite concentrate and a quartz-feldspar tailing (7). Similar magnetic and gravity products were combined and submitted for quantitative X-ray fluorescent and chemical analyses.

Metallurgical data for this test work are shown in table 3 and a flowsheet in figure 1.

TABLE 3. - Metallurgical data from sizing, magnetic separation, and tabling of Dismal Swamp columbite-bearing black-sand concentrate

| Product | Weight, percent | Analysis, percent | | Distribution, percent | |
|---|---|---|---|---|---|
| | | $Cb_2O_5$ | $Ta_2O_5$ | $Cb_2O_5$ | $Ta_2O_5$ |
| Ilmenite (1) 1/ | 5.12 | 2.0 | <1.0 | | |
| Garnet (2) | 3.70 | 1.7 | <1.0 | | |
| Columbite (3) | 67.47 | 60.0 | 6.0 | 88.00 | 88.00 |
| Samarskite (4) 2/ | 6.14 | 23.0 | 2.3 | 3.07 | 3.07 |
| Monazite (5) 3/ | 3.49 | 1.4 | <1.0 | | |
| Zircon (6) 4/ | 5.58 | <.5 | <1.0 | | |
| Quartz and feldspar (7) | 8.50 | <.5 | <1.0 | | |
| Assay head | 100.00 | 46.0 | 4.6 | | |

1/ Numbers in parentheses refer to products made in operation, which are similarly numbered on flowsheet, fig. 1.

2/ Also analyzes 2.25 percent uranium.

3/ Also analyzes 50.8 percent REO+Th.

4/ Also analyzes 6.37 percent tin, and 59.3 percent ZrO in which the $\frac{Hf}{Hf+Zr}$ x 100 ratio is 4.6.

## Conclusions

The columbite and samarskite present in the alluvial black sands from Dismal Swamp, Idaho, were readily concentrated by sizing, gravity, and magnetic separation. As much as 88.0 percent of the columbium was recovered in a columbite concentrate with an analysis of 66.0 percent $(Cb, Ta)_2O_5$. Appreciable quantities of samarskite, monazite, and zircon concentrates produced also would be valuable byproducts in the economic consideration of this black-sand sample.

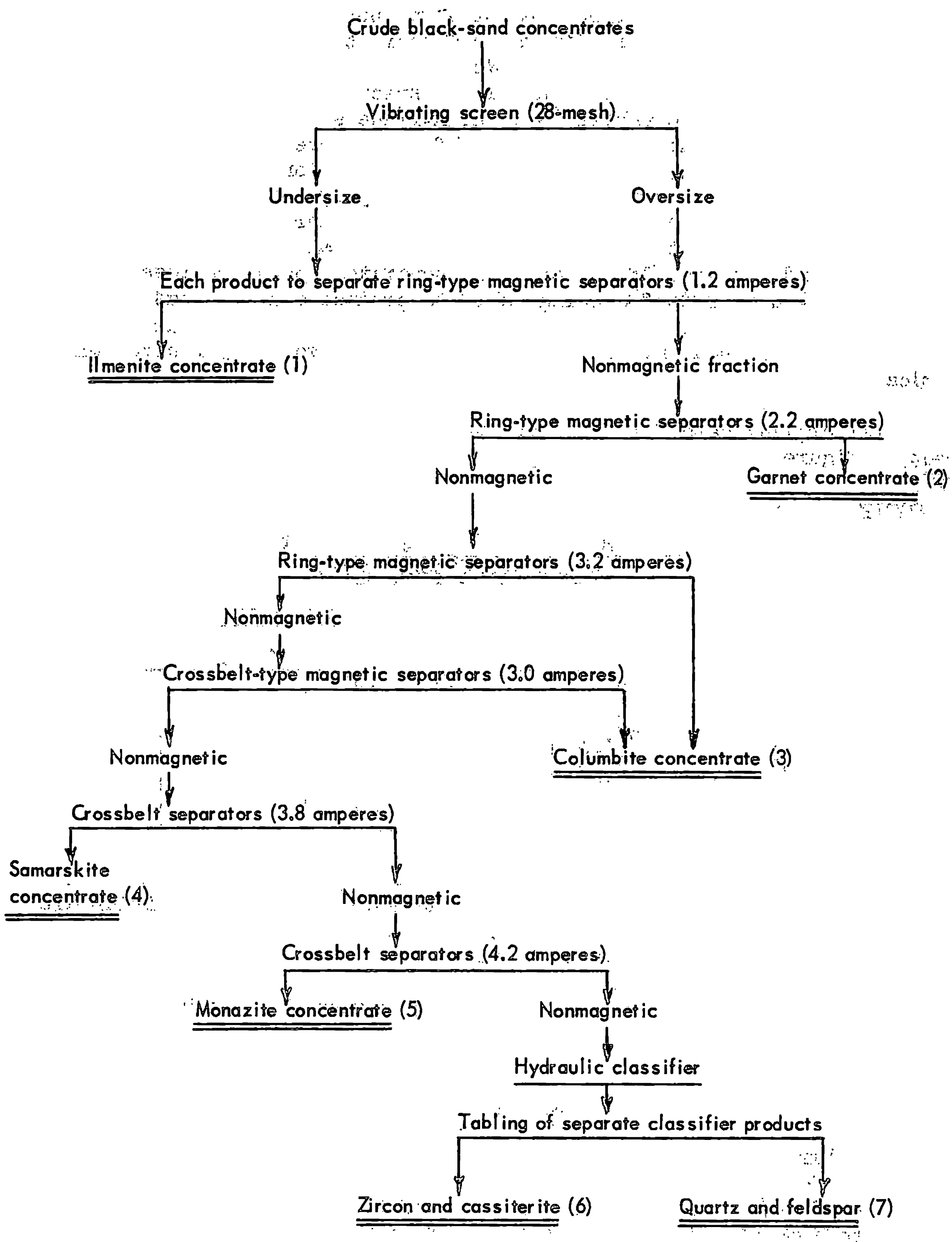


Figure 1. - Test flowsheet for Dismal Swamp black-sand concentrate.

# BEAR VALLEY

## Summary

The Bear Valley placer deposits have been estimated from drilling tests to contain substantial quantities of euxenite. Minus-16-mesh gravity concentrated black sands from these placer deposits were tested in the laboratory. The original gravel had been jigged and tabled to produce a heavy black-sand fraction free from light sand and slimes.

The Bear Valley black sands are a complex mixture of many minerals having closely related magnetic susceptibilities, specific gravity, and electrostatic or high-tension conductivity. In general, electrostatic separation makes use of a process employing a charged field with little or no current flow and utilizes the attraction of particles of one charge toward an electrode of the opposite charge. High-tension separation, on the other hand, utilizes a very high rate of electrical discharge, with electron flow and gaseous ionization playing a major part. The minerals receive a spray discharge of electricity, which gives them a high surface charge attracting them to a grounded spinning rotor. The particles with a relatively high conductivity will readily leak off this charge to the grounded rotor and be thrown from the rotor. These particles of a relatively low conductivity will be attracted and pinned to the rotor. Separations of this type will be referred to in this report as high-tension separation.

About 85 percent of the columbium-tantalum-bearing minerals can be recovered, but a spectrographic analysis of handpicked ilmenite grains showed the ilmenite mineral to contain 0.80 percent $Ta_2O_5$ and 0.32 percent $Cb_2O_5$. Owing to this condition, 40 to 50 percent of the combined oxides are lost in the ilmenite, which contains about 0.60 percent $(Cb, Ta)_2O_5$.

Test work demonstrated that attrition scrubbing, roasting, and magnetic and high-tension separation were effective methods in concentrating the euxenite, ilmenorutile, and columbite minerals. Preconditioning the scrubbed concentrates with HCl and benzoic acid changed the mineral surfaces and mineral response to an electric field. The euxenite and ilmenorutile then acted as conductors during high-tension separation. Heat treating this material produced the same results, and also had a pronounced effect on many of the minerals by increasing their magnetic susceptibilities. It was demonstrated that roasting the material, following removal of the magnetite, titaniferous magnetite, ilmenite, and garnet, was of definite benefit in obtaining cleaner magnetic separations with higher recovery. Roasting of this small fraction, representing about 9 percent of the original weight of the heavy sands, resulted in a product, concentrated by magnetic and high-tension separation methods, with an analysis of 26.4 percent $(Cb, Ta)_2O_5$, 3.48 percent $U_3O_8$, 17.3 percent $REO+ThO_2$, and an overall recovery of 47.69 percent, that represented 1.12 percent of the original weight.

## Beneficiation of Bear Valley Black Sands

### Nature of Ore

The sample was submitted by Mr. R. H. Storch, mining engineer, Bureau of Mines, Region II. Representative portions were prepared and submitted for the analyses shown in tables 4 and 5.

TABLE 4. - Chemical analyses

| $Cb_2O_5$ | Fe | $TiO_2$ | $U_3O_8$ | $REO+ThO_2$ |
|---|---|---|---|---|
| 0.62 ... | 48.6 | 14.85 | 0.08 | 0.03 |

TABLE 5. - Spectrographic analysis

| Constituents | Percent |
|---|---|
| Fe .................. | Over 10 |
| None ................ | 5 to 10 |
| Si, Ti .............. | 1 to 5 |
| Mn, U, Cb ........... | 0.1 to 1 |
| Ca, Cu, Zr, Th ...... | 0.01 to 0.1 |
| Mg .................. | 0.001 to 0.01 |
| $Cb_2O_5$ ............... | 0.61 1/ |
| $Ta_2O_5$ ............... | <0.5 1/ |

1/ Quantative X-ray fluorescent analysis.

A representative sample of the ore was examined in the petrographic laboratory and was found to contain magnetite, titaniferous magnetite, some ilmenite, garnet, sphene, quartz, feldspar, small amounts of euxenite, hornblende, allanite, biotite, epidote, limonite, hematite, sericite, ilmenorutile, and very small amounts of monazite, columbite, spinel, rutile, zircon, octahedrite, and pyrite.

Handpicked concentrates of the tantalum-solumbium-bearing minerals were submitted for quantative X-ray spectrographic analyses. The results of these analyses are shown in table 6.

TABLE 6. - X-ray spectrographic analyses

| Minerals | $Cb_2O_5$ | $Ta_2O_5$ | U | Th | Y | La | $CeO_2$ | $ErO_2$ | $Dy_2O_3$ | Fe |
|---|---|---|---|---|---|---|---|---|---|---|
| Euxenite ..... | 24.0 | 2.0 | 15.0 | 5.8 | 11.0 | 1.0 | 1.0 | 5.2 | 4.0 | 0.36 |
| Columbite .... | 58.0 | 14.0 | | | | | | | | |
| Ilmenorutile . | 18.0 | 11.0 | | | | | | | | |
| Ilmenite ..... | .32 | .80 | | | | | | | | |

## Discussion of Procedure

The tests have been directed toward beneficiating the radioactive black sands with studies to determine the effects of scrubbing, sizing, and magnetic and electrostatic separation on roasted and unroasted minerals, the principle aim being concentration of euxenite, ilmenorutile, columbite, and monazite. Studies were made also to determine the optimum roasting temperature, output voltage, and polarity of the electrostatic separator for separation of these minerals.

Examination of the black sands showed that only trace amounts of columbite and monazite were present and evaluation of the concentration of these minerals would be difficult because of the limited amount of black sands and the small-scale equipment available for test work.

Microscopic identification of the several columbium-tantalum bearing minerals is difficult even for the trained petrographer, and is accurate only with the petrographic microscope; therefore, in all test work the Tracerlab, 64 scaler Geiger counter was used to trace the path of the highly radioactive mineral euxenite. Ilmenorutile, essentially nonradioactive, having similar magnetic and high-tension properties, was concentrated with the euxenite. It was observed that, by correlation between the $(Cb, Ta)_2O_5$ analysis and radioactivity of the tantalum-columbium-bearing minerals, a reasonably accurate estimate could be made of the combined oxides content of any particular magnetic product.

## Procedure and Results

Microscopic examination of the black sands showed surface oxidation, and many of the mineral surfaces were iron stained. This considerably altered the magnetic properties of the minerals, and since electrostatic separation of the minerals is principally a surface phenomena, it was found necessary to clean the mineral surfaces before further treatment. The Wemco attrition-scrubber operated at 80 percent solids was used to clean the mineral surface as a preliminary step in all test work.

Since examination of the black sands showed they contain essentially magnetite, titaniferous magnetite, and ilmenite, the Davis tube or the Stearns wet-type magnetic separators were used to remove the magnetite and titaniferous magnetite from the minerals of lower magnetic susceptibility.

To determine the ratio of concentration obtained by magnetic separation, representative samples of the Bear Valley sands were attrition scrubbed and magnetite and titaniferous magnetite were separated from the remaining minerals. The nonmagnetic fraction was sized on 28- and 48-mesh screen, and separate sized fractions were passed across the Stearns ring-type number 0 magnetic separator, with the air-gap spacing set at 3/8 inch, at increasing intensities from 1.1 to 3.5 amperes. The nonmagnetic fractions from the ring separator were passed across the Stearns Wetherill crossbelt type R number 1-B magnetic separator set at 3/8-inch air-gap spacing and 4.0 amperes. Similar magnetic products for each size fraction were combined and submitted for X-ray fluorescent analyses. The combined concentrates had an analysis of 4.55 percent $(Cb, Ta)_2O_5$, with an overall recovery of 30.01 percent and a concentration ratio of 7.34:1.

Comparison tests using heat treatment on the small weight fraction remaining after removal of the magnetite, titaniferous magnetite, ilmenite, and garnet, by magnetic separation demonstrated that an increase in recovery and selectivity was obtained during magnetic separation when roasting was employed.

A representative sample of the black sands was treated by removing the magnetite and titaniferous magnetite by wet magnetic separation and the bulk of the ilmenite and garnet by a ring-type magnetic separator.

The final nonmagnetic fraction from this procedure was given an oxidizing roast for 2 hours at $800^\circ$ C. in an electrically heated muffle furnace. It is doubtful if this roast would require 2 hours, but time and material did not permit an investigation of this test variable. The roasted product was then sized on 28- and 48-mesh screens, and the separate size fractions were passed across the ring separator set at 3/8-inch air-gap spacing and 2.8 amperes to remove the remaining ilmenite and garnet.

After roasting, the ilmenite could be removed at a lower intensity, thus having little effect on the columbium-tantalum-bearing minerals. This resulted in a cleaner separation at increased recovery. The nonmagnetic fractions were repassed at 3.4 amperes to separate the euxenite, ilmenorutile, epidote, and allanite from the minerals of lower susceptibility.

The combined euxenite-ilmenorutile rich product had an analysis of 4.12 percent $(Cb, Ta)_2O_5$, with a recovery of about 50.0 percent. In an attempt to further upgrade this material it was passed across the Deister-Overstrom one-quarter deck shaking table to produce a rougher concentrate. Each product was submitted for X-ray fluorescent analyses. The combined table concentrates had an analyses of 9.6 percent $(Cb, Ta)_2O_5$ with an overall recovery of 15.33 percent at a concentration ratio of 15.3:1.

The minerals in this black-sand sample were observed to have the approximate order of decreasing magnetic susceptibility as follows: Magnetite, titaniferous magnetite, ilmenite, columbite, garnet, epidote, ilmenorutile, euxenite, allanite, hornblende, biotite, sericite, sphene, monazite, and pyrite, with spinel, octahedrite, rutile, zircon, quartz, and feldspar being nonmagnetic at the intensities used. Hematite and limonite psuedomorphs were observed in all of the higher intensity magnetic fractions.

It was noted that the small amount of columbite present in this sample was susceptible at the highest intensities required for complete removal of the ilmenite. After roasting the material the intensities required for complete removal of ilmenite and garnet were lowered. Examination of these magnetic products showed that columbite was not affected as much by roasting as the ilmenite and garnet; therefore, columbite reported in the high garnet fraction.

Since the columbite and ilmenite both act as conductors, whereas the garnet is a nonconductor, it was advantageous to collect the columbite with the garnet. This would permit a separation of the columbite from the garnet by electrostatic methods.

Results of the test work showed that magnetic separation and tabling of this complex ore were not adequate in producing a high-grade $(Cb, Ta)_2O_5$ product commensurate with good recovery, because the closeness of magnetic susceptibility and specific gravity prohibited clean separations of any minerals.

After scrubbing and removal of magnetite and titaniferous magnetite, electrostatic separations were made with the broad beam large diameter electrode at 10,000 volts, a rotor speed of 40 r.p.m., an air-gap spacing of sufficient distance to prevent arcing between electrode and rotor, and the output electrode either positive or negative. The separations of euxenite, ilmenorutile, columbite, and ilmenite, from epidote, garnet, sphene, and quartz were not successful in these tests.

Separations were made on representative samples of the black sands, after scrubbing and removing magnetite and titaniferous magnetite for 1 sample and scrubbing and removing magnetite, titaniferous magnetite, ilmenite, and garnet for the other sample, with the broad beam wire electrode set at a positive output voltage of 20,000 volts, a rotor speed of 500 r.p.m., an air-gap spacing sufficient to prevent arcing, and a circulating load of 70 to 80 percent. Examination of the conductor and nonconductor fractions indicated that the euxenite was divided between both fractions, and repassing of each fraction to produce cleaner products resulted in resplitting the euxenite. The conductor and nonconductor fractions in each test were passed across the ring separator at increasing intensities, and the euxenite-ilmenorutile-rich products were tabled to produce final concentrates. Changing the polarity of the electrode had little effect on the separations.

Results of the test in which the magnetite and titaniferous magnetite were removed before separation showed that a final concentrate could be made that had an analysis of 19.5 percent $(Cb, Ta)_2O_5$, with an overall recovery of 13.7 percent. The test in which the ilmenite and garnet were removed before separation produced a final product that recovered 10.1 percent $(Cb, Ta)_2O_5$, with an analysis of 25.0 percent $(Cb, Ta)_2O_5$.

After attrition scrubbing and removal of the magnetite, titaniferous magnetite, ilmenite, and garnet by magnetic separation, the nonmagnetic fraction was roasted at 700° C. and passed across the electrostatic separator with a setting the same as in the previous test. It was observed by microscopic examination and the radioactivity of the products, that roasting had, by altering the surface, made the euxenite a conductor to electrostatic forces. The conductor fraction was passed across the ring-type separator at intensities of 2.5 and 3.5 amperes. A sample of the product, nonmagnetic at 2.5 amperes and magnetic at 3.5 amperes, was found to contain 44.55 percent of the $(Cb, Ta)_2O_5$ with an analysis of 19.3 percent $(Cb, Ta)_2O_5$. This product was passed across the laboratory shaking table to produce a final concentrate representing 0.63 percent of the original weight, with an analysis of 32.2 percent (Cb, Ta)$_2$O$_5$ and an overall recovery of 32.9 percent.

Tests were then conducted to determine the optimum roasting temperature. Representative samples were attrition scrubbed, treated by wet magnetic separation to remove the magnetite and titaniferous magnetite, and then passed across a ring separator set at 2.8 amperes to remove the bulk of the ilmenite and garnet. Similar nonmagnetic fractions were roasted at 800°, 900°, and 1,000° C. to determine the effects of the various roasting temperatures. Roasting at 1,000° C. nodulized the sands, making mechanical separation impossible. The remaining two samples, which retained their granular form, were treated by electrostatic separation, with the output voltage set at 20,000 volts. The conductor and nonconductor fractions were cleaned once. The conductor fractions were passed across the ring-type separator at 2.5 amperes and 3.5 amperes. The products were submitted for X-ray fluorescent analyses. The sample that was roasted at 800° C. produced an euxenite-ilmenorutile concentrate that had an analysis of 26.5 percent (Cb, Ta)$_2$O$_5$ with an overall recovery of 45.81 percent and represented 1.07 percent of the original weight. A final product, representing 1.11 percent of the original weight, was produced from the sample that was roasted at 900° C., which had an analysis of 23.4 percent (Cb, Ta)$_2$O$_5$ with an overall recovery of 41.94 percent and a concentration ratio of 37.7:1.0.

The remaining material was prepared for a final test on a slightly larger scale. The sample of black sands was attrition scrubbed at 80 percent solids to remove a slime fraction (1),[4] which represented 3.23 percent of the sample weight. The sand fraction was treated on a wet-type magnetic separator to remove the magnetite and titaniferous magnetite (2), which was considered a final product and represented 43.85 percent of the weight. The remaining nonmagnetic fraction was dried and passed across a ring-type magnetic separator at 2.8 amperes to remove an ilmenite-garnet product (3). The nonmagnetic fraction from this step was then roasted at 800° C. and treated on a Carpco "high-tension" separator to produce conductor and nonconductor fractions. The final conductor portion was further treated on a ring-type magnetic separator to remove a second garnet-ilmenite product (4), with the nonmagnetic material re-treated at higher intensity to make a final euxenite-ilmenorutile product (5) and a nonmagnetic product.

Those minerals, which reported as nonconductors from the "high-tension" separator, were also treated on the ring-type magnetic separator to remove a garnet-ilmenite product (7) and a comparatively large nonmagnetic fraction, which contained minor amounts of euxenite and ilmenorutile. Re-treating this nonmagnetic product at higher intensity on a crossbelt magnetic separator removed most of the minerals in a final nonmagnetic product (10). The final magnetic concentrate was passed across a shaking table to scavenge the remaining columbium-tantalum-bearing minerals (8). A locked series test was not conducted, but it is proposed that the table concentrate be returned to the roasted material coming to the "high-tension" separator.

---

4/ Numbers in parentheses refer to products made in operation, which are similarly numbered on flowsheet, fig. 2.

The euxenite-ilmenorutile rich product, which was magnetically separated from the conductor fraction, represented 1.12 percent of the original weight and recovered 47.69 percent of the $(Cb, Ta)_2O_5$ at a concentration ratio of 42.58:1, with an analysis of 26.4 percent $(Cb, Ta)_2O_5$, 17.3 percent $REO+ThO_2$, and 3.48 percent $U_3O_8$. Examination and grain count showed this product to contain approximately 80 percent euxenite, 12 percent ilmenorutile, and 1 percent columbite. The flowsheet for this test is shown in figure 2 and the metallurgical data in table 7.

Test results showed that the ilmenite concentrates contained 40 to 50 percent of the combined columbium-tantalum oxides at an analysis of about 0.60 to 0.7 percent $(Cb, Ta)_2O_5$. Petrographic examination of this product showed that columbite was present, but not in sufficient quantity to account for the large amount of combined oxides. Further studies on a handpicked ilmenite concentrate showed the ilmenite mineral to contain 0.80 percent $Ta_2O_5$ and 0.32 percent $Cb_2O_5$. A sample of the ilmenite product was stage ground in the laboratory ball mill to determine whether any concentration of the combined oxides occurred in the various size fractions. Analyses of the products indicated that only a slight concentration of the $(Cb, Ta)_2O_5$ was made in the minus-200-mesh fraction. This concentration was too minor to warrant the extra treatment.

TABLE 7. - Metallurgical data from results of test work on Bear Valley black-sand concentrate

| Product | Weight, percent | Analyses, percent $Ta_2O_5$ | $Cb_2O_5$ | $(Cb, Ta)_2O_5$ | Units $(Cb, Ta)_2O_5$ | Distribution, percent $(Cb, Ta)_2O_5$ |
|---|---|---|---|---|---|---|
| Slimes (1)1/.... | 3.23 | <0.5 | 0.60 | | | |
| Magnetite (2) .. | 43.85 | <2/.5 | <.20 | | | |
| Ilmenite (3) ... | 44.68 | .40 | .20 | 0.60 | 0.26808 | 43.24 |
| Conductor | | | | | | |
| Ilmenite (4) ... | .92 | < .5 | 1.5 | | | |
| Eux-Ilmen (5) .. | 1.12 | 2.4 | 24.0 | 26.4 | .29568 | 47.69 |
| Nonmagnetic (6) | .77 | < .5 | 2.3 | | | |
| Nonconductor | | | | | | |
| Garnet (7) ..... | .22 | < .5 | 1.2 | | | |
| Table conc. (8) | .24 | 1.3 | 9.6 | 10.9 | .026378 | 4.25 |
| Table tail (9) . | 1.34 | < .5 | 1.4 | | | |
| Nonmagnetic (10) | 3.63 | < .5 | 1.0 | | | |
| | 100.00 | | | | | |

1/ Numbers in parentheses refer to operations so designated in fig. 2.
2/ A sample of magnetite was submitted for spectrographic analyses. Tantalum was reported as not detected and columbium present from 0.01 to 0.1 percent. Owing to difficulty of accurate determinations of $Ta_2O_5$ and $Cb_2O_5$ in the lesser amounts, analyses were reported as less than 0.5 and less that 0.2 percent.

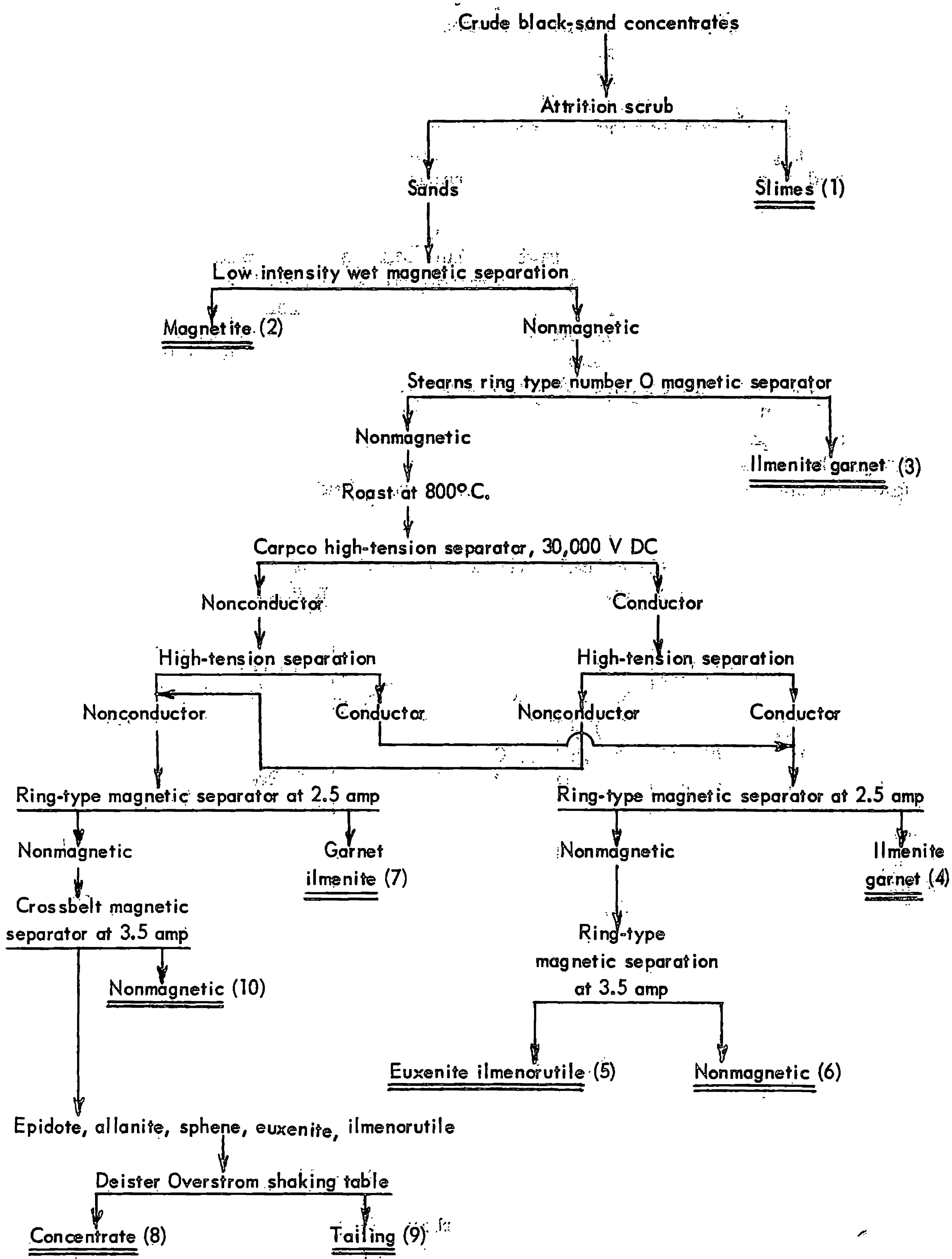


Figure 2. - Flowsheet for test work on Bear Valley black-sand concentrate.

## Conclusions

Tests showed that magnetic separation, combined with gravity concentration, was not adequate in concentrating the euxenite and ilmenorutile from the complex alluvial black sands from the Bear Valley, Idaho, placer sample. Electrostatic separation was required to obtain sufficient grade and recovery of the columbium-tantalum-bearing minerals.

It was demonstrated that a slightly oxidizing roast at 800° C. was beneficial in treating this black-sand sample. Heat treating the minerals permitted cleaner separations by magnetic methods and also changed the surface conductance and mineral response to an electric field.

Owing to the ratio of concentration required to effect an appreciable upgrading of the columbium-tantalum-bearing minerals, and also the limited amount of material with which to work, it was not possible to recycle middling products to determine accurately results and recoveries that may be obtained on a large-scale, continuous operation.

It may be concluded that, although a good recovery of the main columbium-tantalum-bearing minerals, approximately 84 percent of the euxenite and 90 percent of the ilmenorutile, could be obtained, the overall recovery of the combined oxides $(Cb, Ta)_2O_5$ was low. This was due primarily to the large amount of ilmenite present in the ore, which contained 0.60 percent $(Cb, Ta)_2O_5$ and represented 45 percent of the total weight. Grinding the ilmenite product to a minimum size for mechanical separations did not appreciably increase mineral recovery.

It is believed by the authors that treatment methods, other than physical, will be required to recover the remaining $(Cb, Ta)_2O_5$ from this low-grade material.

## CASCADE

### Summary

The Baumhoff-Marshall Co. at Boise, Idaho, has been treating a placer deposit at Cascade, Idaho, to recover monazite. The rougher ilmenite concentrate, obtained by gravity and magnetic procedures, had been observed to contain minor amounts of monazite and radioactive opaque minerals. Owing to the small amount present, accurate determination of the actual mineral content of this product had not been made. However, because of the interest in columbium-tantalum-bearing minerals, it was requested that a sample of the ilmenite concentrate be treated as an aid in determining what minerals were present and to what extent they could be concentrated.

The high ratio of concentration required made it necessary to treat a larger amount of material than could be handled by laboratory equipment. Therefore arrangements were made with Porter Brothers Corp. at Boise, Idaho, to treat a 2,500-pound sample of the ilmenite concentrate from Baumhoff-Marshall in the pilot plant. The treatment procedure was to follow the method used for treating the Bear Valley black sands.

A representative portion of each separation product was returned to the Albany laboratory for evaluation.

## Procedure and Results

The 2,500 pound sample was attrition scrubbed in an amalgam barrel at approximately 80 percent solids. No grinding medium was used and the slime fraction, which represented about 1.0 percent of the weight, was removed by hydraulic classification. The sands were dried at 250° F. and subjected to low-intensity magnetic separation on a Dings fixed magnet roll at a feed rate of 465 pounds per hour. The magnetic fraction weighed 17.5 pounds and was considered a finished product.

The low intensity nonmagnetic product was further treated on an 18-inch Carpco induced roll separator, operating at 1.0 amps with a 7/32-inch air-gap spacing to remove the ilmenite. Each product was recycled for cleaning and the nonmagnetic fraction, which weighed 322.5 pounds, was heated to 265° F. for treatment by "high-tension" separation.

The Carpco "high-tension separator" was operated on a positive setting at 26,000 volts, using a beam-type electrode. The speed of the 18-inch rotor was 100 r.p.m. and the feed rate 51.0 pounds per hour. The conductor and nonconductor fractions were cleaned to remove a high-grade monazite product, which weighed 14.88 pounds. That portion of material that acted as a conductor was essentially ilmenite, less amounts of garnet and monazite, with minor amounts of quartz, and a mineral that appeared to be leucoxene. This conductor portion was then passed across a Stearns cross-belt magnetic separator operating at 3.8 amps with a feed rate of 50.0 pounds per hour. Seven magnetic products were removed by this machine, and in the usual treatment procedure these products are stockpiled for future re-treatment. The nonmagnetic fraction from the crossbelt separator weighed only 1,005.0 grams, and although some euxenite was observed, it was present in only minor amounts. Later petrographic study determined the essential columbium-bearing mineral in this product to be ilmenorutile.

For further up-grading the nonmagnetic material was hydraulically classified and treated on a one-quarter deck Deister Overstrom table. All products were recycled to produce a final table concentrate and table tailing. The table concentrate would, under the regular treatment procedure, be the end product. However, owing to the minor amounts of euxenite and columbite present, it was decided to attempt further concentration by re-treating the dried table concentrate on the "high-tension" separator. This treatment produced a conductor product weighing 239 grams, a small middling, which weighed 8.0 grams, and a nonconductor of 175.0 grams, which appeared to contain only minor amounts of euxenite.

Detailed analyses are given only for the significant products; the great bulk of the ilmenite was removed, and contained a negligible portion of the valuable minerals.

Metallurgical data of this procedure are shown in table 8.

TABLE 8. - Metallurgical data

Twenty-five hundred pounds of an ilmenite concentrate from Cascade, Idaho, black sands were treated in the Porter Brothers pilot plant by: (1) Separation of the magnetite using a fixed magnetic roll, (2) removal of ilmenite by treatment on an induced-roll magnetic separator, (3) separation of monazite by "high-tension" treatment, (4) rejection of remaining ilmenite by use of a crossbelt magnetic separator, (5) removal of leucoxene pseudomorph and other light minerals by subjection to hydraulic classification and tabling, and (6) separating monazite and leucoxene from the table concentrate by final "high-tension treatment" to produce (7) final columbium-bearing concentrate and (8) middling.

| Product | Weight, percent | Analyses, percent | | | | Mineral | Percent |
|---|---|---|---|---|---|---|---|
| | | $Cb_2O_5$ | $Ta_2O_5$ | REO+Th | U | | |
| Final columbium concentrate (7)1/ ...... | 0.021 | 26.0 | 10.0 | 0.60 | 1.1 | Ilmenorutile. | 55.0 |
| | | | | | | Euxenite. | 15.0 |
| | | | | | | Columbite. | 10.0 |
| | | | | | | Monazite. | 1.0 |
| Final "high-tension" nonconductor (6) .... | .015 | 1.4 | .74 | 45.2 | .5 | Monazite. | 70.0 |
| | | | | | | Euxenite. | 3.0 |
| | | | | | | Ilmenorutile. | Trace |
| | | | | | | Columbite. | Do. |
| | | | | | | Leucoxene. | Remainder |
| Table tailing (5) .... | .050 | 1.5 | .92 | .52 | .5 | Ilmenorutile. | 5.0 |
| | | | | | | Monazite. | 2.0 |
| | | | | | | Euxenite. | 1.0 |
| | | | | | | Columbite. | 1.0 |
| Final "high-tension" middling (8) ........ | .0007 | Amount too small to assay | | | | Euxenite. | 20.0 |
| | | | | | | Monazite. | 17.0 |
| | | | | | | Columbite. | 1.0 |
| | | | | | | Ilmenorutile. | 1.0 |
| | | | | | | Allanite. | 20.0 |
| | | | | | | Leucoxene. | 20.0 |

1/ 0.42 pounds of this product per ton of original feed.

## Conclusions

Treatment of the ilmenite concentrate obtained from the Cascade, Idaho, black sands by the Porter Bros. plant procedure indicated that approximately 0.42 pounds of a columbium-bearing product per ton of the rougher ilmenite concentrate can be recovered from this material. This final columbium-bearing product assayed 26.0 percent $Cb_2O_5$, 10.0 percent $Ta_2O_5$, 1.1 percent U, and less than 0.5 percent Th. Part of the euxenite was recovered as a middling from the final high-tension separation. This middling product weighed only 8.0 grams and by quantative petrographic analysis contained approximately 20 percent each of euxenite, monazite, allanite, and leucoxene.

It was noted that the smaller laboratory-scale equipment does not give as good results on the low-grade black sand as the larger equipment, such as is used by Porter Brothers plant at Boise, Idaho. This is particularly true of the magnetic separations and especially of the crossbelt magnetic separators. The larger equipment reduces the proportion of material mechanically carried over and appears, at some stages, to give cleaner separations.