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Tin and Silver Recovery From Coal Creek, AK

By J. L. Johnson and T. Parker

UNITED STATES DEPARTMENT OF THE INTERIOR

BUREAU OF MINES

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

A	ampere	min	minute
°C	degree Celsius	mL	milliliter
cm	centimeter	μm	micrometer
ft	foot	mt	metric ton
ft³	cubic foot	pct	percent
g	gram	ppm	part per million
h	hour	psi	pound per square inch
hp	horsepower	rpm	revolution per minute
in	inch	s	second
kW·h/st	kilowatt hour per short ton	st	short ton
lb	pound	st/d	short ton per day
lb/h	pound per hour	st/h	short ton per hour
lb/st	pound per short ton	tr oz/st	troy ounce per short ton
m	meter	wt pct	weight percent
m²	square meter		

TIN AND SILVER RECOVERY FROM COAL CREEK, AK

By J. L. Johnson[1] and T. Parker[2]

ABSTRACT

The U.S. Bureau of Mines investigated the recovery of tin and silver from the Coal Creek deposit in the Talkeetna Mountains, AK. Approximately 5 million st of reserves grading 0.2 pct Sn with silver credits of 0.2 tr oz/st have been delineated by drilling. A 10- to 25-pct Sn gravity concentrate was produced by treating the minus 20-mesh ore with a spiral, then regrinding to minus 65 mesh and tabling. Sulfide contamination in the gravity concentrate was as high as 60 pct. Sulfide flotation of this concentrate produced tailings containing 40 to 50 pct Sn. Overall recovery was 76 pct for tin but only 5 to 10 pct for silver because of losses during sulfide flotation. Other methods tested to clean the concentrate included cassiterite flotation, gangue flotation, nitric acid leaching, and wet and dry magnetic separation. Tin beneficiation tests on the minus 325-mesh fraction included cassiterite flotation, vanning, sulfide flotation, and fuming. The bench-scale and locked-cycle tests were used to estimate the mass flows for a 1,000-st/d plant.

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INTRODUCTION

The U.S. Bureau of Mines is investigating the mineral potential of Alaska and the Pacific Northwest in order to reduce the dependence of the United States on imports, particularly for materials that are critical to the Nation's economy and strategic capabilities. The United States currently imports 75 pct of the tin consumed each year; the other 25 pct is derived from recycled scrap, solder, brass and bronze, and secondary tin-bearing materials (1).[3] To guarantee the availability of tin during national emergencies, the Department of Defense maintains a stockpile of 190,000 mt (209,400 st) of tin metal, the world's largest readily available supply. The stockpile ensures an immediate supply, but a long-range source would be in question.

The Coal Creek prospect is located in the northwesternmost portion of the Talkeetna Mountains, AK (fig. 1). Approximately 5 million st of geologically inferred reserves grading 0.2 pct Sn with silver credits of 0.2 tr oz/st have been delineated by drilling.

This report describes the response of Coal Creek ore samples to different concentration apparatus. Bench-scale and locked-cycle tests were used to develop a preferred flowsheet, and the mass flow was calculated for a 1,000-st/d mill.

ORE SAMPLE CHARACTERIZATION

The Coal Creek tin prospect is located on the northwest side of the Chulitna River Valley in the Talkeetna Mountains. There are no roads to the immediate area, although the Parks Highway lies on the opposite side of the Chulitna River 5 miles to the southeast. The rock exposure of the deposit is relatively poor and consists of only about 4,000 m² of mineralized greisen-altered granite. The granite intrudes Devonian metasediments of the Chulitna sequence, locally producing hornfels and skarn. A cross section of the mineralized zone is illustrated in figure 2.

The granite can be divided into two distinct textural units: (1) a seriate granite porphyry, which is intruded at depth by (2) a very evolved, originally volatile-rich, fine-grained equigranular granite. Both granites contain quartz, potassium feldspar, albite-rich plagioclase, biotite, white mica, and possibly tourmaline. In most places, the seriate granite has been altered to an assemblage of quartz and white mica.

The contact between the two granites is marked by a 1- to 3-m-thick zone of crenulate and dendritic layers composed of potassium feldspar, quartz, and mica. Locally, above and below this contact zone, mineralization is concentrated in a caplike mass or cupola of highly fractionated seriate and equigranular granitic rock. Mineralization is characterized by varying degrees of greisen alteration and veining.

The tin and silver mineralization is associated with near-vertical 0.5- to 1-cm greisen veins that have 3 to 5 cm silicified alteration envelopes. These veins are concentrated in the upper seriate granite and extend only 10 to 20 m into the lower, and younger, equigranular granite. A small number of veins extend into the hornfels. The minerals that make up these veins are quartz, white mica, fluorite, topaz, blue to green tourmaline, sillimanite, gahnite, cassiterite, marmatite (iron-rich sphalerite), chalcopyrite, pyrrhotite, pyrite, marcasite, arsenopyrite, loellingite, stannite, galena, bismuthinite, and silver sulfosalts.

A petrographic and electron microprobe study of the sulfide mineral assemblage has shown that stannite may represent as much as 3 pct of the total tin-bearing minerals. Stannite generally occurs with or in marmatite and chalcopyrite as rims or small exsolution blebs. The bulk of the silver is contained in stannite and in 2- to 5-μm galena grains. Because of the small grain size of galena, it was identified only with the electron microprobe.

Cores from four diamond drill holes were shipped to the Bureau for metallurgical testing. The samples received representing drill holes 19 and 32 are composed of nearly equal amounts of greisen mineralization from both the seriate and the equigranular granites. Those intervals that represent the seriate granite exhibit a higher degree of surface oxidation or weathering than do the intervals selected from the lower equigranular unit. Composites DH 1 and DH 3 were made of part of the equigranular granite from drill-hole cores 19 and 32, respectively. The

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[3]Italic numbers in parentheses refer to items in the list of references at the end of this report.
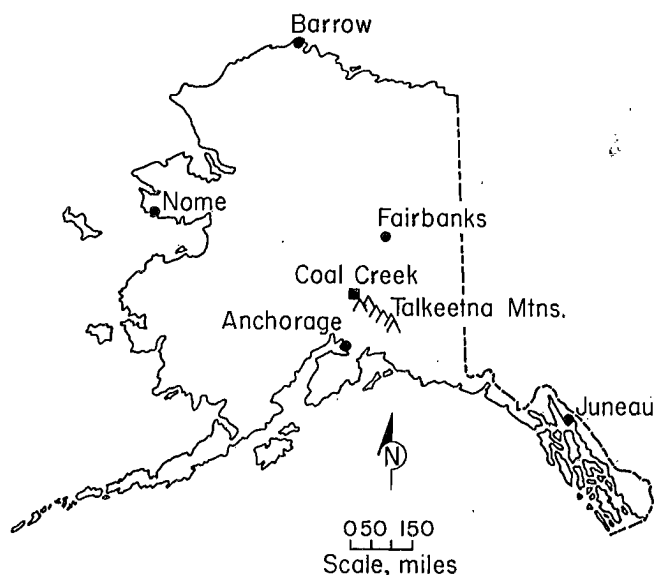
Figure 1.—Map of Alaska showing Coal Creek location.

bulk of the material received from drill-hole core 20 is from the more weathered seriate granite, and a composite, DH 2, was made from this bulk portion of the core. A composite, DH 4, was made from the mineralized rock received for drill-hole core 33, which was within 180 ft of the surface and wholly within the seriate granite.

The oxidation of sulfide minerals—for example, arsenopyrite—results in the transport of arsenic and redeposition of arsenic as arsenic oxide mineral grains and as coatings on silicate minerals. The new oxide grains are lower in density and likely smaller than the original sulfide grains. The density of arsenic-oxide-coated silicate grains is about the same as that of noncoated grains. The significance of this weathering exhibits itself in the physical beneficiation, where the weathered seriate granite samples concentrate less of the arsenic, as discussed later, in the section "Primary Concentration." The degree of weathering might also affect the recovery of the silver.

A profile of the heavy minerals of each composite, determined by examining a pan concentrate using a

LEGEND

Equigranular granite

Contact zone pegmatite

Seriate granite

Devonian metasediments

--?- Geologic contact, dashed where inferred

~~Shear zone

Drill hole (DH)

□ 0.1-0.2 pct Sn

■ >0.2 pct Sn
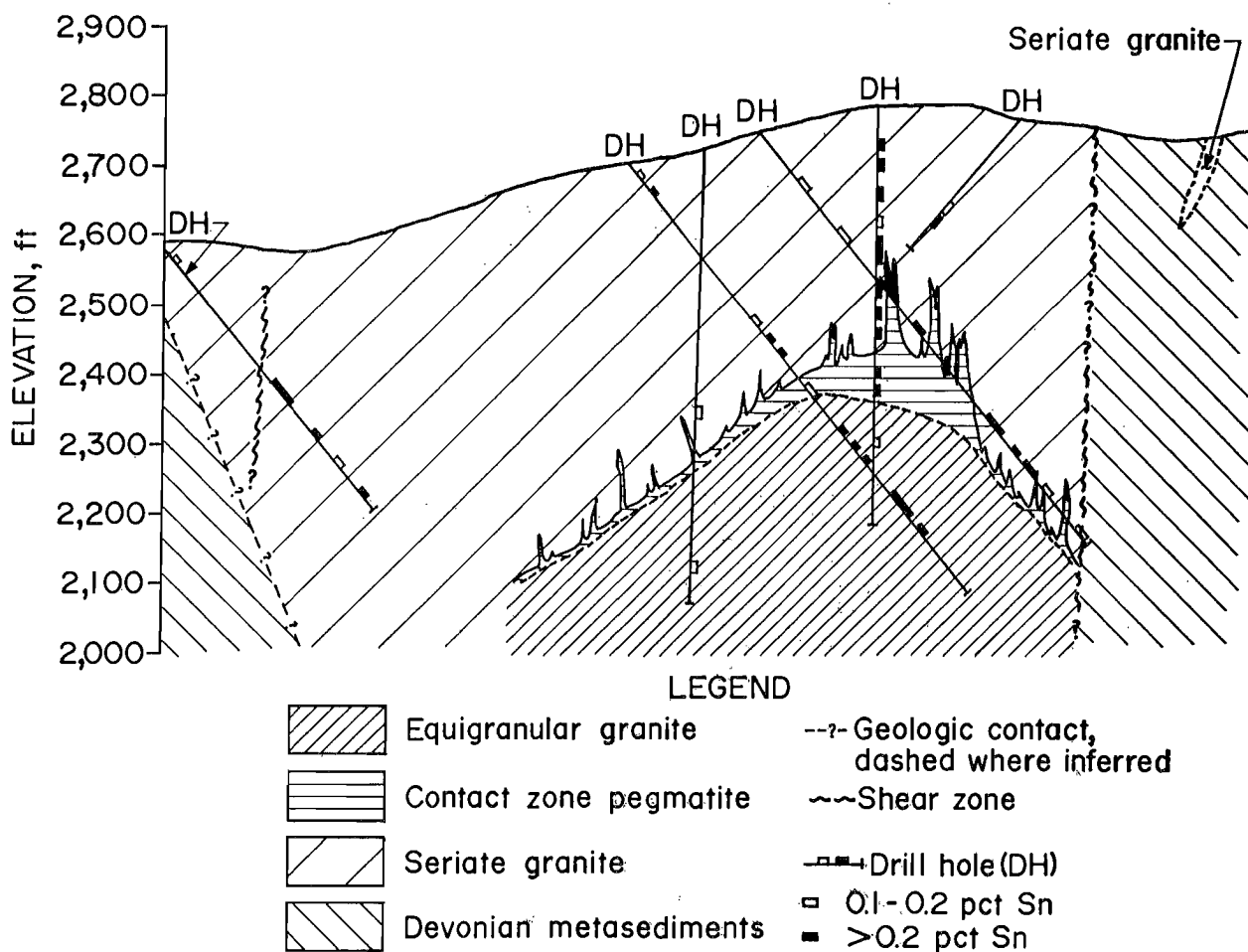
Figure 2.—Cross section of Coal Creek mineralization zone.

scanning electron microscope (SEM) and X-ray fluorescence (XRF), is given in table 1. The heavy minerals include several sulfides that are more abundant than cassiterite, the tin mineral. The sulfides' densities (specific gravity 4.0 to 6.2) are just below that of cassiterite (7.0); therefore, any gravity beneficiation concentrate would include sulfides, particularly arsenopyrite (specific gravity 6.2). The Bond work index was 10.8 kW·h/st, which was determined from a composite of the four drill-hole cores.

The head assays, given in table 2, show that tin is the primary value, followed by silver. The elements zinc, iron, and arsenic were used to track the response to the metallurgical testing of the sulfide minerals marmatite, pyrite, and arsenopyrite, respectively. Although iron is found in many of the other minerals (such as marmatite, which assays 43 pct Zn, 42 pct S, 13 pct Fe, and 1.3 pct Mn), as well as in nonsulfide minerals, it was used as the indicator of pyrite.

Table 1.—Mineral content of pan concentrates from Coal Creek
drill-hole composites, in order of occurrence

Sample	Most abundant	Major	Minor
DH 1 ..	Quartz, K-feldspar ..	Pyrite, marmatite, cassiterite.	Topaz, fluorite, tourmaline, mica, chalcopyrite, arsenopyrite, iron oxide.
DH 2 ..	.. do.	Marmatite, cassiterite,	Arsenopyrite, chalcopyrite, pyrite.mica, iron oxide, bismuthinite, galena, tourmaline, topaz, fluorite.
DH 3 ..	Quartz, K-feldspar, topaz.	Pyrite, marmatite, chalcopyrite, cassiterite.	Tourmaline, fluorite, mica, plagioclase, arsenopyrite, galena, bismuthinite.
DH 4 ..	Quartz, K-feldspar, topaz, fluorite, mica.	Cassiterite	Arsenopyrite, chalcopyrite, pyrite, marmatite, iron oxide, jarosite.

Table 2.—Head assays for Coal Creek drill-hole composites, percent

Sample	Sn	Ag[1]	Zn	Fe	As	Cu	WO₃
18DH 1 . . .	0.14	0.2	0.3	2.0	NA	0.016	0.004
DH 2	.18	.1	.06	1.4	0.04	.007	.004
DH 3	.19	.3	.29	2.8	.05	.06	.006
DH 4	.09	.2	.02	3.2	.13	.011	.003

NA   Not analyzed.
[1]Grade given in troy ounce per short ton.

BENEFICIATION FEASIBILITY STUDY

Beneficiation by gravity techniques is the most common method used to process tin ores. For the Coal Creek ore to be amenable to this method, two basic requirements must be met. A significant difference must exist between the densities of the gangue and the valuable minerals. For the Coal Creek samples, the specific gravity is 3 for most of the gangue, and ranges from 4 to 6 for the sulfides. This compares with a specific gravity of 7 for the cassiterite, which shows that gravity techniques could separate most of the gangue, if the next requirement is met. The other requirement is that the valuable mineral needs to be able to be liberated from the gangue at a size large enough for effective separation by the designated apparatus. The liberation size of the cassiterite and silver was therefore the first characteristic of the ore samples to be determined.

MINERAL LIBERATION SIZE

The liberation size of the cassiterite and silver minerals was determined by screening a crushed sample into size intervals, then hand panning screen fractions between 10 and 150 mesh and analyzing the concentrates and tailings. A liberation step is represented by the size at which a large decrease occurs in the tail grade or a large increase occurs in the recovery over that of the next larger size. Results of the analyses are given in table 3.

Table 3.—Analysis of hand-panned products from composite sample screen fractions, percent

Size fraction Tyler mesh	Sn			Ag		
	Concentrate		Tail	Concentrate		Tail
	Grade	Recovery	grade	Grade[1]	Recovery	grade[1]
DH 1 COMPOSITE						
Minus 10 plus 14	0.77	58	0.054	0.14	9	0.14
Minus 14 plus 20	.86	60	.063	.14	10	.14
Minus 20 plus 28	1.03	67	.066	.14	12	.14
Minus 28 plus 35	.63	79	.046	.14	21	.14
Minus 35 plus 48	1.58	90	.042	.29	33	.14
Minus 48 plus 65	1.32	89	.036	.29	31	.14
Minus 65 plus 100 ...	.86	87	.021	.44	34	.14
Minus 100 plus 150 ..	1.11	93	.015	.44	37	.14
DH 2 COMPOSITE						
Minus 10 plus 14	0.60	50	0.11	<0.1	NC	<0.1
Minus 14 plus 20	.56	35	.14	<.1	NC	<.1
Minus 20 plus 28	.95	77	.08	<.1	NC	<.1
Minus 28 plus 35	1.23	75	.08	<.1	NC	<.1
Minus 35 plus 48	1.52	87	.05	.1	NC	<.1
Minus 48 plus 65	.70	92	.04	.1	NC	<.1
Minus 65 plus 100 ...	1.03	94	.03	.1	NC	<.1
Minus 100 plus 150 ..	.89	96	.02	.1	NC	<.1
DH 3 COMPOSITE						
Minus 10 plus 14	1.3	74	0.062	0.7	49	0.1
Minus 14 plus 20	.71	68	.057	.8	58	.1
Minus 20 plus 28	1	83	.051	1.4	78	.1
Minus 28 plus 35	.59	82	.039	.8	71	.1
Minus 35 plus 48	1.1	90	.026	1	69	.1
Minus 48 plus 65	.89	93	.024	.8	74	.1
Minus 65 plus 100 ...	.97	91	.019	1.1	68	.1
Minus 100 plus 150 ..	1	92	.022	1.1	73	.1
DH 4 COMPOSITE						
Minus 10 plus 14	0.31	64	0.045	0.1	21	0.1
Minus 14 plus 20	.34	69	.038	.1	20	.1
Minus 20 plus 28	.64	78	.030	.1	15	.1
Minus 28 plus 35	.78	82	.030	.1	15	.1
Minus 35 plus 48	.79	87	.025	.1	18	.1
Minus 48 plus 65	1.3	85	.029	.1	11	.1
Minus 65 plus 100 ...	.85	93	.015	.1	19	.1
Minus 100 plus 150 ..	.71	88	.019	.3	36	.1

NC   Not calculated.
[1]Troy ounces per short ton.

The analysis showed that cassiterite liberation had begun at a size larger then 10 mesh because 50 to 74 pct Sn was recovered in less than 25 pct of the weight in the 10- to 14-mesh fraction. The four composite samples were quite similar in liberation size. A liberation step occurred at 20 to 28 mesh and another at 65 mesh. Tailings of 0.02 to 0.03 pct Sn would be predicted at 65 mesh.

Silver liberation was not uniform among the four samples; DH 1 and 2 showed liberation at 35 mesh, DH 3 could be concentrated starting at 10 mesh with a step at 20 mesh, and DH 4 did not begin to liberate until 100 mesh.

EXPLORATORY GRAVITY CONCENTRATION TESTS

With the tin mineralization fairly uniform among samples, as discussed above, one would expect the samples to respond similarly to gravity concentration. About 5,000-g splits from each drill-hole composite were roll crushed to minus 8 mesh, then ball-mill ground at 50 pct solids to minus 20 mesh (28 mesh for DH 1). The sized samples were passed over a Deister[4] shaking table, and

[4]Reference to specific products does not imply endorsement by the U.S. Bureau of Mines.

five products were collected, from concentrate (1) to tailing (5). The table process consists of flowing a slurry of the ore across a plane riffled deck. The deck is shaken in the direction of its long axis, and the shaking motion can be varied in both frequency and intensity. The motion is such that the return stroke velocity is faster than the forward stroke. It is the quickness of the return that causes the material to migrate forward toward the discharge end. Wash water flows evenly at right angles to the shaking motion. The deck can be inclined in the direction of the waterflow up to 8° from the horizontal plane. The higher density minerals are least affected by the current of the wash water, so they are collected and moved to the leading edge of the riffles. The lower density particles tend to be washed over the riffles to the lower edge of the deck. Table 4 gives results of the test series. All four samples demonstrated amenability to gravity concentration with similar recoveries and grades.

The tailings products (4 and 5) ideally would contain only liberated gangue, and the middling (product 3) would contain the slightly heavier particles with locked values. However, the tail tin grades were not as low as predicted by the liberation test, i.e., 0.02 pct. Screening the DH 1 test products into sizes showed that the extra tin loss to the tail was contained in the minus 270-mesh fraction. Figure 3 is a plot of cumulative tin distribution in the table splits for different size fractions. Note that the only fraction that continued to lose tin in the final two products was the minus 270-mesh fraction. A large portion of the tin lost to product 5 is therefore likely contained in the fine particles or slimes (minus 325 mesh), which tend to report to the tailings product. When treating such a large size distribution, the efficiency of the table drops for the fine fractions, because table parameters are set for larger particles separation.
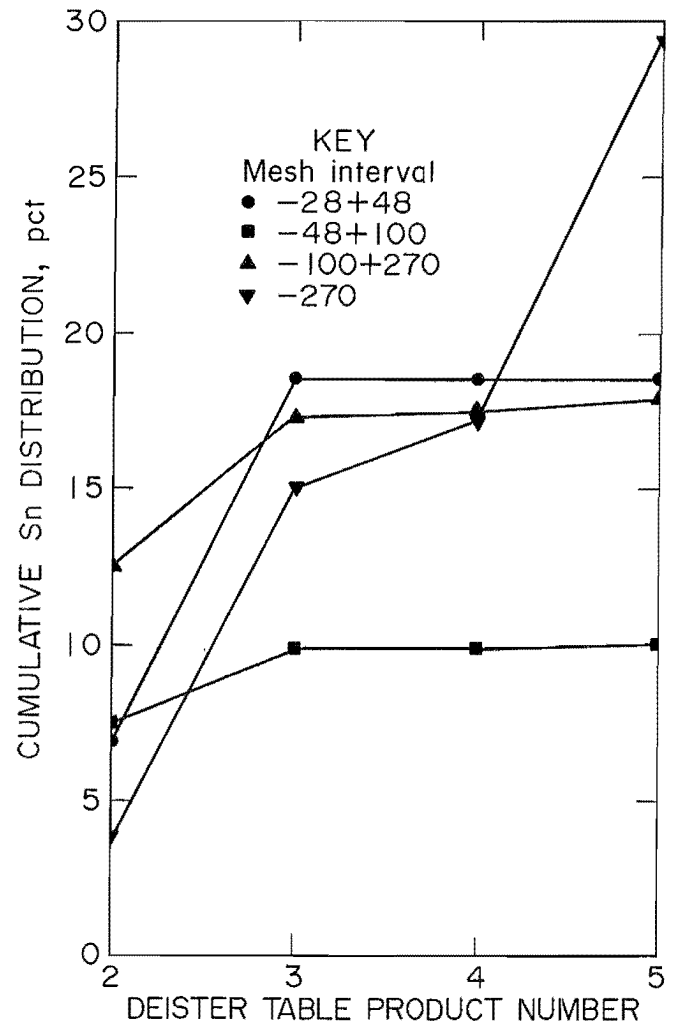
Figure 3.—Cumulative tin distribution of screened DH 1 table products 2 through 5.

Table 4.—Grade and tin distribution of products from single shaking table pass, percent

Product	DH 1			DH 2			DH 3			DH 4		
	Weight dist	Sn		Weight dist	Sn		Weight dist	Sn		Weight dist	Sn	
		Grade	Dist		Grade	Dist		Grade	Dist		Grade	Dist
1 (conc)	0.2	18.0	24.0	0.2	32.0	36.7	0.3	15.0	23.5	0.3	8.5	29.5
2	1.8	2.24	31	3.9	1.27	28.4	18.8	.52	51	14.8	.15	25.7
3	63.1	.07	30	50.4	.06	17.4	64.9	.05	16.9	46.2	.05	26.8
4	7.6	.05	2.4	14.6	.02	1.6	11.7	.09	5.5	20.2	.04	9.4
5 (tail)	27.3	.07	12.6	30.9	.09	15.9	4.3	.14	3.1	18.5	.04	8.6
Total or calc head	100	.14	100	100	.17	100	100	.19	100	100	.09	100

Calc   Calculated.
Conc   Concentrate.
Dist   Distribution.

TIN RECOVERY FLOWSHEET DEVELOPMENT

PRIMARY CONCENTRATION

An effective method of gravity beneficiation is to process the ore as large as possible and still produce a tail product that does not require any further processing. This reduces the weight to be processed further by more expensive and lower tonnage equipment. Two types of spirals, the Humphrey's and the Reichert LG-7, were tested as primary concentrators. The Humphrey's is a spiral-shaped channel of launder with a modified semicircle cross section. The spiral contains the standard five complete turns, with a 13.5-in drop per turn. The flowing pulp progresses from the top to the bottom of the spiral. As this feed slurry progresses down the spiral, particles with the highest specific gravity sink to the bottom and move to the inside of the channel. The lighter particles move to the outside of the channel and are carried away in a faster, more dilute part of the stream. Adjustable concentrate splitters, each consisting of a simple stainless steel belt disk resting over a circular port, are provided along the inside of the channel. Wash water is available along the entire inside edge of the spiral, where it flows in a separate channel. As the gradually impoverished pulp flows down the spiral, wash water is proportioned from the wash water channel by a series of notches and directed to wash repeatedly across the concentrate band to sweep out unwanted gangue particles. The end of the spiral is equipped with five fixed splitters to divide the exiting slurry to allow extensive analysis.

The Reichert LG-7 spiral is also a spiral-shaped channel of launder with a modified semicircle cross section. The LG-7 spiral, however, does not use wash water to clean the concentrate and keep the pulp moving. Instead, the channel is modified to cause the flow to alternately narrow and flatten with each turn of the spiral. This action keeps the solid from dropping out of the pulp as it would in the Humphrey's spiral without wash water. The exit end of the spiral is equipped with three adjustable splitters to give four products: concentrate, middling, sand (coarse) tail, and slime tail.

In addition to the two spirals, the Deister shaking table was tested as the primary concentrator. About 40 to 50 lb of the DH 2 and DH 3 samples were ball-mill ground in stages to minus 20 mesh, and the slimes (minus 325 mesh) fraction was removed by screening. Minimizing of slimes was not an objective; therefore, the weight loss to the slimes was high, 15 to 20 pct, with a tin grade about the same as that of the feed.

Humphrey's Spiral Versus Deister Table

The screened DH 3 sample was placed in the sump and slurried to approximately 20 pct solids. Three tests were run from this sump charge: first the Humphrey's, then the table, and last the Humphrey's with splitters only at the discharge end of the spiral and not along the spiral path. By this procedure, three primary concentration techniques were tested using virtually identical feed material. The order of the three tests was reversed for the DH 2 sample in an attempt to minimize biased results caused by upgrading in the sump.

To provide a basis for comparison between the apparatuses as well as different runs on the same apparatus, the cumulative distribution of each element in tailings that would be produced by combining consecutive splits was plotted versus the cumulative weight percent of feed in the tailing. The amount that would be collected in an 80-wt-pct tailing was read from the plot and used to represent separation efficiency. Figure 4 is the plot used for the DH 3 table test and is given as an example. It can be seen that if products 1, 2, and 3 were collected as the concentrate, they would total 17.5 wt pct, about 2.5 wt pct away from producing the target of 80-wt-pct tails.
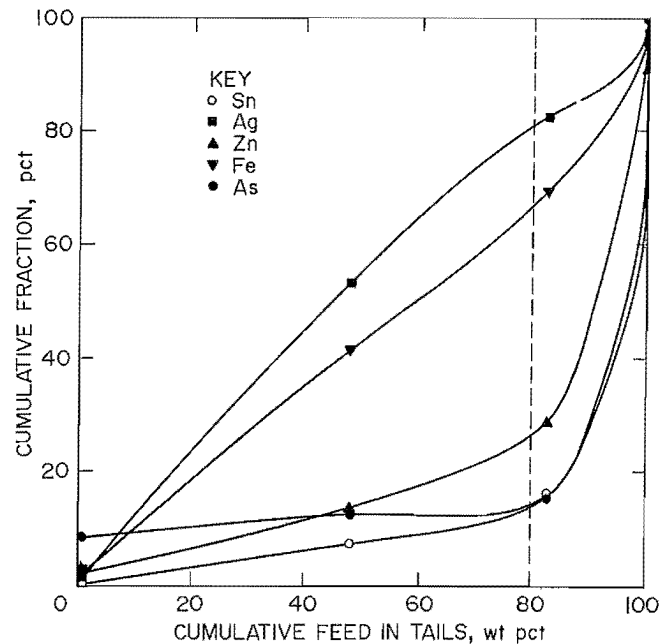

KEY
o Sn
■ Ag
▲ Zn
▼ Fe
● As

Figure 4.—Primary concentration test on DH 3 by Deister table.

The results of the Humphrey's spiral and Deister table tests are given in table 5. The same trends are seen for both DH 2 and 3. The Humphrey's with splitters lost the least amount of tin to the 80-wt-pct tails (13 to 14 pct). Without the use of splitters along the spiral path, tin loss was 18 pct. The table was slightly less efficient than the Humphrey's with splitters in producing a low-grade tail. Note the poor silver response of DH 3; 80 pct Ag would be lost during primary concentration. The sulfides followed the same trend as the tin, with the exception of arsenic, for which the table was able to reject more to the tails.

Table 5.—Elements lost to 80-wt-pct tailing in primary concentration, percent

Sample and apparatus	Sn	Ag	Zn	Fe	As
HUMPHREY'S SPIRAL VERSUS DEISTER TABLE					
DH 2					
Humphrey's spiral:					
With splitters	14	NC	43	77	68
Without splitters . . .	18	NC	52	82	74
Deister table	16	NC	45	77	82
DH 3					
Humphrey's spiral:					
With splitters	13	85	34	68	7
Without splitters . . .	18	82	48	68	10
Deister table	15	82	27	68	15
HUMPHREY'S SPIRAL VERSUS REICHERT LG-7 SPIRAL					
DH 2[1]					
Humphrey's spiral:					
Slimes removed . . .	13	NC	33	74	38
Slimes not removed	28	NC	48	74	68
Reichert LG-7 spiral:					
Slimes removed . . .	15	NC	58	74	33
Slimes not removed	16	NC	39	74	55

NC   Not calculated.
[1]2d composite from drill hole 20.

Humphrey's Spiral Versus Reichert LG-7 Spiral

The original composite of DH 2 had been used up when these tests were conducted; therefore, a second composite was made of adjacent footage from drill hole 20 core. The sample was roll crushed to 70 pct passing 20 mesh, and the minus 20-mesh fraction was used in the test series. The order in which the tests were performed was (1) LG-7 spiral with slimes, (2) Humphrey's spiral with slimes, (3) Humphrey's without slimes, and (4) LG-7 without slimes. These results are also given in table 5. The Humphrey's again lost the least amount of tin to the tails when treating the deslimed feed. However, since the LG-7 lost only 1 to 2 pct more, has a higher capacity, and costs less, it might be chosen over the Humphrey's. The LG-7 test that included slimes lost only 16 pct of the tin to

the tails and far outperformed the Humphrey's spiral with slimes, which lost 28 pct. The LG-7 spiral essentially performed as well as in the test with deslimed feed. The percent of minus 325 mesh was determined for both LG-7 tests. The test with slimes had 15 pct slimes, and the deslimed-feed test had 3 pct, likely produced while pumping. The presence of slimes in the deslimed-feed test may, in part, explain the similar results from the two LG-7 tests. Arsenic fraction in the tail varies greatly between the two samples. In the "Ore Sample Characterization" section, the difference in the degree of weathering between samples DH 2 and DH 3 was mentioned. The result of the high degree of weathering of DH 2 can be seen in table 5, where 33 to 82 pct of the arsenic was lost to the tails. The arsenic-oxide-coated silicate grains ended up in the tails because of their lower density. The finer redeposited grains ended up in the tails because of their size. On the other hand, the nonoxidized sulfides in sample DH 3 were concentrated very easily.

The most efficient primary concentrator, when treating the deslimed feed, was the Humphrey's spiral, but the LG-7 spiral may have the advantage because it has higher capacity and is less expensive. In addition, the LG-7 would be able to effectively treat unclassified feed.

SECONDARY CONCENTRATION

The emphasis in these studies was on grade rather than recovery. Tabling tests were conducted using samples of each drill-hole composite that were ground to minus 20 mesh (28 mesh for DH 1), deslimed, and screened into plus 65-mesh and minus 65 plus 325-mesh fractions. The separate fractions were then passed over the table; the results are given in table 6. Some concentration occurred in the plus 65-mesh tests. However, the concentrate (product 1) tin grades and recoveries were higher and the tails (products 3 through 5) grade was lower in each case for the minus 65-mesh test. Three of the four minus 65-plus 325-mesh tests produced tail products with tin grades less than predicted, i.e., 0.02 pct.

To capitalize on the ability of the table to concentrate at plus 65 mesh and reject a low-grade tail at minus 65 mesh, the flowsheet shown in figure 5 was tested. An eight-cycle locked-cycle test was conducted on the Humphrey's spiral concentrates produced in the primary concentration tests on deslimed DH 2 and 3 feed, and a five-cycle test was run on a Humphrey's spiral concentrate of a composite made of equal portions of deslimed DH 1, 2, 3, and 4 feed. The feed per cycle to the locked-cycle tests was 400 g for DH 2 and the composite and 390 g for DH 3. The concentrate, tails, and slimes from each cycle were collected and analyzed. The recirculating load was determined after the last cycle.

Table 6.—Effect of feed size on distribution and grade of table products, percent

Product	DH 1			DH 2			DH 3			DH 4		
	Weight dist	Sn		Weight dist	Sn		Weight dist	Sn		Weight dist	Sn	
		Grade	Dist		Grade	Dist		Grade	Dist		Grade	Dist
PLUS 65 MESH												
1	0.6	8.7	42.0	0.4	7.7	24.2	0.4	13.0	33.3	0.3	6.8	19.8
2	8.3	.58	38.4	22.2	.38	59.5	20.7	.36	50.6	38.2	.16	66.7
3	90.5	.027	19.5	77.4	.03	16.4	78.6	.03	16.0	45.3	.022	10.8
4	.6	.017	<.1	NP	NP	NP	.3	.06	.1	16.2	.015	2.7
Total or calc head	100	.125	100	100	.14	100	100	.15	100	100	.09	100
MINUS 65 PLUS 325 MESH												
1	0.5	14.0	57.6	0.5	18.0	64.4	0.9	18.0	68.6	0.3	19.0	50.4
2	4.9	.57	24.4	15.3	.24	28.9	6.5	.72	19.7	7.5	.5	37.4
3	71.8	.021	13.1	74.6	.01	5.9	58.3	.04	9.8	49.6	.016	7.9
4	11.5	.020	2	9.6	.01	.7	24.2	.01	1.1	42.7	.010	4.3
5	11.3	.029	2.9	NP	NP	NP	10.1	.02	.8	NP	NP	NP
Total or calc head	100	.114	100	100	.13	100	100	.24	100	100	.10	100

Calc — Calculated.
Dist — Distribution.
NP — No product collected.

Table 7 provides the last cycle assay for each test. The concentrate band collected from the first table included not only the brown cassiterite band but some of the sulfide band. The cassiterite band was separated on the last cycle of the DH 3 test and is reported as concentrate 1; the rest of the usual concentrate is concentrate 2. The tails that would be discarded did not include the sulfide band. The tails had a grade of 0.02 pct Sn or less, as predicted above. The slimes were found to have a higher sulfide content than the feed. The tin recovery for this flowsheet varied depending on the sample: 96 pct for DH 3, 83 pct for both DH 2 and the composite. Arsenic, which must be cleaned from the concentrate, was also concentrated, with 97 pct recovery for DH 3, 49 pct for the composite, and 19 pct for DH 2. Once again, test results imply a large variation in degree of oxidation and its effect on the sample's arsenic response to gravity beneficiation, because the arsenic feed grade for DH 2 and DH 3 is about the same (see table 2). The composite test might give the average response of the ore for arsenic concentration.

The flow scheme was effective for the recovery of tin from a spiral concentrate. Excess tin did not build up in the recirculating stream, and the tails were at or below the 0.02-pct target.

CLEANING OF GRAVITY CONCENTRATE

In general, the gravity concentrates produced contained almost as much sulfide as cassiterite; DH 3 concentrates were particularly high in impurities. Several methods were investigated to produce a high-grade tin product with low arsenic and sulfide content, including cassiterite flotation, gangue flotation, sulfide flotation, leaching, magnetic separation, and further tabling.

A concentrate was made from a composite consisting of 52.3 pct DH 1, 16.2 pct DH 2, and 31.5 pct DH 3, which was tabled at 20 mesh; the tails from the first pass were reground to minus 60 mesh, and the tails were tabled again. The concentrates were combined into one concentrate, which was used in most of the cleaning tests, and included some nonsulfide gangue to determine its response to the tests.
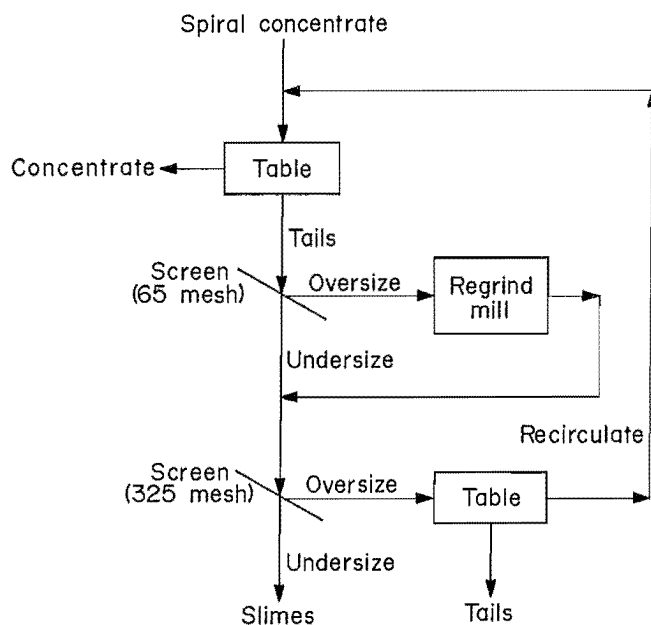
Figure 5.—Flowsheet of locked-cycle secondary concentration tests.

Table 7.—Locked-cycle test of secondary concentration flow scheme for DH 2, DH 3, and composite, percent

	Product		Sn		Ag		Zn		Fe		As	
	Weight, g	Dist	Grade	Dist	Grade[1]	Dist	Grade	Dist	Grade	Dist	Grade	Dist
DH 2												
Conc	2.7	0.7	24.0	83.4	0.7	8.7	2.5	18.4	3.6	2.0	0.75	19.1
Tails	351	93.2	.02	9	.05	80.8	.075	71.7	1.1	78.9	.02	66.1
Slimes	22.8	6.1	.26	7.6	.1	10.5	.16	9.9	4.1	19.1	.069	14.8
Total or calc head .	376.5	100.0	.21	100.0	.06	100.0	.1	100.0	1.3	100.0	.028	100.0
RL	130.6	34.7	.81	NAp	.1	NAp	.58	NAp	1.4	NAp	.052	NAp
DH 3												
Conc 1	2.5	0.8	34.0	41.3	7.6	11.7	1.0	0.02	11.0	2.6	8.65	28.3
Conc 2	16.7	5.1	6.7	54.5	5.8	59.6	4	52.9	21	32.6	3.16	69.2
Tails	291.7	89.6	.015	2.1	.1	18	.14	32.4	1.9	51.5	.001	.4
Slimes	14.6	4.5	.3	2.1	1.2	10.7	1.1	12.7	9.8	13.3	.11	2.1
Total or calc head .	325.5	100.0	.63	100.0	.5	100.0	.39	100.0	3.3	100.0	.23	100.0
RL	137.7	42.3	.35	NAp	2.2	NAp	3	NAp	51	NAp	.14	NAp
COMPOSITE OF DH 1, 2, 3, AND 4												
Conc	6.1	1.6	11.0	83.4	4.1	50.0	4.0	42.9	14.0	14.7	1.7	48.7
Tails	371.5	95.2	.017	7.8	.05	37.2	.062	40.5	1.2	76.5	.025	43.7
Slimes	12.8	3.2	.55	8.8	.5	12.8	.74	16.6	4	8.8	.126	7.6
Total or calc head .	390.4	100.0	.21	100.0	.13	100.0	.15	100.0	1.5	100.0	.05	100.0
RL	163.2	41.8	.15	NAp	.4	NAp	1.4	NAp	2.8	NAp	.042	NAp

Calc   Calculated.
Conc   Concentrate.
Dist   Distribution.
NAp   Not applicable.
RL   Recirculating load.
[1]Troy ounces per short ton.

Figure 6.—Microflotation cell used in concentrate cleaning tests.

Figure 7.—Effect of particle size on cassiterite flotation.

Flotation tests were performed in an in-house-constructed microflotation cell, shown in figure 6, because of the small volume of concentrate available and the large number of desired tests. Five grams of gravity concentrate were added to 100 mL of pH-adjusted water. The depressant, if used, was added next, and the pulp was conditioned for 5 min. The collector was then added and conditioned for 2 min; the air was then turned on and the froth collected for 1 to 2 min.

Cassiterite Flotation
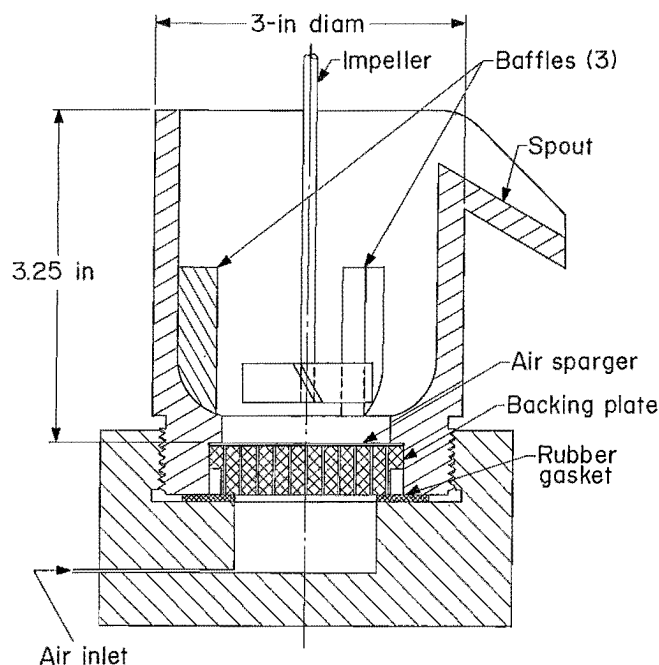
The collector used was American Cyanamid Co.'s Aero 845, which was selected from a reagent screening test performed on the slimes that is discussed in the section "Slimes Beneficiation." First, using a collector addition of 10 lb/st at pH 2, the effect of particle size was determined by floating a series of sized samples of the composite concentrate described above and a concentrate from DH 2. Results are shown in figure 7. Tin recovery showed rapid improvement with size reduction to about 100 mesh; smaller sizes showed little improvement. The weight fraction in the concentrate showed the same increases, however, giving little upgrading. The concentration factor, CF (concentrate grade divided by feed grade), was only between 1.15 and 1.5; very little benefit was obtained.

The next test series objective was to improve the selectivity by the use of depressants. Tannic acid, dextrin (yellow), and quebracho were tested as depressants for sulfides, particularly pyrite, zinc sulfate as a depressant for marmatite, and sodium fluosilicate (Na_2SiF_6) for quartz and silicates (2), with the following results:

- Tannic acid decreased the recovery of zinc, iron, and arsenic by an average of 11 pct at 1 lb/st; however, no further reduction of these elements was observed at 3 lb/st, and the tin recovery dropped 15 pct.
- Dextrin (yellow) had no measurable effect at additions of 1 or 3 lb/st.
- Quebracho was most effective as a general depressant of all elements, including tin. The average decrease in recovery for zinc, iron, and arsenic was 18 pct at 1 lb/st and an additional 10 pct at 3 lb/st. However, both tin and silver recoveries dropped 20 pct over the same range.
- Zinc sulfate was effective in reducing zinc recovery 15 pct at 1 lb/st and 35 pct at 4.6 lb/st. It promoted iron and silver at the higher addition, which was unexpected considering that the characterization study identified the silver as associated with the stannite locked in marmatite. The arsenic was promoted as much as 40 pct over the range.
- The sodium fluosilicate addition effect was only measured indirectly, since SiO_2 was not assayed. With sodium fluosilicate additions of 3 and 9 lb/st, the weight fraction in the concentrate increased slightly, and the tin recovery increased 18 pct over the range.

In summary, no depressant studied was effective enough in increasing selectivity for tin to produce a final concentrate.

As a second method to improve the cassiterite flotation selectivity, oxidation pretreatments using hydrogen

peroxide (H_2O_2) addition and pressure oxidation were applied in an attempt to alter the sulfide surface to inhibit collector attachment. A 30-min conditioning step with 5.5 pct H_2O_2 followed by three washing and filtration steps was added to the basic flotation procedure. The washing steps were required because the collector was destroyed by the excess H_2O_2, and simple aeration was not effective in removing the excess. The pretreatment appeared to have no effect on the flotation of nonsulfide gangue. Zinc recovery dropped 31 pct, iron 8 pct, and silver 27 pct, but tin and arsenic were not affected.

A more severe oxidation treatment was performed in an autoclave with 20 pct solids and 50 psi oxygen overpressure at 220° C for 3 h. The oxidized slurry changed from light brown to dark reddish brown, showing that extensive oxidation had occurred. The minus 65-mesh portion of the residue was then floated with and without 10 lb/st sodium fluosilicate. Recovery was higher for oxidized sulfides than for nonoxidized sulfides, with an increase of 13 to 39 pct for all elements. When the depressant was used, the concentrate tin grade dropped only slightly from that of the test without pressure oxidation, but the impurity content was higher. There was no significant advantage with either oxidation pretreatment.

Nonsulfide Gangue Flotation

Though most of the nonsulfide gangue was easily removed by the table, gangue flotation was tested as a means to upgrade gravity concentrate. The basic flotation procedure described above was modified to include a 20-min conditioning step at pH 2 prior to collector addition. A collector screening test series was conducted using Armac C (coco amine), PA-14 acetate from Exxon Chemical Co., alkyl amine acetate, and Arosurf 70 from Sherex. As can be seen in table 8, all of the collectors tested floated too much of the tin. The concentrates and tails had the same appearance and contained the same fraction of nonsulfide gangue.

Table 8.—Collector screening test for nonsulfide gangue flotation

Collector	Addition, lb/st	Concentrate	
		Weight, pct	Sn dist, pct
Alkyl amine acetate ..	5.5	74.5	63.4
Armac C	2.1	38.3	13.4
Do.	3.3	68.7	67.2
Arosurf 70	7.1	59.6	42.6
PA-14	2.6	49.0	13.4
Do.	5.3	71.4	47.1

Dist   Distribution.

Sulfide Mineral Flotation

The sulfide flotation test series used the same basic procedure except that a 5-min conditioning time after collector addition was used, a frother was required, which was added after the collector conditioning step, and the float time was increased to 3 min. The collector used was a combination of 0.3 lb/st each Aerofloat 208 and Aero 350 from Cyanamid, and the frother was 5 lb/st of Dowfroth 400 from Dow Chemical.

The effect of particle size was determined by floating sized feed: 35 to 48 mesh, 48 to 65 mesh, and minus 65 mesh. The results (table 9) show that the gravity concentrate must be ground to minus 65 mesh to effectively remove the sulfides. The 35- to 48-mesh test was floated twice; the tails from the first float were scavenged using 0.4 lb/st $CuSO_4$ and 0.5 lb/st Aero 350, and the two concentrates were combined.

Oxidation of the gravity concentrate greatly reduced efficiency of sulfide flotation. After the concentrate had been stored in a closed container for several weeks, flotation removed only 65 pct instead of 95 pct Zn and 28 pct instead of 55 pct As (table 10). In addition, tin loss jumped from 1 to 26 pct. To clean off the oxidization layer, an acid scrub step was tested. The oxidized gravity concentrate was agitated in a pH 1.8 slurry for 5 and 10 min, then after the pH was raised to 7 with lime, the concentrate was floated using the procedure discussed above. The 5-min scrub test not only reversed the oxidation effect and returned the tin loss to about 1 pct and the zinc and arsenic recoveries to the mid-90's and mid-50's, respectively, but it also increased the iron recovery to 68 pct. The 10-min scrub time raised the iron and arsenic recoveries even more, but more tin was floated (7.6 pct). The tin loss to the sulfide concentrate was analyzed to determine if stannite (SnS, found in the characterization study) might be the tin mineral being lost. Examination with the SEM and XRF showed only cassiterite present in the sulfide concentrate; therefore, flotation of stannite in the sulfide flotation cleaning step was not a problem for the sample tested.

The sulfide flotation tailing, or the tin product, from the acid scrub test still contained up to 8 pct As and 10 pct Fe. The product, therefore, needed to be processed further. The sulfides were scavenged from the float tail using 0.5 lb/st $CuSO_4$ and 1.5 lb/st Aero 350. Table 11 shows the improvement in the tin product by both sulfide flotation steps. The tin products for the two scrub-time tests were very similar, with the 5-min test producing the same iron and arsenic removal as the 10-min test. The silver reported to the sulfide concentrate, which would be refractory to silver recovery by conventional cyanidation processing because of its high sulfide and arsenic content.

Table 9.—Effect of particle size on sulfide flotation of gravity concentrate, flotation concentrate, percent

Size fraction, Tyler mesh	Sn		Ag		Zn		Fe		As	
	Grade	Dist	Grade[1]	Dist	Grade	Dist	Grade	Dist	Grade	Dist
Minus 35 plus 48 ...	0.05	0.1	0.8	12.1	1.47	43.4	2.0	3.1	0.072	0.4
Minus 48 plus 65	13	.4	2.2	31.4	4.8	62.5	6.2	12.3	.067	.9
Minus 65	18	1	4.2	66.7	8.04	95.4	12	38	.95	55.2

Dist   Distribution.
[1]Troy ounces per short ton.

Table 10.—Effect of surface oxidation and acid scrubbing on sulfide flotation, concentrate analysis, percent

Flotation pretreatment	Weight dist	Sn		Ag		Zn		Fe		As	
		Grade	Dist	Grade[1]	Dist	Grade	Dist	Grade	Dist	Grade	Dist
Fresh	22.2	0.18	1.0	4.2	66.7	8.0	95.4	12.0	38.0	0.95	55.2
Oxidized[2]	34	14.2	25.8	.02	38.2	7.6	65	17.3	33.3	2.23	27.7
Acid scrub:[2]											
5 min	37.2	.85	1.7	12.8	79.1	9	96.3	34.6	68	16.45	54
10 min	45.2	2.87	7.6	10	84.6	7.2	94.3	30.4	75.9	16.19	79.5

Dist   Distribution.
[1]Troy ounces per short ton.
[2]Feed upgraded on table to remove nonsulfide gangue prior to flotation.

Table 11.—Gravity concentrate cleaning by two-stage sulfide flotation, tin product analysis, percent

Product	Weight dist	Sn		Ag		Zn		Fe		As	
		Grade	Dist	Grade[1]	Dist	Grade	Dist	Grade	Dist	Grade	Dist
				5-min ACID SCRUB							
Gravity conc (calc)	100.0	18.3	100.0	6.0	100.0	3.5	100.0	19.0	100.0	11.3	100.0
Rougher tails	62.7	28.6	98.3	2.0	20.9	.2	3.6	9.7	32.0	8.3	46
Scavenger tails	47	33.8	86.9	.8	6.6	.06	.8	4.4	10.8	2.2	9.3
				10-min ACID SCRUB							
Gravity conc (calc)	100.0	17.0	100.0	5.3	100.0	3.5	100.0	18.1	100.0	9.2	100.0
Rougher tails	54.7	28.7	92.4	1.5	15.4	.4	5.7	8	24.1	3.5	20.5
Scavenger tails	49	30.8	88.9	1.3	12.2	.1	1.6	7.2	19.4	2.6	14

Calc   Calculated.
Conc   Concentrate.
Dist   Distribution.
[1]Troy ounces per short ton.

The scrub also increased the amount of silver that floated, from 66 to over 80 pct in the rougher float. Overall, only 7 to 12 pct of the silver in the gravity concentrate remained with the final tin product after the scavenger float. Although the silver grade in the sulfide float was as high as 13 tr oz/st, silver recovery would be difficult because of the refractory nature of this product.

Although the amount of oxidation that would occur in a beneficiation plant under normal conditions or under possible extreme conditions is not known, the preferred sulfide flotation procedure to clean the gravity concentrate would include a 5-min acid scrub pretreatment at pH 2. Not only did the scrub reverse any effects of oxidation, but it almost doubled the iron removal. Therefore, the procedure would regrind the gravity concentrate to minus 65 mesh, acid scrub for 5 min, rougher float with 0.3 lb/st of Aerofloat 208 and Aero 350 and 5 lb/st of Dowfroth 400, and scavenge the rougher tails using 0.5 lb/st CuSO_4 and 1.5 lb/st Aero 350.

Sulfide Mineral Leaching

Selectively leaching the sulfides using nitric acid was investigated. Cassiterite is slowly attacked by acids, while nitric acid decomposes pyrite and arsenopyrite (FeAsS) and is the only acid listed in standard references as one in which loellingite (FeAs_2) is slightly soluble (3-4). Once in solution, arsenic can be precipitated as ferric arsenate, and if the ferric iron-arsenic ratio is greater than 12:1, less than 5 ppm As will remain in solution (5).

The gravity concentrate was first added to a 20-pct nitric acid solution, but no visible decomposition occurred. More acid was added to raise the concentration to 47 pct, and immediately gas was evolved. After 45 min, gas evolution was no longer observed, and the leach was terminated. The residue was filtered, and the pH of the liquor was raised to 5.5 with lime. Air was bubbled through the resulting slurry for 18 h to oxidize the ferrous iron to ferric.

Extraction results are given in table 12. About 84 pct As and 78 pct Fe, but only 29 pct Zn were leached. The

silver appeared to remain in the liquor, because only 39 pct of it was accounted for in the solids. The tin loss was minor: less than 5 pct. Initially, the leach liquor contained 180 ppm Fe and 54 ppm As. The arsenic content was reduced to less than 2 ppm by the pH adjustment step. Apparently the ferric iron-arsenic ratio requirement was met without aeration.

Table 12.—Nitric acid leaching of gravity concentrate, percent

	Weight, g	Sn	Ag[1]	Zn	Fe	As
Analysis:						
Feed	5.0	19.0	1.4	1.3	4.2	1.14
Residue ..	4.3	21	.3	1.07	1.1	.21
Precipitate	3.43	.11	.1	.243	4.7	1.4
Extraction[2] ..	.7	4.9	82	29	78	84

[1]Analysis in troy ounces per short ton.
[2]Percent of feed leached.

The final state of the arsenic could not be determined. Examination of the precipitate by SEM and XRF revealed that the particle size was much less than 0.1 μm, and individual grains could not be analyzed. The sample appeared to be one phase comprising calcium, iron, silicon, arsenic, aluminum, and magnesium, in order of abundance. More control over the precipitation step would be required to grow larger crystals and determine the actual arsenic occurrence. Leaching with nitric acid selectively removed the sulfides, but the economics of the process may not be advantageous.

Magnetic Separation

A sample of dry gravity concentrate was processed through a Dings magnetic separator at the maximum current setting of 3.7 A. The separator produces three products: a permanent magnet scalper product, a variable electromagnet product, and a nonmagnetic fraction. The results given in table 13 show that little cleaning occurred; only iron was separated to any extent.

Table 13.—Dry magnetic separation of gravity concentrate, percent

Product	Weight dist	Sn Grade	Sn Dist	Ag Grade[1]	Ag Dist	Zn Grade	Zn Dist	Fe Grade	Fe Dist	As Grade	As Dist
Scalper	1.4	1.06	0.3	1.8	1.1	1.7	0.7	49.5	7.6	0.18	0.3
Magnetic	2.9	1.17	.8	3.3	4.3	4.8	4	24.4	8.1	.23	.8
Nonmagnetic	95.7	4.4	98.9	2.2	94.6	3.5	95.3	7.8	84.3	.91	98.9
Total or calc head	100.0	4.3	100.0	2.2	100.0	3.5	100.0	8.9	100.0	.88	100.0

Calc   Calculated.
Dist   Distribution.
[1]Troy ounces per short ton.

Wet magnetic separation tests were conducted on a Carpco high-intensity separator. A screened-bottom rectangular cup was filled with 1/4-in steel shot and placed between the two poles. The slurry was poured over the shot, then the shot was washed with water before the power was turned off. The cup and shot were removed and cleaned to collect the magnetic fraction. The results are plotted in figure 8. A current of 3.5 A removed 99 pct of the zinc and 75 pct of the iron. The silver response was within 5 pct of the iron response. Arsenic showed no upgrading in the magnetic fraction; the CF ranged from 0.48 at 1 A to 0.84 at 5.25 A. The CF of the tin also remained below 1.0, ranging from 0.74 at 1 A to 0.98 at 5.25 A.

Magnetic separation did not effectively clean arsenic from the gravity concentrate, and no benefit was seen in use of this method over other methods discussed earlier, wet or dry.

Tabling

The gravity concentrates from cycles 4 and 5 of the DH 3 locked-cycle test were combined and passed over the table. The brown cassiterite band was collected. Products were very similar to those from the DH 3 locked-cycle test concentrates 1 and 2 shown in table 7. The table efficiency was low because of the small difference in specific gravity between the sulfides and the cassiterite; 60 pct of the tin concentrated, and 45 pct of the arsenic was still collected in the concentrate. Though a tin grade of about 30 pct was produced, the impurity content was still too high.

Combined Tabling-Sulfide Flotation Method

The gravity concentrate produced by the locked-cycle test described earlier ("Secondary Concentration" section) had high sulfide contamination, with arsenic being of particular concern, and the tin grade was low. A combination of the concentrate cleaning methods previously discussed in this section was tested on the gravity concentrate produced during cycles 1, 2, 3, and 6 of the DH 3 locked-cycle test. This combined procedure started with upgrading the gravity concentrate on the table. The table concentrate was sized to minus 65 mesh and conditioned at pH 2 for 5 min. Sulfide flotation, described earlier, was performed, the tail being scavenged with $CuSO_4$ plus more collector. The results of this combined method are given in table 14. The table upgraded the tin from 10 to 28 pct and rejected 55 pct of the arsenic. The rougher float then
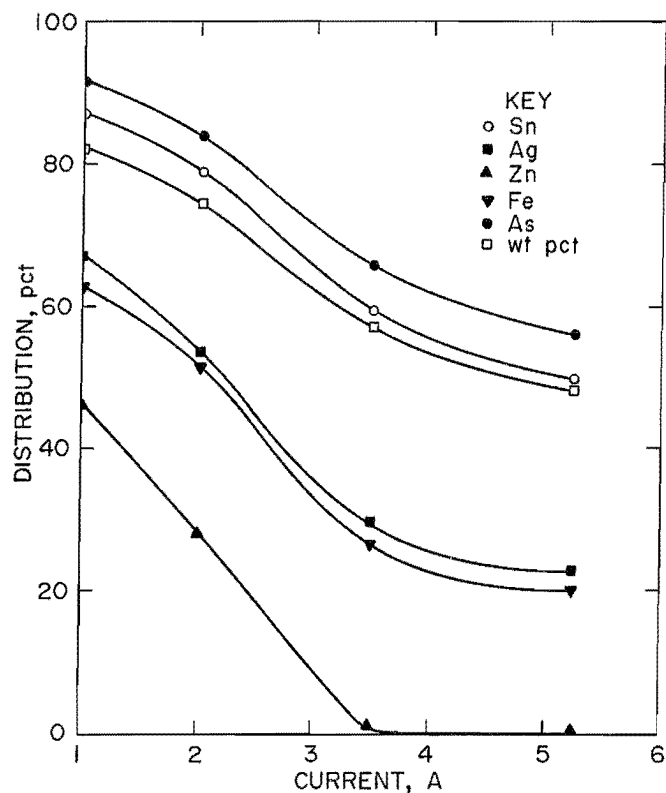
Figure 8.—Nonmagnetic fraction assay from wet magnetic separation of gravity concentrate.

raised the tin grade to 39 pct and also floated 37 pct of the arsenic, 59 pct of the iron, and 97 pct of the zinc. The scavenger float gave a final tin product (flotation tailing) of 50 pct Sn, 1.4 tr oz/st Ag, 0.02 pct Zn, 2.6 pct Fe, and 3.1 pct As, thereby achieving the goal of a 50-pct-Sn concentrate. Only 7 pct of the arsenic from the original gravity concentrate was contained in the final tin product.

SLIMES BENEFICIATION

Slimes (minus 325 mesh) are produced when any grinding is done; therefore, concentration of the slimes was studied. The weight percent of the slimes in the ground ore will vary depending on the grinding circuit. Minimizing of slime generation was not studied in this investigation. The slimes resulting from grinding the Coal Creek ore made up about 9 to 20 wt pct and assayed approximately feed grade. The slime concentration tests included cassiterite flotation, vanning on the Bartles-Mozley separator, sulfide flotation, and fuming.

Table 14.—Tabling-sulfide flotation method for cleaning gravity concentrate, percent

Product	Weight dist	Sn Grade	Sn Dist	Ag Grade[1]	Ag Dist	Zn Grade	Zn Dist	Fe Grade	Fe Dist	As Grade	As Dist
Table:											
Concentrate	20.9	27.7	60.4	7.8	25.4	1.15	5.9	18.6	16.5	8.13	45.2
Tails	79.1	4.8	39.6	6.1	74.6	4.8	94.1	25	83.5	2.60	54.8
Rougher float:											
Concentrate	6.9	5.2	3.7	19.1	20.2	3.3	5.7	33.7	9.8	9.22	16.8
Tails	14	38.7	56.7	2.4	5.2	.07	.2	11.3	6.7	7.60	28.4
Scavenger float:											
Concentrate	5.5	21.4	12.4	3.9	3.3	.15	.2	4.7	5.8	15	21.4
Tails	8.5	49.8	44.3	1.4	1.9	.02	<.1	2.6	.9	3.08	7
Total or calc head[2]	100.0	9.6	100.0	6.5	100.0	4.0	100.0	23.7	100.0	3.76	100.0

Dist   Distribution.
[1]Troy ounces per short ton.
[2]Sum or average of table tails, rougher concentrate, scavenger concentrate, and scavenger tails.

Cassiterite Flotation

The slime fraction that was removed prior to the DH 3 Humphrey's spiral test, which assayed 0.17 pct Sn, 0.7 tr oz/st Ag, 0.35 pct Zn, 2.3 pct Fe, and 0.04 pct As, was used in a collector screening test. The collectors tested were Aero 845 from Cyanamid; cupferron from Eastman Kodak Co.; benzyl phosphonic acid (BPA); oleic acid; and Briphos L2D, Briphos TD2D, Briquest 281, and Briquest 2n81, all from Albright & Wilson Ltd. The procedure for each test consisted of conditioning the slimes in a 500-g Galigher float cell at 33.3 pct solids, pH adjustment with sulfuric acid and/or lime with 4.2 lb/st Na_2SiF_6 at 1,750 rpm for 10 min, adding water to reduce the pulp density to 20 pct solids, adjusting the pH again to its desired value, adding the collector, conditioning for 10 min at 1,000 rpm, and finally adding the methylisobutyl carbinol (MIBC) frother if needed and floating for 4 min.

Table 15 gives the tin and silver assays for the screening test. Aero 845 gave the highest tin recovery of 73 pct but not the highest grade. Briquest 2n81 gave the second highest tin recovery, 5 pct less than that using Aero 845, and produced the highest tin grade of 2.0 pct. The other collectors were less productive in floating the tin, but one

showed ability to float the silver, i.e., cupferron, which floated 62 pct of the silver with only 9 pct of the tin in just 4 pct of the weight. There was no correlation between tin recovery and silver recovery.

Aero 845 was selected for the remaining flotation tests, and several concentration tests were conducted on slimes from DH 2. The DH 2 slimes assayed 0.13 pct Sn, 0.2 tr oz/st Ag, 0.1 pct Zn, 2.8 pct Fe, and 0.1 pct As. As shown in table 16, increasing the collector addition above 7 lb/st had little benefit. Silver recovery was not consistent, and no collector concentration effect could be determined.

The use of cleaning stages was tested on the same DH 2 slimes. Three cleaning steps were performed on a rougher concentrate. Owing to the reduced volume of the concentrate with each step, cell size was also reduced. The rougher step was performed in a 2,000-g Galigher cell, the first cleaner step in a 500-g Galigher cell, and the last two steps in the microflotation cell described earlier. Collector addition was 10 lb/st for the rougher, none for the first cleaner, and 7 lb/st for each of the last two cleaner steps. Table 17 shows that tin grade did rise with each step but did not get above 2.5 pct. The CF was 10 after the first cleaner but only 13 after the third cleaner.

Table 15.—Cassiterite flotation collector screen test on DH 3 slimes, percent

Collector	Amount, lb/st	pH	Concentrate Weight dist	Sn Grade	Sn Dist	Ag Grade[1]	Ag Dist	Tails Weight dist	Sn Grade	Sn Dist	Ag Grade[1]	Ag Dist
Aero 845	10.0	2.0	18.0	0.74	73.0	2.1	52.2	82.0	0.06	27.0	0.6	47.8
BPA	5	5	6.3	1.3	44.3	6.3	58.6	93.7	.11	55.7	.3	41.4
Briphos L2D	8	3.5	22.6	.53	62	2.2	61.7	77.4	.095	38	.4	38.3
Briphos TD2D	8	3.5	25.1	.5	65.6	1.9	68	74.9	.088	34.4	.3	32
Briquest 281	8	5	7.3	1.5	58.2	2.8	24	92.7	.085	41.5	.7	76
Briquest 2n81	8	5	6.1	2	68.4	5.1	45.3	93.9	.06	31.6	.4	54.7
Cupferron	5	2	4.3	.4	9.1	11.1	62.4	95.7	.18	90.9	.3	37.6
Oleic acid	6.4	4.8	21.8	.33	36.4	1.6	42.6	78.2	.16	63.6	.6	57.4

Dist   Distribution.
[1]Troy ounces per short ton.

Table 16.—Flotation of DH 2 slimes with Aero 845 collector, percent

Aero 845 addition, lb/st	Concentrate					Tails				
	Weight dist	Sn		Ag		Weight dist	Sn		Ag	
		Grade	Dist	Grade[1]	Dist		Grade	Dist	Grade[1]	Dist
4.9	14.0	1.1	74.9	1.0	62.0	86.0	0.06	25.1	0.1	38.0
7.2	21.8	.83	85.3	.4	27.1	78.2	.04	14.7	.3	72.9
9.9	23.9	.30	80.1	.5	61.2	76.1	.05	19.9	.1	38.8
12.1 ...	23.9	.67	84	.4	29.5	76.1	.04	16	.3	70.5

Dist   Distribution.
[1]Troy ounce per short ton.

Table 17.—Three-stage flotation cleaning of cassiterite flotation concentrate from DH 2 slimes, percent

Unit operation and products[1]	Weight distribution		Sn			Ag		
	Unit	Overall	Grade	Distribution		Grade[2]	Distribution	
				Unit	Overall		Unit	Overall
Rougher float:								
Conc	16.8	16.8	0.79	74.1	74.1	0.40	44.7	44.7
Tails	83.1	83.1	.06	25.9	25.9	.10	55.3	55.3
Cleaner 1:								
Conc	29.2	4.9	1.85	68.7	50.9	.39	28.9	12.9
Tails	70.8	11.9	.35	31.3	23.2	.40	71.1	31.8
Cleaner 2:								
Conc	73.5	3.6	2.23	88	44.8	.28	51.5	6.6
Tails	26.5	1.3	.82	12	6.1	.70	48.5	6.3
Cleaner 3:								
Conc	83.3	3	2.42	92.4	41.4	.16	49.7	3.3
Tails	16.7	.6	1.13	7.6	3.4	.94	50.3	3.3

[1]Feed for each cleaning operation was concentrate from previous stage.
[2]Troy ounce per short ton.

Vanning

Vanning tests for primary concentration of the slime fractions collected from the spiral tests on DH 2 and 3 were conducted using a Bartles-Mozley (B-M) separator. The B-M is run semicontinuously by feeding a charge of slurry at one end of a circularly vibrating deck that is sloped down between 0° and 2° from the horizontal. The deck motion allows lighter particles to rise and heavier particles to settle. Wash water, which is added at the feed end just after the deck is charged and the feed is turned off, rinses the light suspended particles over the discharge end. The heavier particles are nearer the deck, out of the faster part of the wash water flow, and travel the deck length more slowly. After the lighter particles have spilled over the discharge end, the deck is tilted to approximately 60°, and the concentrate is washed into a separate container. The deck is then returned to its original position, and the cycle is repeated.

The method used to test the ore slimes response to the B-M included collecting the tails in successive timed increments. Each fraction was weighed, dried, and assayed. The optimum wash time for maximum grade and/or recovery in a single test was determined by plotting the cumulative fraction removed versus wash time.

Two tests were performed on DH 3 slimes, using feed slurries of 50 and 20 pct solids. Figure 9 shows that

regardless of the solids content of the feed, the weight fraction removal rate was the same. The two tests showed
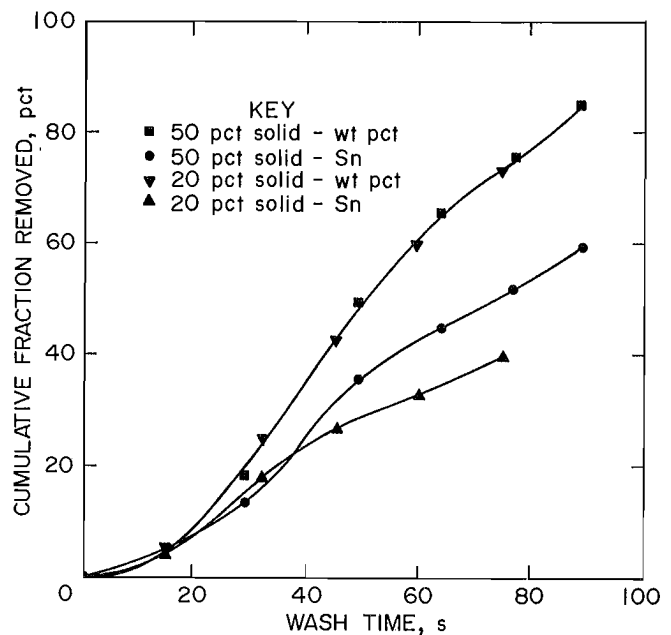

Figure 9.—Effect of pulp density in the Bartles-Mozley separator feed.

the same initial response to the washing; tin was lost to the finer particles that were washed off before they had time to settle. However, the final response and overall concentration was more effective in the 20-pct-solids test.

The 20-pct-solids test was repeated on the DH 2 slimes. Results fell between those from the two DH 3 tests. The concentrates that would be collected with a 75-s wash were calculated and are shown in table 18. A comparison of these values to the tin flotation results (table 17) shows that flotation produced higher grades and recoveries: 0.79 pct Sn at 74-pct recovery for flotation and 0.21 pct Sn at 52-pct recovery for the B-M.

Sulfide Flotation

Sulfide flotation was tested using Aerofloat 208 and Aero 350 collectors and Dowfroth 400 frother. Tests were performed at pH 6 without the acid scrub pretreatment discussed earlier ("Cleaning of Gravity Concentrate" section). Slime samples from DH 2 and 3 responded very similarly, with 6 to 10 pct of the weight floating, a 7- to 8-pct Sn loss to the concentrate, and sulfide recoveries, in percent, of 47 to 50 Zn, 14 Fe, and 19 to 32 As. Although a portion of the sulfides floated, no real advantage was gained by sulfide flotation.

Secondary Concentration by Fuming

Physical methods were unsuccessful in concentrating tin to a satisfactory grade from the slime fraction, as discussed above. Fuming was tested as a secondary concentrating step on the cassiterite flotation concentrate. In fuming, the stannic oxide (SnO_2) reacts with the sulfur present to become volatile stannous sulfide (SnS), which is mechanically removed from the furnace in the offgas. The SnS reacts readily with any oxygen to form SnO_2 dust, which is then scrubbed from the offgas. A sulfur source such as pyrite at a sulfur-to-tin ratio of 3:1 to 5:1 is required for optimum recovery (6). The cassiterite flotation concentrate contained, in percent, 1.7 Sn, 4.4 Fe, 0.33 Zn,

and 0.29 As. Assuming 100 pct of the sulfur in the marmatite and arsenopyrite will be reacted, only 23 pct of the iron need be present as pyrite. This requirement was expected to be met, in spite of the fact that iron was found in many other minerals. A fume test was conducted without sulfide addition to determine if the sulfur requirement was already met. A horizontal one-zone tube furnace was used to heat the concentrate to 1,000° C for 3 h. The one inch diameter reaction chamber was purged with nitrogen for 30 min prior to heating. The offgas was cleaned by passing it through an increased volume knockout chamber, allowing the fume to settle because of the decreased gas velocity, and then bubbling it through an air-sparged water chamber.

The fume residue in the boat assayed 0.49 pct Sn, which accounted for only 23 pct of the tin; i.e., 77 pct extraction was achieved without sulfide addition. The grade of the fume concentrate was very low, 2.3 pct Sn. The fume also contained 88 pct of the zinc and 35 pct of the iron. However, this would account for only a small portion of the weight of the fume. The fume is assumed to have been diluted from mechanical carryover in the offgas as suggested by the iron content, and results should improve greatly on a larger scale. The fume concentrate was very small because of the small charge available for the test; therefore, extensive analysis could not be done.

In summary, the beneficiation flow scheme for the Coal Creek slimes would include primary concentration by a cassiterite float circuit with a rougher and a cleaner bank of cells, then upgrading the cleaner concentrate by fuming.

DESIGN OF 1,000-st/d PROCESS FLOWSHEET

Results of the bench-scale tests were combined and used to calculate the mass flow for a 1,000-st/d beneficiation plant. The Reichert LG-7 spiral was selected as the primary concentrator over the Humphrey's spiral or the Deister table because of the higher capacity and the ability to treat a feed with slimes at an efficiency almost equal to that of the others in treating a feed without slimes.

Table 18.—Bartles-Mozley separator concentrates from DH 2 and 3 slimes, percent

Sample	Percent solids	Weight dist	Sn		Ag		Zn		Fe		As	
			Grade	Dist	Grade[1]	Dist	Grade	Dist	Grade	Dist	Grade	Dist
DH 2 ..	20	32.2	0.21	52.2	0.1	19.4	0.11	33.6	2.3	26.4	0.063	20.7
DH 3 ..	20	27	.31	61.3	.9	35.4	.59	45.8	1.7	20.1	.082	59.2
DH 3 ..	50	26	.33	49	3.4	63.8	.76	40.5	3	26.0	.065	31.5

Dist   Distribution.
[1]Troy ounces per short ton.

The secondary concentration scheme tested in the locked-cycle test was not chosen for the final flow scheme, because it ground the table tail only after the first pass over the table. This circuit produced a concentrate with 20 pct plus 65 mesh. This concentrate was effectively cleaned by sulfide flotation only when finer than 65 mesh; therefore, the table concentrate needed to be ground too. Eventually all the material from the spiral concentrate is ground, so one regrinding circuit is used. Moving the regrind circuit so that it is right after the spiral resulted in another change from the locked-cycle scheme, the elimination of the scavenger tables used to treat the reground first table pass tails. The function of the eliminated tables, to separate out a tail, can be done on the first table. The first table now not only collects the concentrate but, because the ore is minus 65 mesh, can also reject the low-grade tail.

Cleaning of the gravity concentrate was most effectively accomplished by sulfide flotation, which can give a float tail of 50 pct Sn. Cassiterite flotation was not able to produce a final product that did not need further processing, even with depressants or pretreatment. The nitric acid leach was as effective as the sulfide flotation to remove the iron and arsenic, but tin loss was slightly higher and zinc removal was much less. In addition, the leaching would be more reagent consuming. To obtain the 50-pct Sn tail, the sulfide flotation feed needed to be about 25 pct Sn. Sample DH 2 could achieve this grade with the single table pass described above, but DH 3 required a second table pass. Therefore, the final flow scheme included a second table circuit to provide for the needs of the whole ore body represented by the samples received.

Table 19 lists the major equipment required to process crushed ore, including quantity and size for most of the items. The flowsheet, shown in figure 10, begins with primary concentration of minus 20-mesh ore with the slimes fraction included by 10 triple Reichert LG-7 spirals (5.1 st/h per triple spiral with a 1.25 overdesign factor). The spiral concentrate is then ball-mill ground to minus 65 mesh, deslimed at 325 mesh, and tabled on four triple-deck shaking tables, with tails discarded and a middling recycled to the table feed.

The upgrading of the table concentrate prior to sulfide flotation, necessary for DH 3, can be performed on one table that rejects a sulfide tail, recycles a middling, and gives an upgraded tin concentrate. The sulfide flotation circuit was expanded to include a cleaner bank of cells. The results used to calculate cleaner flows were from a bench test performed on an oxidized feed that was not pretreated by acid scrubbing. The efficiency of the cleaner might therefore be low because one of the effects of the pretreatment was the depression of cassiterite.

Table 19.—Equipment list for a 1,000-st/d plant

Rod mill	6-ft diam by 10 ft, 93.5 hp.
Reichert LG-7 spirals	10 triple spirals.
Hydrocyclone	2; classify at 65 mesh and 325 mesh.
Ball mill	6-ft diam by 6 ft, 75.7 hp.
Shaking table	11: 10 triple-deck tables for secondary concentration, 1 full-size table for upgrading prior to flotation.
Thickener	Dewater table concentrate.
Conditioner	5-min residence time for acid scrub.
Sulfide flotation	27-ft^3 cells; 21: 10 rougher, 5 cleaner, 6 scavenger.
Filter	Dewater flotation concentrate.
Slimes treatment:	
Thickener	Dewater slimes from classifiers.
Conditioner	10-min residence time.
Sn flotation	27-ft^3 cells; 21: 18 rougher, 3 cleaner.
Filter	Dewater flotation concentrate.
Drier	Dry filter cake prior to fuming.
Fume furnace	Extract Sn from dried cake.
Bag house	Collect fumed Sn as final slime concentrate.

An optional slimes beneficiation circuit was calculated, although the minor increase of 2 pct in tin recovery would not merit the expense. The slimes available to be treated represent about 9 wt pct of the spiral concentrate or only 1.7 pct of the overall weight and 3.7 pct of the overall tin, because the LG-7 spiral is used and most of the initial slimes have already been treated. After the pulp is thickened, cassiterite is floated using a rougher-cleaner flotation scheme. The concentrate is dried and fumed without sulfide addition. Reagent consumption for the beneficiation plant is given in table 20. The low tin grade of the slimes translates into an Aero 845 dosage of over 1.7 lb/lb of tin, which costs approximately $3.00, about the price of a pound of tin.

Table 20.—Reagent usage for a 1,000-st/d plant

Operation	Reagent	Dosage, lb/st conc
Acid scrub conditioner	H_2SO_4	5.34
Rougher conditioner	Lime	5.37
	Aero 350	2.56
	Aerofloat 208	2.56
	Dowfroth 400	9
Scavenger conditioner	$CuSO_4$	.49
	Aero 350	1.53
	Dowfroth 400	9
Cleaner conditioner	Aero 350	1.38
	Dowfroth 400	4.4
Slime: Sn flotation	H_2SO_4	9.1
	Aero 845	7.2

Conc   Concentrate.

KEY

	Weight	Unit dist	Overall dist
Sn:	lb/h	pct	pct
Ore wt:	st/h	pct	pct

ND = not determined

From grinding circuit

Feed

114.17	100.00	100.00
41.67	100.00	100.00

Spiral

Concentrate

97.39	85.3	85.3
8.33	20.0	20.0

Tail

16.78	14.7	14.7
33.34	80.0	80.0

Screen (65 mesh)
Oversize → Ball mill
Undersize

Screen (325 mesh)
Oversize   Undersize

Slimes

4.28	4.40	3.7
0.72	8.60	1.7

Table   **Middling**

Tail

2.15	2.3	1.9
7.12	93.6	17.1

Middling

39.20	ND	ND
2.42	ND	ND

Thickener

Concentrate

90.96	97.7	79.7
0.49	6.4	1.2

Tail

1.30	31.30	ND
0.10	70.80	ND

Flotation, Sn

Middling   Table

Tail

1.82	2.0	1.6
0.12	31.3	0.3

Concentrate

4.14	74.1	ND
0.14	16.8	ND

Tail

1.44	25.9	1.2
0.68	83.2	1.6

Flotation, Sn

Concentrate

89.14	98.0	78.1
0.37	68.7	0.9

Concentrate

2.84	68.70	2.5
0.04	29.20	0.1

Concentrate

24.09	21.8	ND
0.13	38.2	ND

Tail

4.55	63.1	ND
0.23	58.6	ND

Filter drier

Flotation, sulfide

Fume

Tail

0.66	23.2	0.6
0.03	80.5	0.1

Tail

110.57	93.9	ND
0.34	46.6	ND

Concentrate

7.21	6.1	ND
0.39	53.4	ND

Concentrate

2.18	76.8	1.9
0.01	19.5	<0.1

Flotation, sulfide   Flotation, sulfide

Tail

86.48	78.2	75.8
0.21	61.8	0.5

Concentrate

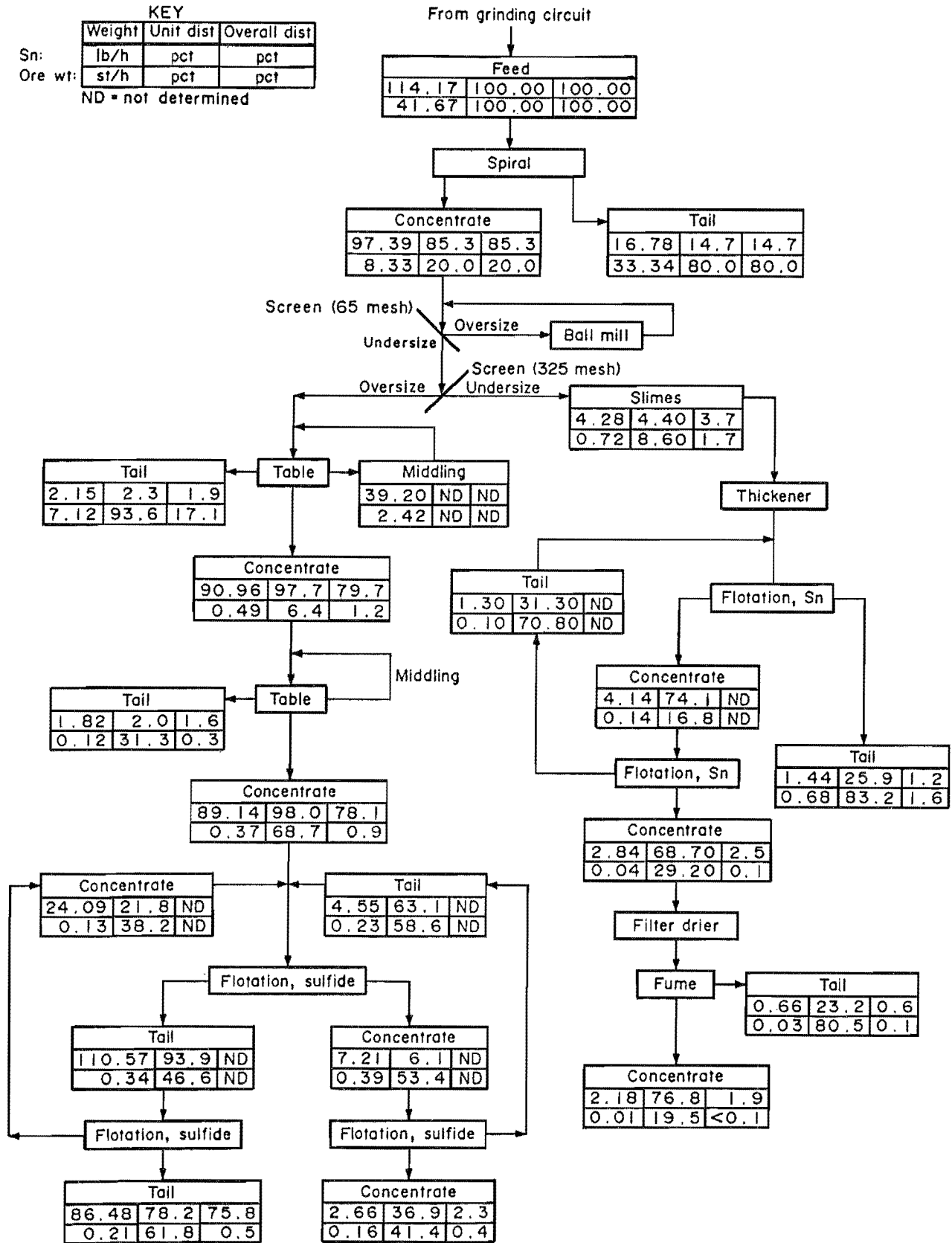| 2.66	36.9	2.3
0.16	41.4	0.4

Figure 10.—Flowsheet and mass flow rate for 1,000-st/d mill.

CONCLUSIONS

Results of laboratory tests showed that tin in the Coal Creek ore can be concentrated effectively by gravity methods, but the gravity product will contain a high concentration of sulfide minerals that include a significant amount of unwanted arsenic. Most of the sulfides, however, can be removed by flotation. Silver will be concentrated with the sulfide minerals, which will probably require an oxidation treatment before the silver can be recovered by cyanidation.

Enough data was obtained in the laboratory tests to design a flowsheet for a 1,000-st/d beneficiation plant. Primary concentration of minus 20-mesh ore is performed by the Reichert LG-7 spiral, which showed the ability to concentrate an unclassified ore (16-pct Sn loss to an 80-wt-pct tail) with only slightly lower efficiency than processing the deslimed ore (15-pct loss). Secondary concentration is performed by tabling after regrinding the spiral concentrate to minus 65 mesh. A tail below 0.02 pct Sn is achieved at 65 mesh on the table.

The gravity concentrate is cleaned by a sulfide flotation scheme that includes rougher, cleaner, and scavenger cell

banks. A final concentrate tin grade of 50 pct is achieved with an overall recovery of 76 pct (fig. 10).

The arsenic level was more of a problem for the deeper equigranular granite part of the ore body than for the more weathered seriate granite. Gravity concentrates may contain up to 10 pct As, which can be lowered 2 to 3 pct by sulfide flotation. Arsenic in the sulfide concentrate also presents a problem for silver recovery, since almost 90 pct of the silver values in the gravity concentrate are found in these refractory products.

The volume of slimes to be processed separately is minimized by the use of the LG-7 spiral. Tin flotation gave the highest grade and recovery for primary concentration of the slimes. The CF was as high as 13 for a 2.4-pct Sn concentrate. Fuming at 1,000° C for 3 h without sulfide addition extracted 77 pct of the tin from the flotation concentrate. The slimes beneficiation circuit would be optional because it is cost intensive but raises the overall tin recovery by only 2 pct.

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