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Report of Investigations 4867

TREATMENT TESTS OF SCHEELITE ORES AND TAILINGS

BY A. L. ENGEL

United States Department of the Interior — April 1952

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UNITED STATES DEPARTMENT OF THE INTERIOR
Oscar L. Chapman, Secretary
BUREAU OF MINES
J. J. Forbes, Director

April 1952

TREATMENT TESTS OF SCHEELITE ORES AND TAILINGS

by

A. L. Engel[1]

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1/ Metallurgist, Hydrometallurgical and Ore-Dressing Branch, Metallurgical Division, Bureau of Mines, Reno, Nev.

Report of Investigations 4867

INTRODUCTION AND SUMMARY

This report is a compilation of results of preliminary tests on scheelite (tungsten) ores and tailings. These investigations were made to assist in establishing satisfactory treatment methods for small-scale concentrator operations, or to indicate a method for conserving previously lost mineral values in old tailings, if possible.

A discussion of test procedures is included, as it was felt that this would add to understanding the results reported under the several projects.

The treatment of scheelite ores has become more or less standardized. Important differences occur in textures of ores from various mining districts, however, and this must be considered when planning ore-dressing operations. In most of the present investigations, only a limited amount of work was done, usually just enough to answer a specific question in regard to a particular ore or concerning local conditions.

The methods of treatment included gravity concentration, magnetic separation, and flotation.

Three projects came from California. The rest were from Nevada.

A summary of test results is shown in table 1.

TABLE 1. - Summary of test results

Project No.	Source of sample: State	Source of sample: District	Heads, percent WO_3	Preferred treatment	Recovery, percent
Re-0-2.13	Calif.	Victory Mine, Kings County	1.50	Gravity concentration at -10-mesh	60
Re-0-2.52	do.	Mono County	.26	Gravity and magnetic concentration at 48-mesh	54
Re-0-4.30	do.	Red Mountain, San Bernardino County	.34	Flotation	60-80
Re-0-2.45	Nevada	Gabbs, Mineral County	1.8	Gravity or flotation	65 99
Re-0-2.19	do.	Rawhide, Mineral County	.3	Flotation at -200-mesh	70
Re-0-2.55	do.	Oak Springs, Nye County	.76	Gravity at -40-mesh	72
			.61	do.	58

ACKNOWLEDGMENTS

The work described in this report was done under the general supervision of J. B. Zadra, chief of the Hydrometallurgical and Ore-Dressing Branch, Metallurgical Division, Bureau of Mines, Reno, Nev. Portions of the test work were done by E. S. Shedd and H. J. Heinen, metallurgists at the Reno Station. The detailed microscopic examinations of test products from project Re-0-2.13 were made by E. T. Schenk, mineralogist at the Boulder City station.

TEST PROCEDURES

Gravity Concentration

Gravity concentration of scheelite is based on the fact that the specific gravity or unit weight of the mineral is considerably greater than those of the minerals with which it is usually found. This difference in specific gravity is accentuated when the minerals are suspended in water; and, providing the scheelite is liberated from the other minerals by crushing or grinding, the separation is readily accomplished, usually on such standard apparatus as the shaking table.

A preliminary examination of any sample to be studied is made by crushing the ore to a suitable size for screen analysis and sink-and-float tests. Scheelite seldom occurs in other than a disseminated, fairly fine-grained form, and it is necessary to crush or grind the ore to obtain satisfactory liberation, or at least to be able to discard a large amount of the ore as tailings.

The ore, having been crushed to minus-10-mesh, for example, is screened and separated into as many size products as desired using standard screens on a shaking frame. These sized products are weighed to determine the proportion of the total weight represented by each. If the screened portions appear to need washing to remove adhering fines, this is done, and the fines are wet-screened into their respective categories and all products are dried. The separate products usually are combined into three or more groups, roughly corresponding to the size ranges of feed for concentration on shaking tables, as, for example, coarse sands 10- to 65-mesh, fine sands 65-to 200-mesh, and slimes. Sometimes the minus-200-mesh screened portion is deslimed by washing, and an additional fine sands portion is segregated.

The several table-feed portions are separated into sink products roughly corresponding to concentrates and float products corresponding to tailings by immersion in acetylene tetrabromide, a heavy liquid with 2.9 specific gravity. All minerals or rock fragments that have higher specific gravity than 2.9 sink in the liquid, while the particles with lower gravity float on top. The products of this separation are drained to recover most of the heavy liquid, and the rest of the liquid is removed by repeated washing with benzol. The washed products are dried, weighed, and prepared for chemical analysis.

When the ore contains garnet and other heavy minerals as well as scheelite, the sink products are contaminated and usually quite low in tungsten content. Examination of both sink and float products with the microscope is a standard procedure employed in addition to chemical analysis and enables close determination of the grain size at which optimum liberation has occurred. It also shows the form in which scheelite occurs in the middlings, whether or not, locked with gangue and the form in the tailings.

If desirable, this preliminary gravity-concentration test is followed by a laboratory-scale test in which the ore is crushed and carefully stage-ground to the most suitable size for liberating an optimum amount of scheelite without overgrinding and consequent production of excess slimes. The ground ore is classified hydraulically into two or more sand products and slimes. Each of these products is individually passed over a shaking table and separated into concentrates, middlings, and tailings. This type of test yields results that are frequently comparable to results to be expected in plant operation, both in regard to grade of concentrates and tailings and recovery of tungsten.

The table concentrates may be contaminated with garnet and other heavy minerals. High-intensity magnetic separation is used to remove garnet and other minerals that are weakly magnetic. When this treatment is necessary, the final tungsten product usually is conformable to market specifications for high-grade concentrates.

Flotation

In crushing or grinding most scheelite ores, a considerable amount of slimes is formed. This material is usually minus-200-mesh and, in fact, includes particles of ore that are very much finer. Most of the losses of tungsten in gravity concentration are in these slimes, which do not separate readily by gravity treatment. Other losses in gravity concentration are found in locked grains of either middlings or tailings. These locked grains contain more or less scheelite mineral unliberated from the gangue minerals in grains whose specific gravity is not high enough to cause them to report in the gravity concentrates. To recover scheelite from such locked grains, it would be necessary to regrind them, which probably would result in formation of more slimes and would not improve the over-all recovery of tungsten by gravity concentration alone. Thus the slimes from either original or later grinding of the ore are the most frequent source of flotation feed. Also, in some ores, particularly low-grade ores, the grains of scheelite are very small and disseminated so that original fine grinding is necessary to liberate them. Their fineness when liberated prevents much recovery by gravity treatment, and in such cases flotation is indicated.

In laboratory testing, even when results indicate considerable recovery of scheelite by gravity concentration, it may be convenient to grind portions of the original ore for flotation. The results of this type of test will indicate the over-all recovery of tungsten. Concentrate grade will be lower than in plant operation, but the tailings analysis will be an accurate guide to the extraction that may be expected.

The reagents used for flotation have become quite standardized over a period of about 15 years. Sodium carbonate is used to adjust the pH of the ground ore to the proper point of alkalinity. Sodium silicate is sometimes used to assist dispersion of colloidal matter. A fatty acid reagent is the usual collector, and this can be modified to give greater selectivity by combination in dilute solution with an anionic reagent such as Aerosol OT. In most cases a reagent also is needed to depress such gangue minerals as calcite, which float fairly readily with fatty acids. Satisfactory depressant reagents are the various tannins, such as quebracho or AC 651.

Several cleaning stages usually are required in plant operation to produce a fairly high-grade flotation concentrate. If a still-higher-grade product is desired, the flotation concentrates are re-treated by gravity concentration on tables and possibly by magnetic separation also. Any tailings or middlings from such re-treatment are returned to the flotation circuit, or, if necessary, reground before returning to flotation. More recently, such cleaner tailings are treated by a wet chemical process.

In the laboratory, however, small quantities of final or semifinal products do not allow very elaborate or complicated re-treatment. Also, as many of the investigations are concerned with small-scale operations, it is more suitable to determine results of the simplest kind of treatment only, commensurate with satisfactory economical operations. For these reasons, it was not unusual that laboratory testing was terminated with the production of comparatively low-grade concentrates and without attempting to recover further amounts of scheelite from cleaner tailings or middling products.

SCHEELITE ORE FROM VICTORY MINE, KINGS COUNTY, CALIF.

A sample of ore was submitted to establish a basis of comparison between experimental laboratory results and plant results obtained in the concentrator at the mine. A small sample of mill tailings also was submitted for study, since losses of scheelite seemed excessive.

The ore sample was crushed in the laboratory rolls to pass a 10-mesh screen. The crushed ore was screened into coarse, medium, and fine portions, and all portions except a minus-200-mesh portion were treated by sink-float tests in a heavy liquid, acetylene tetra-bromide, with specific gravity of 2.9. By this means, the scheelite and heavy gangue minerals were separated from the lighter gangue minerals. The gangue minerals were garnet and quartz, with minor amounts of epidote, calcite, and pyrite. Chemical analyses of the test products were made, and excellent liberation of the scheelite was indicated by the low tungsten contents of the float products, as shown in the following table:

Product, size, mesh	Weight, percent	WO_3, percent	Total WO_3, percent
Sink -10+28........	30.1	1.51	30.3
Sink -28+65........	18.8	2.21	27.7
Sink -65+200.......	11.8	2.82	22.2
Total sink.....	60.7	1.98	80.2
Float -10+28........	11.9	.04	.31
Float -28+65........	8.8	.01	.05
Float -65+200.......	5.6	.01	.04
Total float....	26.3	.02	.40
Minus-200-mesh......	13.0	2.24	19.4
Composite heads.....	100.0	1.50	100.0

A screen analysis of the mill-tailings sample indicated that 90 percent of the loss was in the minus-200-mesh portion, and that possibly the current mill practice resulted in overgrinding the ore before concentration. Details of this test were:

Product, size, mesh	Weight, percent	WO_3, percent	Total WO_3, percent
-28+65.............	25.1	0.06	2.3
-65+200............	44.7	.11	7.6
-200...............	30.2	1.92	90.1
Composite..........	100.0	.64	100.0

Since the preliminary sink-float test had given satisfactory indications, 20-kilograms of minus-10-mesh ore was hydraulically classified in the constriction-plate tube classifier. Four products were made: Coarse, medium, and fine sands and slimes. Each of these was treated separately on the laboratory Wilfley table to produce gravity concentrates, middlings, and tailings.

The middlings and coarse tailings contained very little free scheelite, the values in them consisting of coatings of scheelite on gangue grains and, to a small extent, of very fine particles of scheelite locked with gangue. Apparently, about 80 percent of the total scheelite had been liberated by crushing the ore to minus-10-mesh. Of this, about 60 percent was liberated in grains that could be recovered by gravity concentration, and another 20 percent was too fine for gravity treatment. The remaining 20 percent of the scheelite in the ore could not be liberated except by finer grinding and when liberated also would be too fine for gravity treatment.

If the mill heads corresponding to the sample of mill tailings tested were the same as the ore investigated, a recovery of about 60 percent at the mill was indicated. Apparently, the ore at the mill was ground to 99.5 percent minus-28-mesh, as against 70 percent minus-28-mesh when the ore was crushed to minus-10-mesh only. 90 percent of the scheelite lost in the mill tailings was in the minus-200-mesh portion representing about 36 percent of the total scheelite in the heads, while, of the sheelite not recovered, in the laboratory gravity concentration test, only about 10 percent was in the minus-200-mesh size range, and the remainder was mostly locked in the middlings.

The results of the laboratory test suggest that crushing the ore to minus-10-mesh, followed by regrinding the middlings to at least minus-28-mesh might have some operating advantages over the mill practice of grinding the entire mill feed to minus-28-mesh. To increase the recovery of scheelite beyond about 60 percent, it would be necessary to treat the slime tailings by flotation, as has been done successfully in a number of plants.

A detailed tabulation of the laboratory gravity-concentration tests on ore crushed to minus-10-mesh follows:

TABLE 2. - Laboratory gravity concentration of scheelite ore from Victory mine, Kings County, Calif.

Product		Weight, percent	WO_3, percent	Total WO_3, percent
Concentrates -	Coarse	1.120	47.68	35.0
	Medium	.215	53.50	7.5
	Fines	.375	57.04	14.0
	Slimes	.046	47.00	1.4
Total		1.756	50.40	57.9
Middlings -	Coarse	21.881	1.24	17.7
	Medium	5.645	.73	2.7
	Fine	2.723	2.31	4.1
Total		30.249	1.24	24.5
Tailings -	Coarse	21.104	.28	3.9
	Medium	11.985	.06	.4
	Fine	22.512	.18	2.6
Total sand tails		55.601	.19	6.9
Slimes		12.394	1.32	10.7
Total tails		67.995	.40	17.6
Composite heads		100.000	1.53	100.0

SCHEELITE ORE FROM MONO COUNTY, CALIF.

This ore was submitted by the former Mining Divsion, Bureau of Mines. The purpose of the test was to produce high-grade concentrates and to determine if the amount of molybdenum present in them would be above the limit of 0.40 percent. The sample contained 0.26 percent WO_3 and 0.015 percent Mo. The minerals present were scheelite, powellite, wollastonite, garnet, quartz, and calcite.

Preliminary gravity-concentration tests on a small portion of the sample crushed to minus-10-mesh indicated that the scheelite was well-liberated at 48-mesh, while the powellite was liberated at about 100-mesh. Concentrates containing 23.63 percent WO_3 also contained 0.17 percent Mo.

To obtain more conclusive results, the entire remainder of the sample was stage-ground to minus-48-mesh, hydraulically classified into two sand products and slimes, and concentrated on the laboratory Wilfley table. The table concentrates were separated into several size portions by screening. Each portion of concentrates was then treated on the high-intensity magnetic separator, to remove garnet and other gangue minerals. The nonmagnetic sand concentrates were again cleaned by panning to produce as high-grade scheelite concentrates as possible.

Recovery of scheelite was 54 percent, at a grade of 50.65 percent WO_3. This product contained 0.49 percent Mo. As shown in table 3, 40.9 percent of the scheelite recovery was in a concentrate carrying 70.62 percent WO_3 and 0.68 Mo. Only the concentrates were analyzed for molybdenum. Results from tabling the slimes were poor, so the products were recombined.

TABLE 3. - Gravity and magnetic separation test of scheelite ore from Mono County, Calif.

Product	Weight, percent	Analysis, percent WO$_3$	Analysis, percent Mo	Distribution percent, WO$_3$
Nonmagnetic panned concentrates				
Coarse..........................	0.145	70.62	0.68	40.9
Medium..........................	.108	26.98	.19	11.6
Fine............................	.014	27.78	.77	1.5
Composite concentrates............	.265	50.65	.49	54.0
Magnetic middlings				
Coarse..........................	.453	4.46	-	8.0
Medium..........................	.301	.48	-	.6
Magnetic tailings				
Coarse..........................	.736	.39	-	1.2
Fine............................	1.145	.69	-	3.2
Composite middlings...............	2.635	1.23	-	13.0
Med. magnetic tails...............	7.990	.08	-	2.6
Coarse table tails............	43.110	.03	-	5.7
Fine table tails..............	14.090	.01	-	.7
Composite sand tails..............	65.190	.03	-	9.0
Slimes............................	31.908	.19	-	24.0
Calculated heads..................	100.000	.25	-	100.0

SCHEELITE TAILINGS FROM SAN BERNARDINO COUNTY, CALIF.

Two sacks of tailings from previous milling operations were submitted for flotation testing. The minerals present were quartz, garnet, altered silicates, and scheelite. The average content of the material was 0.34 percent WO$_3$. Most of the scheelite was in the finer portions, but some free scheelite grains were noted in the coarser sands and also scheelite middlings or locked grains, although not enough to warrant separation by gravity concentration for possible regrinding for flotation.

A screen analysis was made with results shown in table 4. There was so much clayey material in the sample that very thorough washing on the screens was necessary. Most of the scheelite was in the minus-200-mesh slimes. As indicated, little recovery could be expected by gravity concentration.

TABLE 4. - Screen analysis - scheelite tailings from San Bernardino County, Calif.

Product, size, mesh	Weight, percent	WO_3, percent	Distribution, percent
+20..................	4.6	0.10	1.4
-20+48...............	4.0	.13	1.6
-48+65...............	4.1	.17	2.1
-65+100..............	10.4	.16	5.0
-100+150.............	8.5	.13	3.3
-150+200.............	11.0	.03	1.0
-200 sands...........	12.5	.34	12.8
-200 slimes..........	44.0	.54	72.8
Composite............	100.0	.33	100.0

The simplest form of treatment appeared to be flotation of the entire material after thorough repulping to free as much scheelite as possible. The use of flotation cells with scrubbing action as well as thorough agitation would be preferable, as the flotation feed required prolonged agitation and conditioning before actual flotation.

It was necessary to agitate each charge of flotation feed for 1/2 hour to obtain satisfactory dispersion of the clayey portion so that the scheelite in the slimes could be liberated. Some froth was formed, indicating the presence of previous flotation reagents or perhaps a natural constituent of the tailings. The pulp had a natural pH of 8.0.

2.0 pounds of soda ash and 0.25 pound of sodium silicate per ton of ore were used as conditioning reagents, which caused adjustment of the pH to about 10.0.

The other reagents used for flotation were 0.5 pound oleic acid, 0.25 pound Aerosol OT-85, and 0.1 pound frother B-23 per ton of ore. These produced a voluminous, slimy froth. The froths from six charges were combined for partial cleaning. A tannin reagent, A. C. 651, was used for cleaning. In four stages, 2.0 pounds of A. C. 651 per ton of ore were used. Concentrates carrying 5.33 percent WO_3, representing about 60 percent recovery of scheelite, were made. The rougher tailings carried 0.08 percent WO_3. Over-all recovery was about 80 percent, and, in continuous plant operation, this figure should be approximated, together with better grade concentrates.

Several gallons of water representing the supply available for a proposed treatment plant were used in flotation tests in comparison with Reno tap water. Results were similar, so the water was apparently satisfactory.

This material required a rather delicate balancing of flotation reagents, particularly the preliminary conditioning reagents, as slight excess of either soda ash or sodium silicate depressed the scheelite. At the same time, both reagents were required to enable proper dispersion of the pulp and satisfactory collecting action from the oleic acid and Aerosol OT-85.

SCHEELITE ORE FROM MINERAL COUNTY, NEV.

This was a comparatively high-grade tungsten ore, containing about 1.8 percent WO_3 as scheelite. The gangue minerals included garnet, quartz, calcite, feldspar, and a little pyrite.

Preliminary screen analysis and gravity concentration indicated the necessity of grinding the ore to minus-48-mesh for satisfactory liberation of the minerals. However, by crushing the ore to minus-14-mesh only, apparently about 85 to 90 percent recovery of scheelite could be made by gravity concentration in medium-grade concentrates. The results of these tests are shown in table 5. The separation into sink and float products was made by introducing screen-sized portions of the ore, which had been crushed to minus-8-mesh, into acetylene tetrabromide, a heavy liquid with 2.9 specific gravity.

TABLE 5. Sink and float test, scheelite ore, from Mineral County, Nev.

Product, size, mesh	Weight, percent	WO_3, percent	Total WO_3, percent
Sink			
-8 + 14	4.21	8.25	19.2
-14 + 28	1.74	15.53	14.5
-28 + 48	1.10	27.42	16.5
-48 + 150	1.44	29.84	23.1
-150 sands	.56	32.40	9.8
Total sink	9.05	17.02	82.9
Float			
-8 + 14	46.79	.32	8.1
-14 + 28	18.76	.17	1.7
-28 + 48	8.90	.16	.8
-48 + 150	7.85	.05	.2
-150 sands	6.44	1.41	4.9
Total float	88.75	.33	15.7
Slimes	2.20	1.18	1.4
Composite	100.00	1.86	100.0

Several flotation tests were made on ore ground to minus-100-mesh. Nearly complete recovery (99.8 percent) was achieved with concentrates containing about 12 percent WO_3. Most of the pyrite remained in the tailings. The reagents used were conventional. The ground ore was conditioned for 10 minutes with 0.5 pound of sodium silicate per ton. This changed the pH of the pulp from 6.0 to 7.5. Two increments of 0.15 pound per ton each of oleic acid with 0.10 pound Aerosol OT-85, and 0.10 frother B-23 produced a typical lacy froth. The tailings contained less than 0.01 percent WO_3.

SCHEELITE TAILINGS FROM MINERAL COUNTY, NEV.

Two small samples of gravity-concentration tailings were received for investigation of possible re-treatment methods to recover scheelite left in them. The old tailings sample was said to be representative of 100,000 tons of tailings and the new tailings samples, of 5,000 tons. The old tailings was highly altered with heavy koalinization of the silicate minerals. The WO_3 content was about 0.3 percent. The sample also contained 0.04 percent Mo, 0.35 percent Cu, and 0.1 percent S. No free scheelite or sulfide minerals were noted. All the material was minus-10-mesh but fairly coarse. The new tailings was little altered. The WO_3 content was also about 0.3 percent. Other constituents were 0.03 percent Mo, 0.07 percent Cu, and 0.38 percent S. No free scheelite but some sulfides were noted. Also, all the material was minus-10-mesh but somewhat coarser than the old tailings, possible because it was less altered. Screen analyses of both samples were as follows:

TABLE 6. - Screen analyses - scheelite tailings from Mineral County, Nev.

Product size, mesh	Weight, percent	WO_3, percent	Total WO_3, percent
New tailings			
-10 + 48	42.3	0.16	23.6
-48 + 100	17.0	.16	9.1
-100 + 200	13.3	.15	7.0
-200 + 325	7.3	.20	5.1
-325	20.1	.79	55.2
Composite	100.0	.29	100.0
Old tailings			
-10 + 48	32.0	.15	16.0
-48 + 100	29.5	.23	22.5
-100 + 200	16.1	.30	16.1
-200 + 325	6.3	.33	6.9
-325	16.1	.72	38.5
Composite	100.0	.30	100.0

As indicated, the largest part of the scheelite in each sample was in the minus-325-mesh slimes. Some of this may have been free scheelite. Preliminary experiments on the minus-325-mesh portion of the old tailings showed that even after thorough conditioning with several usually efficacious dispersing reagents, very little concentration could be achieved by gravity methods of treatment.

The best flotation results were achieved by grinding the old tailings to minus-200-mesh and adding 3.5 pounds of soda ash per ton of ore to adjust the pH to 9.4. Further conditioning was given with 0.75 pound of sodium silicate and 0.25 pound quebracho, for 10 minutes. Oleic acid modified with Aerosol OT-85 was used as collector reagent, in two increments of 0.25 pound of oleic per ton and 0.125 pound of OT-85. Two increments of 0.1 pound of frother B-23 also were used. The rougher concentrates were cleaned once without further reagents, and a product containing 2.30 percent WO_3 was made. This represented 68 percent recovery of WO_3, with 11 percent more in the cleaner tailings. The rougher tailings assayed 0.10 percent WO_3.

Similar treatment of the new tailings produced approximately the same results.

SCHEELITE ORE FROM NYE COUNTY, NEV.

Two small samples were submitted to determine the lead and molybdenum contents of the ore as well as the tungsten, and the corresponding lead and molybdenum contents of gravity concentrates to be produced by tabling the ore. This was in connection with condemnation of the property for bombing range purposes.

The samples were crushed to minus-10-mesh and then ground to minus-40-mesh for table concentration after hydraulic classification into two sand products and slimes in each case. The products were all tabled separately, but the individual concentrates, middlings, and tailings were combined for assay.

Sample E. - Heads analyzed in percent: WO_3, 0.76; Mo, 0.04; Pb, 0.11 and Cu, 0.01

Product	Weight, percent	Analyses, percent			Total WO_3, percent
		WO_3	Mo	Pb	
Tables concentrates........	2.45	24.07	0.17	1.31	72.3
Middlings..................	11.65	.52	-	-	7.5
Tailings...................	85.90	.19	-	-	20.2
Composite..................	100.00	.81	-	-	100.0

Sample 4. - Crystal 2, Heads analyzed, in percent: WO_3, 0.61; Mo, 0.06; Pb, 0.01; Cu, 0.01.

Product	Weight, percent	Analyses, percent			Total WO_3, percent
		WO_3	Mo	Pb	
Table concentrates.........	1.27	29.83	1.07	0.03	58.4
Middlings..................	13.84	.86	-	-	18.0
Tailings...................	84.89	.18	-	-	23.6
Composite..................	100.00	.65	-	-	100.0