AMENABILITY OF SOME ARIZONA MANGANESE ORES TO CONCENTRATION BY FLOTATION

by

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AMENABILITY OF SOME ARIZONA MANGANESE ORES TO CONCENTRATION BY FLOTATION

CHAPTER I - INTRODUCTION

Current interest in the manganese ore resources of the United States, and the consideration given them in the Strategic War Minerals Program of the United States Government, is a consequence of the dependence of the American steel industries upon imported manganese ores. This situation is not due, primarily, to a shortage of manganese in this country, but to the low-grade nature of domestic deposits. As much of the higher-grade manganese ore in Arizona was mined and shipped during the World War, the present problem consists of devising satisfactory methods for the concentration of the lower-grade materials which remain.

Although a survey of the situation revealed possibilities of treatment along gravity, magnetic, flotation, and hydrometallurgical lines, the experimental work described in this paper was limited to flotation methods. The primary reason for this limitation lay in the character of the ores themselves. Fine grinding is necessary for good liberation of the manganese minerals in many Arizona ores, and, consequently, neither gravity nor magnetic methods seemed likely to provide a complete process.

Because of their possible application to Arizona ores, several experimental and plant flotation operations on manganese ores deserve mention. The outstanding instance is that of the Cuban-American Manganese Corporation concentrator in Cuba,(10) [1] where several thousand tons of manganese oxide ore, containing about 20 per cent of manganese, have been concentrated to 48 to 50 per cent of manganese by flotation. The method in use is based upon the patent disclosures of A. J. Weinig(16,17) and the reagents used consist of Lousiana crude oil, a fatty acid, and pine oil in the rougher circuit with tannin being added to each of the two cleaning stages of flotation.

In the Cuyuna district of Minnesota, a flotation unit treated ferruginous manganese slimes which were the tailings from a tabling operation.(3) On flotation heads assaying about 16 per cent of manganese, concentrates containing 23 per cent of manganese were produced with an extraction of 66 per cent. The reagents used were oleic acid, sodium silicate, sodium carbonate, and pine oil.

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1 A numerical system of reference is used throughout this paper. Numerals in parentheses refer to publications listed in the bibliography.

In the Chiaturi district of Russia, flotation has been tried in an attempt to recover the manganese in the slimes from a washing plant. Basmanov (11) reports agitating the slime with sulphuric acid for 10 to 15 minutes and then adding sodium oleate and water glass and finally floating the manganese minerals with pine oil as a frother. On sized material, minus 100 plus 250 mesh, the use of 0.2 to 0.5 pound of sulphuric acid and 1.0 pound of sodium oleate per ton of slime resulted according to published statements in an extraction of 96 per cent, with a concentrate containing 48 per cent of manganese.

At the Emma Mine of Butte, Montana, several thousand tons of rhodochrosite ore averaging about 30 per cent of manganese and 20 per cent of silica have been concentrated by flotation to a product containing 38 to 40 per cent of manganese and less than 6 per cent of silica, with an extraction of 95 per cent. The process, as developed by F. D. DeVaney and J. B. Clemmer (4,5) for the treatment of the Emma Mine ore, consists of a preliminary removal of the sulphide minerals by flotation employing a small amount of copper sulphate, xanthate, and pine oil, after which flotation of the rhodochrosite was practiced using crude sodium oleate and pine oil.

When the originators of this process applied it to oxide manganese ores, it was found that the use of sodium silicate and sodium carbonate was a necessary modification in order to obtain satisfactory silica dispersion. It was noted also, that oleic acid was preferable to the crude sodium oleate and palmitic acid was found to be more effective for some ores. Heating proved beneficial, particularly when employing the higher fatty acids, and in all cases a high pulp density was found to be desirable in the rougher flotation.

A report of the successful flotation of manganese oxides on a laboratory scale has been made by A. M. Gaudin and R. H. Behrens.[(7)] The work was done on the tailings from a magnetic concentrator at Philipsburg, Montana. The problem involved the separation of the manganese oxides from both carbonates and silica. As a promoter of manganese, oleic acid was found to be the most adaptable, but greater selection in removing the carbonates was obtained with palmitic, stearic, and undecylic acids. Fuel oil was used and was found to improve the recovery of manganese and the rejection of the silica. Sodium silicate was the most satisfactory dispersant for silica. Reducing agents, with a few outstanding exceptions such as citrates, tartrates, and dextrin, appeared to assist the flotation of the manganese oxides, while oxidizing agents such as potassium chlorate and chloride of lime, depressed them. The theory advanced for this latter effect was based on the fact that manganese soaps are manganous rather than manganic, and, hence, a prior surface oxidation is detrimental and a prior surface reduction essential for chemical filming.

Several United States patents covering the flotation of manganese minerals have been issued. Weed (15) explained the difficulties of the flotation of manganese on the basis of poor reagent-ore contact which is due to improper dispersion of the fatty acids and oils which are insoluble in water and in his patent proposed the use of a colloidal dispersion of the fatty acid and fuel oil. This emulsification was produced by the addition of triethanolamine, which forms a soap with a small part of the fatty acid and promotes its dispersion. The advantages claimed are both greater selectivity toward the manganese minerals and a much lower consumption of reagent.

Gutzeit (8) recommended the use of sodium stannate and sodium vanadate as activators of manganese oxides, with quebracho bark as a silica disperser. His patent covers the use of the isopolyacids in an activating capacity as distinct from the known action of the heteropolyacids. Flotation tests were made on manganese oxide ores at a temperature of 35 degrees Centigrade using stearic acid and amyl xanthate as promoters and terpin oil as the frother.

The use of the heteropolyacids such as phosphomolybdic and phosphotungstic as oxide mineral activators was patented by Arnold.[11] These activators have been employed in the flotation of chrome ores and are said to be satisfactory activators for manganese oxides.

Only a few instances of experimental work done on the flotation of southwestern manganese ores have been found. DeVaney and Coghill[3] made flotation tests on manganese ore from the Artillery Peak district, west of Congress Junction, Arizona, with the following results:

> "Concentrates were made, but their silica content was always high. ......concentration by present ore dressing methods appears hopeless."[3]

Blessing,[1] in his work on southwestern manganese-silver ores from the standpoint of the recovery of silver, had little success in floating the manganese minerals. The reagents and combinations tried were oleic acid, oleic acid with kerosene, paraffin, Emulsol X_1, mixed xylidines, Aerofloat, and xanthate. Unfortunately, only silver assays were reported, and the degree of success in floating the manganese minerals must be estimated on the basis of an assumed manganese-silver association.

The requirements of manganese ores and concentrates for the manufacture of ferromanganese are a minimum content in the vicinity of 40 per cent of manganese and a silica content not greatly in excess of 10 per cent. The object of the experimental work described herein is the investigation of the possibility of the production of concentrates meeting these requirements by the flotation of Arizona manganese ores.

CHAPTER II

MATERIALS AND EXPERIMENTAL PROCEDURES

Materials

The Arizona manganese ores, which the writer has had the opportunity to examine, may be classified roughly into three types. The highest grade and, to the writer's experience, the most common type, is a black, dense ore containing psilomelane and small amounts of pyrolusite distributed through a siliceous gangue. Calcite may or may not be present. The ores of the Patagonia, Tombstone, and Yuma areas are frequently of this type. Ore from the Bender Mine near Patagonia, Arizona, was selected as representative of this type, and microscopic examination of polished sections indicated that a reasonably complete liberation of the manganese minerals would be attained by grinding to minus 100-mesh. The calcium carbonate in the ore permitted the investigation of the separation of the manganese oxides from both siliceous and calcareous minerals.

A second type of ore consists of particles of siliceous gangue imbedded in a matrix of manganese minerals. Psilomelane is the chief manganese mineral, but pyrolusite is present in a much larger proportion than in the ores of the first type. Clay and chalcedony are present as free particles as well as in the manganese mineral groundmass. The only example of this type of ore which was observed came from the Artillery Peak district west of Congress Junction, Arizona. This is the largest known deposit of manganese in Arizona.

The third, and least extensive type of deposit noted, is that of the Cecil Martin claims near Sentinel, Arizona. The manganese oxides occur there in an almost exclusively calcareous gangue.

Partial analyses of the three ores are given in Table I.

Table I - Partial Analyses of Ores Tested

	Manganese %	Insoluble %	$CaCO_3$ %
Bender	22.91	43.14	10.81
Artillery Peak	12.90	64.82	-
Cecil Martin	11.46	-	-

Flotation Procedures

For purposes of qualitative estimation of the flotability of the manganese minerals, and the effects of various reagents, "tube" tests were made. Wark (13) gives a description of this method which consists of shaking a few grams of deslimed ore in a 50 cc. stoppered glass cylinder with the reagents to be tested. The black manganese minerals provided sufficient contrast to the light colored gangue to permit a rough visual interpretation of the degree of selectivity. In no case were the results of tube tests taken as conclusive of the selective action of reagents, but they were accepted as valid, when no tendency toward flotation could be observed. Thus, the chief usefulness of the tube method lay in the elimination of unlikely possibilities.

Flotation tests were made in a 100-gram capacity, transparent, plastic flotation cell of the mechanical type and of local design. If the results of the 100-gram tests warranted it, further tests were made in a 500-gram capacity Denver laboratory flotation cell.

Distilled water was used in all tests unless otherwise noted.

Grinding

Ores were crushed to minus 20-mesh in available laboratory crushing equipment and the fine grinding done in a porcelain Abbé mill using silex pebbles as the grinding media with a pebble to ore ratio of three to one. The grinding was standardized for the Bender ore when using the 5 inch diameter 100-gram capacity Abbé mill. When the 500-gram capacity Abbé mill was used, the grind was adjusted to suit the ore being tested.

Wet and dry grinding were compared from the standpoint of their effect upon flotation. It was foundthat a dry Abbé mill grind would give flotation results similar to those of a wet grind provided the dry ground ore was kept wet for several hours before making the flotation test.

Sizing

Sizing tests were made by hand using Denver Equipment Company sieves. The screen sizes used were 100, 200, and 350-mesh, and the size of opening of the 200-mesh screen was measured and found to conform to the Tyler Standard opening of 0.074 mm. for this mesh sieve. Sizing tests on concentrates or other agglomerated material were preceded by an alcohol, and, if necessary, a benzene wash, to break up the agglomerates. This was considered preferable to prolonged screening, as the latter may result in grinding of the softer material on the screen.

Dispersion Tests

To ascertain the minimum quantity of a dispersing agent, such as sodium silicate or tannin, necessary to effectively disperse the fine portion of an ore, dispersion tests were made. A series of 10 test tubes, each containing 4 grams of ore and 12 cc. of water was used, increasing amounts of the reagent tested being added to successive tubes. After shaking, the settling time was noted and the optimum or minimum concentration of the reagent for adequate dispersion was determined.

Heavy Liquid Separations

Heavy liquid separations were made using acetylene tetrabromide as the heavy medium. Its specific gravity of about 2.9 was satisfactory for the separation of the manganese oxides from siliceous and calcareous gangue minerals. It was found necessary to remove the minus 350-mesh material before making the separation. Five lots of 1 gram each of the plus 350-mesh material were separated by stirring the suspension vigorously, allowing the heavy fraction to settle, and then collecting each fraction on a filter. The heavy and light portions were allowed to accumulate on the filter papers and were each given a second treatment to assure getting the best possible separation.

Briquetting

To permit a microscopic examination of finely divided products of flotation tests, bakelite briquettes were made using a briquette mould of local design. A composition of 30 per cent of bakelite was found satisfactory with a heating period of one hour at a temperature of 120 degrees Centigrade.

Microscopic Procedure

The methods of Short[12] were followed in the microscopic identification of the manganese minerals. Etching tests were used as well as the microchemical, sodium bismuthate, test for manganese. The microscope was provided with polarizing equipment which permitted the identification of pyrolusite and manganite by their anisotropism.

Analytical Methods

For the analysis for manganese, a modification of Low's[9] oxalic acid method was used. As a quick approximation, in the case of the Artillery Peak ore, "spot" tests were made. The method could be used only on tailing samples and consisted of grinding a few tenths of a gram of the sample in an agate mortar and then pressing a small pat of the material adjacent to a similar pat of ground ore of known manganese content on a white paper. A series of samples of known manganese content permitted the determination of the manganese content of a tailing within one per cent on samples running not over 10 per cent.

Calcium carbonate determinations were made using the standard calcium oxalate method. In the case of the

Bender ore, an abbreviated method was devised. An excess of one tenth normal sulphuric acid was added to the sample which was then filtered. The filtrate was titrated with one tenth normal sodium hydroxide to a methyl orange end-point. The calcium carbonate content was calculated on the basis of the acid consumed. The method gave satisfactory comparative results on a series of products from a single flotation test, although the calculated head assay might be several per cent above the true value.

Insoluble determinations were made using Low's[(9)] method. If a silica analysis was desired, the insoluble residue was treated with hydrofluoric and then sulphuric acids and the silica content determined from the loss in weight of the residue.

Iron determinations were made using the permanganate method recommended by Low.[(9)]

Phosphorus determinations were made using the volumetric phosphomolybdate method given by Low.[(9)]

pH measurements were made using the LaMotte colorimetric indicators where possible and an instrument for the potentiometric determination of pH where necessary, due to turbidity of solutions or the presence of a substance antipathetic to the indicators.

CHAPTER III

EXPERIMENTAL WORK ON FLOTATION

Tests on Samples from the Bender Mine

Preliminary tests were made on the Bender ore to estimate the highest degree of extraction and selection possible by physical methods. A sample of the ore was ground to pass a 200-mesh sieve and then screened on a 350-mesh sieve. A heavy liquid separation was made, as described in Chapter II, on the plus 350-mesh portion, and all products were analysed to determine their manganese content. The results are given in Table II.

Table II - Tests to Determine the Degree of Liberation

	Weight %	Manganese %	% of total Manganese
Head	100.0	22.79	100.0
Plus 350-mesh light fraction	13.5	4.19	2.5
Plus 350-mesh heavy fraction	8.8	42.08	16.3
Minus 350-mesh	77.7	23.79	81.2

Referring to Table II, if nearly complete liberation of the manganese minerals in the minus 350-mesh material is assumed, a total extraction of over 90 per cent should be possible from the standpoint of liberation of the manganese minerals. Assuming, also that a separation of the manganese minerals from the gangue on the minus 350-mesh material will be as satisfactory as that observed on the plus 350-mesh, a concentrate grade of over 40 per cent of manganese appears possible.

Before proceeding with flotation tests, the grinding technique was standardized for tests made in the 100-gram capacity flotation cell. The ore, at minus 20-mesh, was wet ground as described in Chapter II, using a two to one ratio of solids to liquid. The time of grinding was varied until, at a time of 15 minutes, the screen analysis given in Table III was obtained and accepted as satisfactory for flotation testing. All subsequent tests on this ore, unless otherwise specified, were made using this grind.

Table III - Screen Analysis of Standard Grind

	Weight %	Cumulative %
Plus 100-mesh	2.4	2.4
Minus 100, plus 200	37.7	40.1
Minus 200, plus 350	22.9	63.0
Minus 350-mesh	37.0	37.0
Totals	100.0	

Series I - Effect of pH

The purpose of these tests was to determine the effect of pH on the flotation of both the manganese minerals and the calcite in the ore. The pH was varied using sulphuric acid or sodium carbonate and soap as the promotor. Ivory soap was selected as a pure and easily reproducible product. The soap was added in lots of 0.1 pound per ton of ore, and the addition continued until manganese mineral was not visible in the froth, the quantities varying from 1 to 5 pounds. DuPont frother B48 was used in Test 1, but a frother was not needed in the succeeding tests. A summary of the results of tests 1 to 5, inclusive, appears in Table IV.

Table IV - Effect of pH Variation (a)

Test No.	pH	Manganese in concentrate, %	Manganese extracted, %	Calcite extracted, %
1	6.7	23.5	57.1	94.4
2	7.6	25.9	59.6	96.5
3	8.2	22.9	48.0	91.0
4	9.1	24.1	50.7	90.7
5	10.2	20.1	32.8	82.6

(a) Heads contained 22.91 per cent of manganese

Interpretation of Results of Tests 1 to 5, inclusive

Referring to Table IV, the results of Tests 1 to 5, inclusive, indicate the following conclusions:

1. A pH of 7.6 (the pH of the ore in distilled water) is most satisfactory for the flotation of both the manganese minerals and calcite when soap is used as the promoter.

2. It is evident that calcite floats more readily than the manganese minerals under these conditions. These results confirm the observations of Gaudin and Behrens.[7]

Series II - DeVaney-Clemmer Procedure

Series II consists of tests based on the DeVaney-Clemmer patent [4] which involves the flotation of the manganese minerals as a high pH using a fatty acid as the promoter and sodium carbonate and sodium silicate to give the desired alkalinity and dispersion. The quantities of sodium carbonate and sodium meta silicate (2.5 pounds of the former and 10.0 pounds of the latter per ton of ore) were determined by dispersion tests, as described in Chapter II. These reagents were added in the grinding stage, but it was found necessary to add the fatty acid in the flotation cell to prevent excessive frothing during grinding. In each case, the pulp was conditioned for 5 minutes with the promoter, the froth collected for 20 to 25 minutes, and the tests stopped when manganese mineral was no longer visible in the froth. The addition of a frother was not necessary.

The first of the tests followed the patent disclosure in using 6.0 pounds of oleic acid per ton of ore. The results of Test 6 are given in Table V.

The second test was made using 6.0 pounds of fish oil fatty acids per ton of ore, as recommended by Weinig[16,17] as promoters of the manganese minerals. The results of Test 7 appear in Table V.

As the patentees suggested a high pulp density for improved extraction in rougher flotation, a third test was made using 200 grams of ore in the 100-gram capacity flotation cell, giving a dilution of about 50 per cent of solids. Dispersion tests had indicated that the concentrations of sodium carbonate and sodium silicate in the solution were the governing factors, rather than the quantities per ton of ore, so, 1.25 pounds of sodium carbonate and 5.0 pounds of sodium silicate per ton of ore were used. Oleic acid was added at the rate of 6.0 per pounds per ton of ore. The results of Test 8 appear in Table V.

Table V - DeVaney-Clemmer Procedure (a)

Test No.		Manganese extracted	Manganese in concentrate,%
6	Oleic acid	85.0	26.2
7	Fish oil fatty acids	71.6	27.2
8	High pulp density	86.0	24.4

(a) Heads containing 22.91 per cent of manganese.

Interpretation of Results of Tests 6 to 8, inclusive.

Referring to Table V, the results of Tests 6 to 8, inclusive, the conclusions which follow are indicated.

1. Both extraction and the grade of concentrate obtained show an improvement over those obtained in the tests of Series I.
2. The fish oil fatty acids appear to be a more selective promoter than oleic acid, although not as powerful.
3. A high pulp density does not appear to be desirable, although this effect may be due to slightly poorer dispersion as a result of the decrease in the quantities (per ton of ore) of sodium carbonate and sodium silicate used in Test 8.

Series III - Oleic Acid with Kerosene

Both Weinig [16, 17] and Gaudin and Behrens [7] mention the use of hydrocarbon oils to assist the flotation of the manganese oxides. Series III consists of tests made to determine the effect of kerosene, when used in conjunction with oleic acid, upon the flotation of manganese minerals. In each of these tests, 3.0 pounds of oleic acid per ton of ore were used, with variations in the amounts and order of addition of the kerosene. The flotation time varied from 7 to 11 minutes, the tests being stopped when no manganese mineral was visible in the froth. As a frother, 0.3

pound of pine oil per ton of ore was used.

The first test was made using oleic acid and conditioning was done in the flotation cell. The results of Test 9 appear in Table VI.

In the second test 2.0 pounds of kerosene per ton of ore were added in the Abbé mill and the oleic acid added during the conditioning period in the flotation cell. The results of Test 10 appear in Table VI.

For the third test the order of addition of the kerosene and oleic acid was reversed, the quantities remaining the same. The results of Test 11 appear in Table VI.

In the fourth test, the ore was conditioned in the flotation cell with the oleic acid, and 6.0 pounds of kerosene per ton of ore. The results of Test 12 appear in Table VI.

Table VI - Oleic Acid with Kerosene (a)

Test No.		Manganese extracted, %	Manganese in concentrate, %
9	Oleic acid only	38.6	22.5
10	Kerosene in grind	36.5	22.0
11	Oleic acid in grind	51.8	23.5
12	Six pounds of Kerosene	35.4	22.1

(a) Heads containing 22.91 per cent of manganese.

Interpretation of Results of Tests 9 to 12, inclusive.

Referring to Table VI, the results of Tests 9 to 12, inclusive, the conclusions which follow are indicated.

1. It appears that it is detrimental to both the extraction of manganese and the grade of the concentrate to add kerosene to the grinding circuit.
2. Large amounts of kereosene are of little value.
3. The results of Test 11 are of interest in that both the grade of concentrate and the extraction were benefitted by the addition of the oleic acid to the grinding circuit.
4. The poor extractions observed in these tests are probably due to the restriction in the quantity of oleic acid used.

Series IV - Effect of Sulphur Dioxide

F. S. Wartman[14] suggested that sulphur dioxide might be useful in depressing silica and activating the manganese minerals. Series IV consists of tests made using this reagent in conjunction with oleic acid, sodium carbonate, and sodium silicate.

The first of these tests was made for the purpose of establishing a basis for comparison, and 6.0 pounds of oleic acid per ton of ore were used without sulphur dioxide. The oleic acid was added in the flotation cell and the froth collected for a period of 15 minutes.

The results of Test 13 appear in Table VII.

The second test was made using 6.0 pounds of oleic acid and 6.0 pounds of sulphur dioxide per ton of ore, the latter being added in an aqueous solution. The ore was conditioned first, with the sulphur dioxide, and second, with the oleic acid, and the froth collected for 20 minutes. The water added during the test contained the same concentration of sulphur dioxide as that in the flotation cell. The results of Test 14 appear in Table VII.

The third test was made using 10.0 pounds of sodium meta silicate, 12.0 pounds of sulphur dioxide, and 12.0 pounds of oleic acid per ton of ore. The pulp was conditioned first, with the sulphur dioxide which was followed by the sodium silicate and oleic acid. The froth was collected over a period of 30 minutes. The results of Test 15 appear in Table VII.

A fourth test was made using the DeVaney-Clemmer (4) procedure with sulphur dioxide. The pulp was conditioned with 3.0 pounds of sulphur dioxide per ton of ore, following which, the pH value was increased to 7.5 with sodium hydroxide. Sodium carbonate, sodium silicate and oleic acid were added in the same proportions as in Test 6 of Series II, and the froth collected over a period of 20 minutes. The results of Test 16 appear in Table VII.

A fifth test was made which was similar to Test 16 in all respects, except that 6.0 pounds of sulphur dioxide per ton of ore were used, and 7.0 pounds of oleic acid per ton of ore were needed. The results of Test 17 appear in Table VII.

A sixth test was made using 12.0 pounds of sulphur dioxide and 8.0 pounds of oleic acid per ton of ore, but in other respects the same as Tests 16 and 17. The results of Test 18 appear in Table VII.

Table VII - Effect of Sulphur Dioxide (a)

Test No.		Manganese in concentrate %	Manganese extracted %
13	Oleic acid alone	23.3	76.7
14	Sulphur dioxide added	25.3	92.0
15	Sulphur dioxide and sodium silicate added	26.4	83.2
16	DeVaney-Clemmer with 3 lbs. sulphur dioxide	27.0	83.2
17	DeVaney-Clemmer with 6 lbs. sulphur dioxide	27.9	83.3
18	DeVaney-Clemmer with 12 lbs. sulphur dioxide	28.2	78.1

(a) Heads containing 22.91 per cent of manganese.

Interpretation of Results of Tests 13 to 18, inclusive.

Referring to Table VII, Tests 13 to 18, the conclusions which follow are indicated.

1. A comparison of the results of Test 6 which appears in Table V with those of Tests 13 to 18 shows that sulphur dioxide improved the grade of concentrate obtained in every case.
2. Large amounts of sulphur dioxide apparently increase the amount of oleic acid necessary

and decrease the extraction.

3. When used in moderate amounts, sulphur dioxide seems to improve both the grade of the concentrate and the extraction.
4. When sodium silicate is used with sulphur dioxide at a relatively low pH value, the grade of concentrate is improved at the expense of increased consumption of promoter.

Series V - Separation of Calcite

To investigate the possibility of a separation of the calcite from the manganese minerals as well as the latter from the silica, a test was made in which the promoter was added in small portions and a separate concentrate collected after each addition. The reagents and their order of addition, with the exception of the promoter, were the same as those in Test 17 which was described in Series IV. To obtain better reagent-mineral contact and eliminate the possibility of the oily oleic acid being carried off in the froth instead of assisting the flotation of the manganese minerals and calcite, a colloidal dispersion of 10 parts oleic acid, one part triethanolamine, and 2000 parts water was used as the promoter in accordance with the Weed[15] disclosures. The promoter was added in the quantities indicated in Test 19 of Table VIII, and the froth was collected after each addition until manganese mineral was not visible in it. The results of Test 19 appear in tabulated form in

Table VIII - Separation of Calcite

	Oleic Acid lbs per ton	Weight %	Manganese %	Calcite %	Insoluble %	% Extraction Cumulative Manganese %	Calcite %	Insoluble %
Head		100.0	22.81	14.9	41.5	100.0	100.0	100.0
Concentrate 1	0.2	9.8	14.31	55.0	15.4	6.1	35.6	3.5
Concentrate 2	0.4	7.2	20.41	37.0	23.8	12.6	52.4	7.6
Concentrate 3	0.6	4.6	25.20	22.0	31.3	17.8	59.1	11.1
Concentrate 4	0.8	3.7	27.38	16.3	33.7	22.3	63.1	14.1
Concentrate 5	1.0	2.6	28.34	13.0	34.7	25.6	65.1	16.3
Concentrate 6	1.5	4.1	24.35	20.5	30.2	30.1	70.5	19.4
Concentrate 7	2.0	3.4	23.90	19.6	31.6	33.7	75.2	22.0
Concentrate 8	3.0	6.3	22.30	20.3	30.1	39.8	83.9	26.4
Concentrate 9	5.0	9.3	27.53	17.5	32.7	51.1	94.6	33.6
Concentrate 10	7.0	10.4	35.02	3.0	35.4	67.1	96.6	42.3
Concentrate 11	10.0	30.6	22.75	1.5	58.4	97.3	99.3	84.3
Tailing		8.0	7.54	1.7	83.5	2.7	0.7	15.7

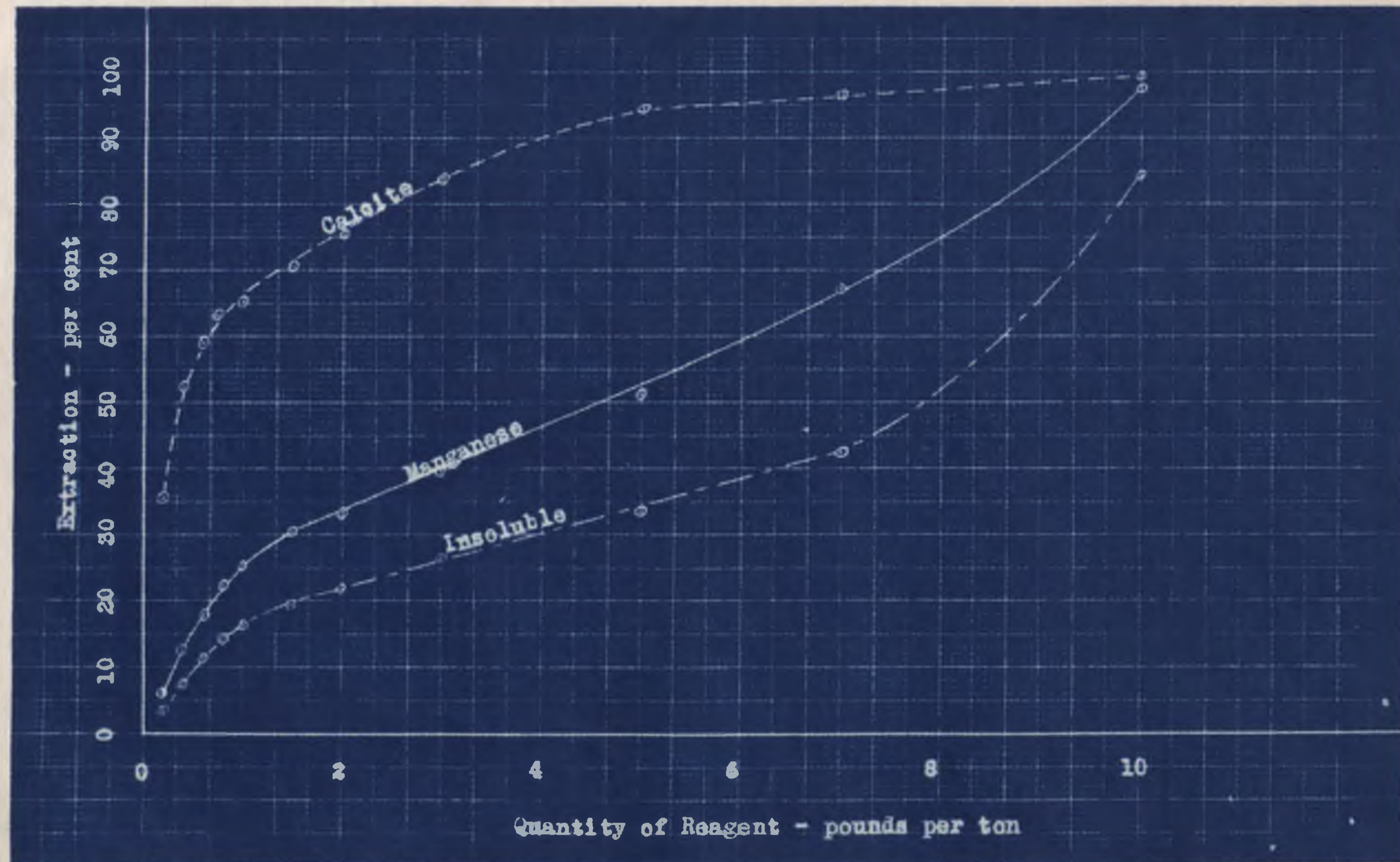
Extraction - per cent
0
10
20
30
40
50
60
70
80
90
100
Calcite
Manganese
Insoluble
0
2
4
6
8
10
Quantity of Reagent - pounds per ton

Figure 1.

Table VIII and in graphical form in Figure 1.

Interpretation of Results of Test 19.

Referring to Table VIII and Figure 1, the conclusions which follow are indicated.

1. The calcite is more floatable than the manganese minerals, but the possibility of a satisfactory separation of the two by this method appears unlikely.
2. The separation of the manganese minerals from the silica is also unsatisfactory, and large amounts of promoter apparently cause even less selectivity. This latter effect may be due to the decreased concentrations of dispersing agents toward the end of the long test in a small flotation cell.

Series VI - Effect of Sherwood Reagent 407

A sample of a reagent made by the Sherwood Petroleum Company and termed "Mahogany Oil" or Sherwood 407, gave encouraging results when investigated by means of tube tests as described in Chapter II. The composition of the reagent was not disclosed by the manufacturer. The tube test results may be summarized as follows: first, sulphur dioxide is necessary to obtain the desired selection and is more effective if added between two additions of promoter; second, both kerosene and oleic acid seem to enhance the selective action of Sherwood 407..

In the first flotation test of this series, the pulp was conditioned in three stages; first, with 5.0 pounds of Sherwood 407, 3.25 pounds of kerosene, and 3.75 pounds of oleic acid; second, with 23.1 pounds of sulphur dioxide; and, third, with 9.0 pounds of Sherwood 407 and 1.25 pounds of kerosene per ton of ore. A frother was not needed. The results of Test 20 appear in Table IX.

A second test was made eliminating the sulphur dioxide, but keeping the other reagents and their quantities constant. The results of Test 21 appear in Table IX.

A third test was made at a pulp density of nearly 50 per cent of solids, using a 200-gram charge in the 100-gram capacity flotation cell, in an effort to reduce the amounts of reagents required. The pulp was conditioned in three stages, using first, 4.0 pounds of Sherwood 407, 1.3 pounds of kerosene, and 1.5 pounds of oleic acid; second, 16.5 pounds of sulphur dioxide, and third, 8.0 pounds of Sherwood 407 and 3.3 pounds of kerosene per ton of ore. The results of Test 22 appear in Table IX.

A fourth test was made, also at a high pulp density, with the oleic acid omitted. The three-stage conditioning procedure as described for Tests 20 to 22 was followed and the total amounts of reagents were 9.5 pounds of Sherwood 407, 6.9 pounds of kerosene, and 6.6 pounds of sulphur dioxide per ton of ore. The results of Test 23 appear in Table IX.

Table IX - Effect of Sherwood 407 [(a)]

Test No.		Manganese in concentrate,%	Manganese extracted,%
20	Initial test	28.0	97.4
21	Sulphur dioxide omitted	26.2	83.7
22	High pulp density	28.8	84.4
23	Oleic acid omitted	28.9	93.4

(a) Heads containing 22.91 per cent of manganese.

Interpretation of Results of Tests 20 to 23, inclusive.

Referring to Table IX, Tests 20 to 23, the conclusions which follow are indicated.

1. It is evident that the Sherwood 407 reagent combination was more effective from the standpoint of both grade of concentrate and extraction, than the best results obtained heretofore.
2. Sulphur dioxide is an essential constituent of the reagent combination, but oleic acid does not appear to be necessary.
3. A high pulp density in the flotation cell results in lower reagent consumption with

little decrease in extraction and an actual increase in the grade of concentrate obtained.

Series VII - Miscellaneous Tests

Several tests were made to determine the value of a miscellaneous group of reagents and reagent combinations that gave inconclusive qualitative results when tested by means of tube tests as described in Chapter II.

The first of these tests was made following the Weed[15] patent procedure as closely as possible. As a pulp density of 33 per cent of solids was specified for flotation, a 150-gram charge of ore was ground with 1.2 pounds per ton of ore (in terms of oleic acid) of the following reagent emulsion:

	mg. per cc.
Oleic acid	500
Kerosene	30
Triethanolamine	5

A total of 8.4 pounds per ton of ore of the reagent were required and the froth was collected over a period of 35 minutes, using 0.3 pound of pine oil per ton of ore. The results of Test 24 appear in Table X.

The second of these tests was made using alkaline potato starch, which F. S. Wartman[14] had heard was a promoter of the manganese minerals. The pulp was conditioned with 0.1 pound of the reagent, and a total of 1.0 pound per ton of ore was used. The last 0.3 pound addition was of no apparent value in floating the manganese minerals as the froth was white. The results of Test 25 appear in Table X.

Again on F. S. Wartman's[14] suggestion, a reagent combination consisting of an emulsion of 1 per cent of Emulsol Corporation reagent X_1 and 6 per cent of kerosene was used. The pulp was conditioned with 0.1 pound of the reagent and floated using a total of 1.0 pound of the reagent per ton of ore. The last 0.4 pound of the reagent appeared to collect little manganese mineral. The results of Test 26 appear in Table X.

Table X - Miscellaneous Tests[(a)]

Test No.		Manganese in concentrate, %	Manganese extraction, %
24	Weed patent method	24.9	91.0
25	Alkaline starch promoter	24.0	40.5
26	Emulsol X_1 with kerosene	23.8	42.8

(a) Heads containing 22.91 per cent of manganese.

Interpretation of Results of Tests 24 to 26, inclusive.

Referring to Table X, Tests 24 to 26, inclusive, the conclusions which follow are indicated.

1. The results of Test 24 indicate that the grade of concentrate obtained, when following the Weed patent disclosures, is not as satisfactory

as that obtained when the DeVaney-Clemmer procedure is used, and a greater consumption of promoter is experienced.

2. Alkaline potato starch is not a satisfactory promoter of the manganese minerals as employed in Test 25.

3. An emulsion of kerosene and Emulsol X_1 is not effective as a promoter of the manganese minerals present in this ore as evidenced by Test 26.

Tests on Samples from the Artillery Peak District

A heavy liquid separation was made on a sample of ore from the Artillery Peak district to determine the degree of liberation of the manganese minerals after grinding to a size of minus 200-mesh. The sample was ground to minus 200-mesh, the test made on the plus 350-mesh portion, and the products analyzed for manganese and insoluble. The results of the test appear in Table XI.

Table XI - Heavy Liquid Separation

	Weight %	Manganese %	Insoluble %	% of total Manganese
Head	100.0	12.93	67.9	100.0
Light Fraction	77.5	4.92	83.0	29.5
Heavy Fraction	22.5	40.49	16.2	70.5

Referring to Table XI, it is evident that a concentrate of low silica content cannot be obtained on the minus 200 plus 350 mesh fraction of this ore. If it is assumed that the minus 350-mesh material would show a similar degree of liberation, an extraction of 70 per cent of the total manganese in a concentrate analyzing 40 per cent of manganese appears possible in so far as liberation is concerned.

Flotation tests were made to compare the effect of a dry ground charge, which had been soaked in water for several hours, with a charge which had been wet ground. For the first test, a sample of the ore, crushed to a size of minus 20-mesh was screened on a 100-mesh sieve and the oversize ground for 10 minutes without water in an Abbé mill. The minus 100-mesh material was again removed, and procedure repeated until the entire lot had been reduced to minus 100-mesh.

For the second test, a 100-gram sample of the ore was wet ground for 15 minutes in the manner described in Chapter II, using a dilution of 66 per cent of solids.

Flotation tests were made in exactly the same manner on both samples. The results of Tests 27 and 28 appear in Table XII.

Table XII - Wet and Dry Grind

Test No.	Manganese in concentrate, %	Manganese extracted, %
27 Wet grind	24.1	75.7
28 Dry grind	23.5	74.6

Referring to Table XII, the results of Tests 27 and 28 were considered sufficiently similar to justify the use of a dry grind on this ore for the comparison of the effects of various reagents upon its flotation. Subsequent tests on this ore were made using a dry grind, unless otherwise noted.

Series I - Oleic Acid-Sulphur Dioxide

A series of tests was made to determine the effects of various quantities of sulphur dioxide, pulp density and fineness of grinding on this ore when using oleic acid as the promoter.

The first test of this group was Test 28, which appears in Table XII. The ore was conditioned in three stages using first, 1.0 pound of oleic acid, second, 6.6 pounds of sulphur dioxide, and third, 2.0 pounds of oleic acid per ton of ore. A total of 5.0 pounds of oleic acid and 0.5 pound of duPont frother B48 was used. The results of Test 28 appear again in Table XIII.

A second test was made in the same manner as Test 28, but 0.66 pound of sulphur dioxide per ton of ore was used. The results of Test 29 appear in Table XIII.

A third test was made in the same manner as Test 29, but the sulphur dioxide concentration was sustained at 0.66 pound per ton of ore and a total of 6.0 pounds of oleic acid was used. The results of Test 30 appear in Table XIII.

A fourth test was made in the same manner as Test 30, but 3.3 pounds of sulphur dioxide were used in the conditioning. The results of Test 31 appear in Table XIII.

In a fifth test a charge of 200 grams of ore, to give a dilution of about 50 per cent of solids, was conditioned first, with 0.5 pound of oleic acid; second, with 0.66 pound of sulphur dioxide; and third, with 1.0 pound of oleic acid per ton of ore. A total of 3.0 pounds of oleic acid, and 0.5 pound of duPont frother B48 per ton of ore was used, and the concentration of sulphur dioxide was sustained at its original value. The results of Test 32 appear in Table XIII.

A sixth test was made in the same manner as Test 32, but 0.33 pound of sulphur dioxide and a total of 5.0 pounds of oleic acid were used per ton of ore. The concentration of sulphur dioxide was sustained at its original value. The results of Test 33 appear in Table XIII.

A seventh test was made in the same manner as Test 30, but the ore was wet ground in an Abbé mill for 30 minutes, to observe the effect of a very fine grind on the flotation of the manganese minerals in this ore. The results of Test 34 appear in Table XIII.

An eighth test was made using a sample of ore which had been dry ground to minus 200-mesh and soaked in water for several hours. A total of 4.5 pounds of oleic acid per ton of ore was used, but the test was the same as Test 32 in other respects. The results of Test 35 appear in Table XIII.

A ninth test was made using 1.0 pound of sulphur dioxide and 5.0 pounds of oleic acid per ton of ore, and sustaining the concentration of the sulphur dioxide throughout, but in other respects the same as Test 35. The results of Test 36 appear in Table XIII.

A tenth test was made using an initial and sustained concentration of 2.0 pounds of sulphur dioxide per ton of ore, but in other respects the same as Test 36. The results of Test 37 appear in Table XIII.

An eleventh test was made using 3.3 pounds of sulphur dioxide per ton of ore in the conditioning, and not allowing its concentration to drop below 0.66 pound per ton of ore, but in other respects the same as Test 37. The results of Test 38 appear in Table XIII.

Table XIII - Oleic acid, - Sulphur Dioxide [a]

Test No.	Grind	Pulp Density	Sulphur Dioxide Lbs. per ton	Oleic Acid Lbs. per ton	Manganese in Concentrate, %	Manganese Extracted,%
28	(b)	(e)	6.6	5.0	23.5	74.6
29	(b)	(e)	0.66	5.0	19.9	85.4
30	(b)	(e)	0.66 (h)	6.0	23.7	77.8
31	(b)	(e)	3.3 (h)	6.0	26.7	78.7
32	(b)	(f)	0.66 (h)	3.0	21.1	75.8
33	(b)b	(f)	0.33 (g)	5.0	23.0	78.5
34	(c)	(e)	0.66 (g)	6.0	24.5	84.8
35	(d)	(f)	0.66 (g)	4.5	19.4	96.3
36	(d)	(f)	1.0 (i)	5.0	20.6	95.5
37	(d)	(f)	2.0 (j)	5.0	23.4	92.4
38	(d)	(f)	3.3 (h)	5.0	21.5	94.0

(a) Heads contained 12.90 per cent of manganese.
(b) Dry grind to minus 100-mesh.
(c) Fine, wet grind.
(d) Dry grind to minus 200-mesh.
(e) Normal pulp density (about 25 per cent of solids)
(f) High pulp density (about 50 per cent of solids)
(g) SO_2 concentration sustained at 0.33 lbs. per ton of ore.
(h) SO_2 concentration sustained at 0.66 lbs. per ton of ore.
(i) SO_2 concentration sustained at 1.0 lb. per ton of ore.
(j) SO_2 concentration sustained at 2.0 lbs. per ton of ore.

Interpretation of Results of Tests 28 to 38, inclusive.

Referring to Table XIII, Tests 28 to 38, inclusive, the conclusions which follow are indicated.

1. The presence of sulphur dioxide reduces the extraction of the manganese minerals, but improves the grade of the concentrate obtained as indicated by the results of Tests 28 and 29.
2. When smaller amounts of sulphur dioxide are added continuously, there is a slight improvement in extraction as evidenced by the results of Tests 28 and 30.
3. A higher initial concentration of sulphur dioxide with a lower sustained concentration improves the grade of concentrate obtained as shown by the results of Tests 30 and 31.
4. A high pulp density results in a lower reagent consumption as indicated by the results of Tests 32 and 33.
5. A fine grind improves both the grade of concentrate obtained and the extraction of the manganese minerals as shown by the results of Test 34.
6. Using a high pulp density and a fine grind, an increase in the amount of sulphur dioxide employed results in an increase in the grade of concentrate obtained as evidenced by Tests 35 to 38, inclusive.

Series II - Oleic Acid-Kerosene Emulsion

A series of tests was made to determine first, the effect of kerosene when used in conjunction with oleic acid in an emulsion stabilized with Sherwood 407, and second, the value of the three-stage conditioning procedure used when sulphur dioxide was employed. A total of 2.0 pounds of the emulsion per ton of ore (in terms of oleic acid), was used. The emulsion consisted of 100-mg per cc. of oleic acid with 2-mg per cc. of Sherwood 407, and varying amounts of kerosene. Each test was made on a charge of 200 grams of minus 200-mesh dry ground and water soaked ore, with 0.5 pounds per ton of duPont frother B48. Tap water, with the pH value reduced to that of distilled water by means of sulphuric acid, was used in these tests.

The first test was made without kerosene, the pulp being conditioned in three stages with first, 0.5 pound of oleic acid; second, 1.0 pound of sulphur dioxide; and third, with 1.5 pounds of oleic acid per ton of ore, and the concentration of sulphur dioxide was sustained at 1.0 pound per ton of ore. The results of Test 39 appear in Table XIV.

The second test was the same as Test 39, but 0.2 pound of kerosene was added, after being emulsified with the other reagents. The results of Test 40 appear in Table XIV.

The third test was the same as Test 40, but 2.0 pounds of kerosene were used. The results of Test 41 appear in Table XIV.

A fourth test was made in the same manner as Tests 40 and 41, but 4.0 pounds of kerosene were used. The results of Test 42 appear in Table XIV.

A fifth test was made without sulphur dioxide. In other respects, the test was the same as Test 40. The results of Test 43 appear in Table XIV.

A sixth test was made in which the three-stage conditioning procedure was abandoned, the initial 1.0 pound of sulphur dioxide per ton of ore being added first, and followed by the emulsified reagent. In other respects, the test was the same as Test 40. The results of Test 44 appear in Table XIV.

A seventh test was made in which the three-stage conditioning procedure was again abandoned, the emulsified reagent being added first and followed by the sulphur dioxide. In other respects, the test was the same as Test 40. The results of Test 45 appear in Table XIV.

Interpretation of Results of Tests 39 to 45, inclusive.

Referring to Table XIV, Tests 39 to 42, inclusive, the conclusions which follow are indicated.

1. Small amounts of kerosene improve the extraction of the manganese minerals, but even large amounts have little effect upon the grade of

concentrate obtained.

2. Sulphur dioxide assists the flotation of the manganese minerals.

3. The three-stage conditioning procedure seems to be essential in order to obtain satisfactory extraction, but the grade of concentrate is not as high as when the sulphur dioxide is added first.

Table XIV - Oleic Acid-Kerosene Emulsion (a)

Test No.		Manganese in Concentrate,%	Manganese extracted,%
39	No kerosene	19.9	85.2
40	0.2 lb. of kerosene	19.9	90.1
41	2.0 lb. of kerosene	20.2	89.8
42	4.0 lb. of kerosene	20.8	89.2
43	No sulphur dioxide	22.3	50.9
44	Sulphur dioxide first	25.0	72.4
45	Promoter first	21.4	76.5

(a) Heads containing 12.90 per cent of manganese.

Series III - DuPont Reagent

The duPont Company recommends the following reagent combination for the flotation of the manganese minerals:

	mg. per cc.
Dupanol L. S. paste	0.53
Rosin	4.00
Sodium hydroxide	5.00
Crude oil	30.00
Rosin oil	4.00

A series of tests was made using this reagent on 200-gram lots of minus 200-mesh dry ground ore which had been soaked for several hours in distilled water. In each case, the ore was conditioned with 40 cc. of the reagent, which was equivalent to 12.0 pounds of crude oil per ton of ore, and 0.5 pound of duPont frother B48 was used. The crude oil used consisted of No. 5 California fuel oil which had been thinned with 10 per cent of No. 1 California fuel oil.

The first test was made using this reagent in the manner described above. The results of Test 46 appear in Table XV.

A second test was made using the reagent in conjunction with sulphur dioxide. The pulp was conditioned in three stages, using first, 10 cc. of the reagent, second, 1.0 pound of sulphur dioxide per ton of ore, and third, 30 cc. of the reagent. The sulphur dioxide concentration was sustained at the initial concentration. The results of Test 47 appear in Table XV.

The reagent emulsion was highly alkaline and Test 48 was made to determine the effect on flotation of lowering its pH. Sulphuric acid was added to the reagent, reducing its pH value to about 7.0. The test was made using the neutral reagent, but was the same at Test 46 in other respects. The results of Test 48 appear in Table XV.

Table XV - DuPont Reagent (a)

Test No.		Manganese in concentrate, %	Manganese extracted, %
46	Reagent as specified	22.3	82.6
47	Sulphur dioxide added	18.9	85.7
48	Neutral reagent	20.4	72.7

(a) Heads containing 12.90 per cent of manganese.

Interpretation of Results of Tests 46 to 48, inclusive.

Referring to Table XV, Tests 46 to 48, inclusive, the conclusions which follow are indicated.

1. The duPont reagent gives a satisfactory grade of concentrate, but the extraction is not as high as can be obtained with a dispersion of oleic acid and kerosene.
2. Sulphur dioxide decreases the selectivity of this reagent in respect to the manganese minerals. This effect may be due, in part, to the acidity of the aqueous solution of sulphur dioxide.
3. Neutralizing the reagent with sulphuric acid decreases its usefulness as a promoter for the manganese minerals.

Series IV - Cleaner Flotation

Several tests were made in an effort to determine the most satisfactory procedure for the cleaner flotation of rougher concentrates recovered from this ore. In order to have the same rougher concentrate for use in these tests, rougher flotation tests were made in a 500-gram capacity Denver laboratory flotation cell on samples which were wet ground and wet screened to give a minus 180-mesh product. The oleic acid-sulphur dioxide procedure described in Test 36 of Series I was used and concentrates having an average content of 21.96 per cent of manganese and representing 86.6 per cent of the total manganese in the ore, were obtained. The moisture content of the combined concentrates was determined and a sample of the wet material equivalent to a 100-gram dry weight was used for each test. Two stages of cleaner flotation, 0.5 pound of duPont frother B48 per ton of feed being added in the first, were used for each test appearing in Table XVI.

The first test of this series was made with the addition of the frother only. The results of Test 49 appear in Table XVI.

The second test of this series was made using an initial concentration of 3.0 pounds and a sustained concentration of 1.0 pound of sulphur dioxide per ton of feed for each stage of cleaner flotation. The results of Test 50 appear in Table XVI.

In the third test, the pulp was conditioned in three stages with, first, 1.0 pound of oleic acid, second, 6.0 pounds of sulphur dioxide, and third, 1.0 pound of oleic acid, per ton of feed. A total of 5.0 pounds of oleic acid per ton of feed was used in the first cleaner float and the concentration of sulphur dioxide was sustained at 2.0 pounds per ton of feed. The same concentrations of sulphur dioxide were used in the second stage of cleaner flotation, but oleic acid was not added. The results of Test 51 appear in Table XVI.

For the fourth test, a sample of the rougher concentrate was ground for 15 minutes in a 100-gram capacity Abbé mill and the flotation test performed in the same manner as Test 51. The results of the first two stages of cleaner flotation appear as Test 52A in Table XVI. In this case the cleaning operation was repeated using an initial concentration of 6.0 pounds and a sustained concentration of 2.0 pounds of sulphur dioxide per ton of feed in each stage. In all, four stages of cleaner flotation were used. The results appear as Test 52B in Table XVI.

A fifth test was made using tannin to improve the grade of concentrate as disclosed by Weinig[17]. The pulp was conditioned with 0.25 pound of tannin per ton of feed before each of the two stages of cleaner flotation. The results of Test 53 appear in Table XVI.

<u>Interpretation of Results of Tests 49 to 53, inclusive.</u>

Referring to Table XVI, Tests 49 to 53, inclusive, the conclusions which follow are indicated.

1. Sulphur dioxide is of little benefit in the cleaner flotation of the rougher concentrate tested.
2. Regrinding the rougher concentrate before proceeding with cleaner flotation improves the grade of concentrate obtained with only a small sacrifice in extraction.
3. As employed in Test 53, tannin does not greatly improve the grade of concentrate obtained, but does cause a decrease in extraction.

Table XVI - Cleaner Flotation (a)

Test No.		Manganese in concentrate, %	Manganese (b) extracted, %
49	Frother only	32.3	85.1
50	Sulphur dioxide added	32.8	72.9
51	Additional oleic acid with sulphur dioxide.	32.6	84.7
52A	Regrind	35.2	82.4
53	Tannin	33.2	55.4

(a) Tests made on rougher concentrate analyzing 21.96 per cent of manganese and containing 86.6 per cent of the manganese in the ore.
(b) Per cent extraction in cleaner flotation.

Test 52B - Four Stages of Cleaning

	Weight, %	Manganese, %	% of Total Manganese
Head	100.0	21.26	100.0
Concentrate	38.5	38.6	70.5
Middling	61.5	10.3	29.5

Tests on Ore from the Cecil Martin Claims

Flotation tests were made on samples containing 11.3 per cent of manganese from the Cecil Martin Claims near Sentinel, Arizona. The predominant gangue mineral in this ore was calcite and as the experimental work on samples from the Bender Mine had indicated that calcite was more readily floated than the manganese minerals when oleic acid was used as the promoter, an attempt was made to float the calcite away from the manganese minerals which would become concentrated in the tailing.

Series I - Inverted Flotation

A series of tests was made using 100-gram samples of the ore which had been wet ground for 15 minutes in an Abbé mill at a dilution of 66 per cent of solids.

In the first test the pulp was conditioned with 2.0 pounds of oleic acid per ton of ore and the calcite floated using 0.2 pounds of duPont frother B48 per ton of ore. The results of Test 53 appear in Table XVII.

A second test was made using sulphur dioxide to determine whether a high concentration of this reagent would act as a depressant for the manganese minerals, when the quantity of promoter was restricted. The pulp was conditioned first, with 2.0 pounds of oleic acid, and second, with 10.0 pounds of sulphur dioxide per

per ton of ore. The concentration of sulphur dioxide was sustained at 5.0 pounds per ton of ore and 0.2 pounds of duPont frother B48 was used per ton of ore. The results of Test 54 appear in Table XVII.

F. S. Wartman (14) suggested the use of sodium hexametaphosphate, which is sold under the trade name of Calgon, as a possible depressant for calcite. A test was made using this reagent, the pulp being conditioned first, with 0.5 pounds of Calgon and second, with 1.0 pound of oleic acid per ton of ore. A total of 4.5 pounds of oleic acid and 0.5 pound of duPont frother B48 per ton of ore was used in the test. The results of Test 55 appear in Table XVII.

Tube tests, made as described in Chapter II, had indicated that duPont reagent M. P. 189, which is an aliphatic sulphonate, would float calcite, but not manganese minerals. A test was made using this reagent, the pulp being conditioned with 0.1 pound of M. P. 189 per ton of ore. A total of 1.2 pounds of the reagent were used per ton of ore. A frother was not needed. The results of Test 56 appear in Table XVII.

Interpretation of Results of Tests 53 to 56, inclusive.

Referring to Table XVII, Tests 53 to 56, inclusive, the conclusions which follow are indicated.

1. Oleic acid is the most effective of the reagents and reagent combinations used.
2. Activation of the manganese minerals by sulphur dioxide is indicated by the results of Test 54.
3. Calgon is a more effective depressant of the manganese minerals than of calcite.
4. DuPont reagent M.P. 189 has a tendency, not observed in tube tests, to float a part of the manganese minerals.

Table XVII - Inverted Flotation (a)

Test No.		Manganese in concentrates, %	Manganese extracted, %
53	Oleic Acid	19.4	81.7
54	Oleic Acid with Sulphur dioxide	14.3	76.9
55	Oleic Acid with Calgon	22.6	50.7
56	DuPont M.P. 189	19.2	76.9

(a) Flotation Tests on ore containing 11.3 per cent of Manganese.

Series II - Inverted Flotation of Wilfley Table Concentrates

Tests were made on the concentrate from a Wilfley table test on this ore. The table concentrate assayed 31.0 per cent of manganese and contained 41 per cent of the total manganese in the ore. The flotation tests were made using samples of the concentrate which had been ground in the manner described in Series I.

In the first test, the sample was conditioned with 1.0 pound of duPont reagent M.P. 189 per ton of feed and the calcite floated. The results of Test 57 appear in Table XVII.

For the second test 5.0 pounds of Calgon and 11.0 pounds of oleic acid per ton of feed were used. The results of Test 58 appear in Table XVIII.

Table XVIII - Tests on Wilfley Table Concentrate[(a)]

Test No.	Manganese in concentrate, %	Manganese Extraction, %
57 DuPont M.P. 189	37.5	87.2
58 Oleic acid and Calgon	41.7	85.5

(a) Flotation tests on concentrates containing 31.0 per cent of manganese.

Interpretation of Results of Tests 57 and 58

Referring to Table XVIII, Tests 57 and 58, the conclusions which follow are indicated.

1. A satisfactory separation of the manganese minerals and the calcite can be obtained on the table concentrate.
2. The consumption of oleic acid is excessive when the quantity of Calgon used in Test 58 is employed.
3. The contrast in the results of Test 57 with those of Test 56 which appear in Table XVII may be due to the absence of the primary slimes of the ore in the former case.

CHAPTER IV - CONCLUSIONS

The results of the experimental work on the flotation of manganese minerals indicate that different types of ores may require very different methods of treatment. Despite this fact, the experimental work indicates that the factors which follow had a similar effect on all ores tested and appear to justify the conclusions given in so far as those ores are concerned.

1. Oleic acid is a powerful promoter of the manganese minerals and calcite.
2. Sulphur dioxide improves the separation of the manganese minerals from siliceous gangue minerals and is most effective when added during the conditioning period in the flotation cell between two additions of promoter.
3. A high pulp density in the flotation cell decreases the consumption of reagents without causing an excessive decrease in the grade of concentrate obtained.
4. Calcite is more readily floated than the manganese minerals.

The results of tests made on samples of ore from the Bender Mine, the most difficult type of ore tested, indicate the conclusions which follow.

5. A reagent combination consisting of kerosene and Sherwood 407 when used with sulphur dioxide gave the most satisfactory results.

6. A Procedure based on the DeVaney - Clemmer[4] disclosures yielded moderately good results, but the reagent consumption was high.

7. A satisfactory separation of the calcite from the manganese minerals was not obtained when oleic acid was used as the promoter.

8. The pH value of the ore in distilled water was satisfactory for the flotation of the manganese minerals with soap.

9. Fish oil fatty acids, alkaline potato starch, Emulsol X_1-kerosene emulsion, and soap are not as powerful as promoters of the manganese minerals as oleic acid.

The results of tests made on samples of ore from the Artillery Peak district indicate the conclusions which follow.

10. A fine grind is essential for satisfactory recovery.

11. Oleic acid when used in conjunction with sulphur dioxide is most effective particularly when used in an emulsion stabilized with Sherwood 407.

12. Kerosene, when added in an emulsion, improves the extraction to a slight extent, but does not effect the grade of concentrate obtained.

13. Grinding of the rougher concentrate before proceeding with cleaner flotation is beneficial.

14. Neither sulphur dioxide nor tannin are effective in raising the grade of the cleaner concentrate when added to the cleaner flotation circuit.

15. The reagent combination recommended by the Du-Pont Company is effective, but the quantity necessary to obtain satisfactory extraction is excessive.

The results of the tests made on samples of ore from the Cecil Martin claims indicate the conclusions which follow.

16. Selective flotation separation of the calcite from the manganese minerals was not satisfactory on the undeslimed ore, but oleic acid was the most effective of the reagents tested in this respect.

17. A satisfactory separation of the calcite from the manganese minerals of a Wilfley table concentrate is possible.

BIBLIOGRAPHY

1. Blessing, Lee R. ...... Experimental Work on Manganese Silver Ores. Thesis, University of Arizona. 1936

2. Coghill, W. H. and Clemmer, J. B...... Soap Flotation of the Nonsulphides. American Institute of Mining and Metallurgical Engineers. Technical Publication 445. 1932

3. Dean, R. S., DeVaney, F. C., and Coghill, W. H............... Manganese: Its Occurrence, Milling Metallurgy. Part I, Chapter IV, United States Bureau of Mines Information Circular No. 6768, May,1934.

4. DeVaney, F. D. and Clemmer, J. B.... Process for Recovering Manganese from Ore. United States patent No. 1,951, 326. March 13, 1934.

5. DeVaney, F. D. and Clemmer, J. B.... Floating of Carbonate and Oxide Manganese Ores. Engineering and Mining Journal, September, 28, 1929.

6. Gaudin, A. M........... Flotation. First Edition, McGraw-Hill Book Co., 1932.

7. Gaudin, A. M. and Behrens, R. H....... A Manganese Recovery Problem and Its Solution. Engineering and Mining Journal, November, 1937.

8. Gutzeit, Gregoire...... Process for Concentrating Oxidized Ores by Means of Froth Flotation. United States patent No. 2, 125, 631. August 2, 1938.

9. Low, A. H............. Technical Methods of Ore Analysis, Fifth Edition, John Wiley and Sons, Inc.1914.

10. Norcross, F. S. ... Development of the Low-grade Manganese Ores of Cuba. American Institute of Mining and Metallurgical Engineers. Technical Publication No. 1188. 1940.

11. Ralston O. C. ..... Flotation and Agglomerate Concentration of Nonmetallic Minerals. United States Bureau of Mines. Report of Investigations No. 3397. May, 1938.

12. Short, M. N. ...... Microscopic Determination of the Ore Minerals. Part III, Determinative Table. United States Geological Survey, Bulletin No. 825. Revision by M. N. Short, September, 1934.

13. Wark, I. W. .... Principles of Flotation. Australasian Institute of Mining and Metallurgy. 1938.

14. Wartman, F. S. .... Personal Communication. F. S. Wartman, Associate Metallurgist Southwestern Experiment Station, United States Bureau of Mines.

15. Weed, Floyd .... Flotation of Manganese Ores. United States patent No. 2,014,407. September 17, 1935.

16. Weinig, A. J. ..... Concentrating Manganese Ores. United States patent No. 1,955,039. April 17, 1934.

17. Weinig, A. J. ..... Concentrating Manganese Ores. United States Patent No. 1,911,855. May 30, 1933.

18. Weinig, A. J. and Carpenter, C.B. . The Trend of Flotation. Colorado School of Mines Quarterly. Vol. XXXII, No. 4. October, 1937.

19. Wilson E. D. and Butler, G. M. ... Manganese Ore Deposits in Arizona. University of Arizona Bulletin No. 127, February 15, 1930.