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CONCENTRATION OF MANGANESE DIOXIDE
FROM PHILIPSBURG, MONTANA

By

W. M. McAuliffe

of

Anaconda, Montana

A Thesis

Submitted to the Department of Mineral
Dressing in partial Fulfillment of the
Requirements for the Degree of Bachelor
of Science in Metallurgy.

MONTANA SCHOOL OF MINES
BUTTE, MONTANA
May 5, 1948

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CONCENTRATION OF MANGANESE DIOXIDE FROM PHILIPSBURG, MONTANA

INTRODUCTION

Manganese is one of the most important strategic raw materials. In the most current list of strategic and critical materials, published by the Army-Navy Munitions Board, (1) manganese, both battery and metallurgical grades are listed in the first group (A-group) and for this group, this statement is made, "those strategic and critical materials for which stock-piling is the only satisfactory means for insuring an adequate supply for the future". The reason for stock-piling is obviously due to the fact that our domestic production is insufficient. Its insufficiency prevails in time of peace, and certainly would be magnified in time of national emergency.

Imports of manganese ore probably supply a major proportion of the needs of the United States. Domestic production is reported to be higher than pre-war levels, but does not equal that of the peak production year of 1943. In 1946, the Anaconda Copper Mining Company accounted for 90 percent of the total shipments of manganese nodules, and this company is the largest producer of domestic metallurgical ore in the United States. By the fact that this company is a major domestic producer of manganese reflects in turn the fact that Montana is a

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(1) Reference in bibliography.

major domestic source of manganese. In addition, Montana has two producers of battery grade domestic manganese. In 1946, all of the battery grade ore produced domestically, came from Montana.

The companies responsible for the production of battery manganese of the American Machine & Metals Inc., and the Taylor-Knapp Company, both in the Philipsburg district, some forty six miles west of Butte.

The Philipsburg district constitutes one of the major (if not the major) source of domestic battery manganese. It is with the beneficiation of this ore that this preliminary investigation is concerned. One of the producing companies, the Taylor-Knapp Company, expressed concern over their present benificiating methods, and suggested that a study of their benificiation operations might result in a more satisfactory method of treating the crude material. This thesis summarizes a study of the present plant, plant changes, and some laboratory investigative work for the beneficiation of battery grade manganese. Additive to the above list of objectives is the consideration of the possibility of producing from one fraction of the ore, a material that upon proper treatment might be suitable for metallurgical uses.

General Survey of Manganese Benificiation

Manganese ore beneficiation has long been a major problem of national importance. Since it is indispensable to the nations steel and battery industries, and since domestic producers have been unable to supply more than a small part of the national needs, it is vitally necessary that processes for the beneficiation of low grade manganese ores be developed. A number of schemes for treatment of the manganese crude have been investigated since 1930, and some of them have been, to a certain degree, successful. The problem encountered have been numerous, and difficult to over come. Some of the problems and methods of overcoming them will be discussed.

Western Manganese Deposits

A large amount of experimentation has been done by the Bureau of Mines on western manganiferous ores. The Bureau of Mines Division at the Missouri School of Mines conducted an investigation concerning the beneficiation of manganese oxide ores from Alabama, Colorado, Idaho, Missouri, and Nevada.(2) From the results of this work it was noted that the Colorado manganese ore was the only one that showed possibilities of becoming economically feasible. This was a pyrolusite ore, on which an attempt was made to producea material suitable for battery use. Gravity concentration methods were used in beneficiation of this material. The crude was up-graded by tabling from a 50 percent manganese dioxide content to one of 68.22 percent.

At that time this product would not meet the specifications of the battery industry, but as these specifica-

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(2) Reference in bibliography

tions have been lowered, the project may again come to life.

An investigation of the possibilities of the concentrating the manganese ores of Gila, Greenlee, and Grahan Counties, Arizona, was carried out by the Bureau of Mines Station at Salt Lake City.(3)

The Gray-claims lot-B ore from Gila County was a hand sorted ore and had a manganese content of 44 percent. With sintering the material was up-graded to a 51.9 manganese, content and was salable as metallurgical manganese.

Davis claim ore from Graham County. The manganese was present as pyrolusite, intimatly associated with iron in the ocherous form. This close association eliminated coarse gravity methods of concentration. Investigations were made into the possibility of beneficiation by : (1) Selective flotation of calcite; (2) tabling sized fractions and (3) Magnetic separation. It was found that none of these methods were successful because the manganese and iron oxides were interlocked to such a **degree** that even very fine grinding would not liberate them.(4)

South Dakota Deposits. For many years manganese deposits have been known to occur in the bluffs of the Missouri River Valley. Various attempts have been made to develop these deposits from time to time, but they have met with little success. Under pressure of the war emergency, when much of our foreign source of manganese was shut off, the Federal Bureau of Mines conducted an investigation into the possibilities of developing these deposits.

(3&4) Reference in bibliography.

An experimental pilot was set up near Chamberlain, South Dakota. This plant had a capacity of 300 tons per 24 hours and was later enlarged to a 1000 ton capacity.

The manganese occurs an concretionary nodules of iron-manganese carbonates, in a host rock of Pierre shale, composed of inter-bedded layers of dentonite and montmorllonite-beidellite clay minerals. In the vicinity of the deposits, the ground is covered with gumbo soil, and the bluffs with weathered nodules. The manganiferous beds average 40 feet in thickness, varying from 26 to 64 feet. Weathering helps to liberate the nodules and condition the shale so that it may be easily dug by modern earth moving equipment. A typical analysis of the nodules shows 18.3 percent manganese and 6.6 percent iron.

Many methods of treating the manganiferous shale were considered and tried, and most of them were found to be unsuccessful commercially. Among the more promising methods were: (1) disintegration of ore by trommel breaker, (2) screening air dried samples, (3) washing raw ores, (4) differential grinding, (5) explosive shattering, and (6) kilm drying, followed by dry screening to recover clean, coarse nodules, and reject fine material, in which the nodules in the intermediaterange were recovered by slaking and washing. Of the six methods, the last was deemed the most promising and was selected as the basis of pilot plant study.

After two years of experimentation with the pilot plant, the investigators concluded that the amount of beneficiation obtained by the kiln, did not justify the cost of its operation.

At that time, the International Minerals & Chemical Corp., in anticipation that the manganese situation would remain critical, took over the experimentation of work at the plant. After three months of investigation of sink-float, jugging, and tabling, International decided that the use of jugs offered the best possibility of concentration.

All tests conducted on this material had the primary objective of producing Iron-manganese nodules from the host shale. Since the nodules show a manganese content of only17 percent, it is doubtful that they would be economically feasible, except as an emergency product.

Cuban Manganese Deposits. Large, low grade deposits of manganese have been known to exist in Cuba since the Spanish-American War, but very little effort was made to develop these deposits until 1930.(5) The development of a process from the beneficiation of the Cuban ore is significant, because it assures the United States of a near by source of manganese ores, and it may be applied to domestic manganese deposits which are similar to the Cuban ores. For this reason some of the plant factors are reviewed at thistime.

The manganese crude of these deposits are a mixture of psilomelene, pyrolusite and other manganese oxides.(6) The crude is characterized by the extreme variability in physical structure, and chemical composition. Various oxides exist in all possible proportions from that of a pure state, to

(5 & 6) Reference in bibliography

that of a complex aggregate. There exists an extremely wide range in particle size from relatively large nodules to a lower limit below minus 600 mesh.

Methods of concentration and processes investigated are: (1) jigging, (2) tabling, (3) flotation and (4) sintering of flotation and table concentrates. Pilot plant studies eliminate tabling, and shows a doubtful value for jigging. Flotation, however, showed satisfactory results, and that method of concentration was chosen as the basis of beneficiation.

Some of the difficulties, encountered in putting the plant on a production basis, and the solutions of these problems are given in the following paragraphs.

Settling. One of the most pronnounced characteristics of manganese oxide minerals is their rapid rate of settling. This property of the mineral caused the operators to abandon the use of thickeners and filters, and to adopt the use of the Esperanza classifier for dewatering. This apparatus works out of a pool, into which all concentrates are fed, and discharges into mechanical storage bins, where water is allowed to drain off the top.

Belts. The flotation plant experience has been that the oil and fatty acid reagents caused belts to curl and disintegrate. This was remedied by the use of drag or screw conveyers.

Flotation and froth-handling. In flotation, difficulty has been encountered in pumping scavenger cell froth from the lower scavenger level, to the upper flotation level. The scavenger cells were placed on the same level as the rougher cells and the difficulty was overcome.

Sliming. Manganese minerals are very friable and tend to produce a large amount of fines. When a ball mill is used, the material runs as high as 70 percent minus 200 mesh. The use of a rod mill in the fine grinding circuit in conjunction with Akins classifiers, cut the slime production appreciably.

Flotation difficulties. Because of varying mineral characteristics, the rougher flotation cells were forced to treat varying feed under fixed conditions. The rod mill grind gave a more uniform control by giving a more uniform product. Better control of the flotation operation was also obtained by use of automatic classifier control, and solution density control.

Reagent Control. Variations in the feed, water, and human errors of judgment causes much wastage of reagents. An automatic reagent and water control system that was synchronized with the ore feed at the head of the mill, was installed.

General Results. These changes in operation of the plant, resulted in an increased effeciency and increased production. This is evidenced by the fact that Cuban manganese constituted less than 1 percent of the United States imports in 1931, and had increased to 20 percent in 1945.

Montana Deposits. The Butte District first attracted attention during 1916 and 1917, when the price of manganese was high and the possibility of large domestic production was first considered. Two types of ore was found in this district. The black oxide ores of the out-crops, and weathered zones were a high grade material, and these were mined in the early days. Below the oxidized zone, the manganese crude occured in the carbonate form, commonly known as pink manganese or rhodochrosite, with smaller amounts of rhodonite. The gangue minerals are quartz, and the small amounts of galena, sphalerite and pyrite. Flotation studies, as a means of concentration, were begun in 1927, and proved amenable to concentrating the manganese crude. The Domestic Manganese & Development plant, at Butte, began operations in 1929 and treated the ore at a rate of 300 tons daily, producing a material that had a 60 percent manganese content, with an over-all recovery of 95 percent. Later the operation of the plant was discontinued.

During the recent war emergency, the Anaconda Copper Mining Company, erected a plant at Anaconda, and since that time has produced 96 percent of the nations domestic metallurgical grade manganese, and 90 percent of the total domestic production. This district is by far the most important manganese producer in the nation.

The mineral crude, as mined, has a manganese content that varies from 12 to 35 percent. It is transported to the manganese plant at Anaconda, located 26 miles northwest of Butte. The ore passes through a crushing and grinding circuit where it is ground to a minus 200 mesh size. The sulphides are recovered by the use of a small amount of xanthate, copper-sulphate and pine oil. The tails from this operation go to the manganese flotation, where the rhodochrosite is floated by addition of sodium oleate, oleic acid and pine oil or similar fatty acid reagents, and rhodonite are rejected tailings. The manganese concentrate contains approximately 40 percent manganese and contains 67 percent of the total manganese of the crude. After filtering, the flotation concentrate is passed through a rotary kiln where; the remaining moisture is driven off, the carbonate calcined to the oxide and the resulting oxide nodulized. This is the finished product and has a manganese content of at least 60 percent.

Philipsburg Manganese Deposits

History: The Philipsburg District, located 46 miles northwest of Butte, is the chief producer, (if not the only producer) of battery grade manganese in this country. Manganese ore was shipped from this district as early as 1900 but substantial production did not began until 1916. Much of the material produced at that time was used as metallurgical and chemical grade manganese. It was found that physical properties of this ore were superior quality to foreign ores,(7) and that even though the available

(7) Reference in bibliography

free oxygen content was not as great as in the foreign ores, the product gave better service.

Minerology. Rhodochrosite is believed to be the source of the manganese oxide ores of the district, and has been found to occur below the oxidized zone. Pyrolusite is the chief mineral of the black oxide ore. Psilomelene is abundant and braunite, manganite and wad are sometimes found. Most of the pyrolusite is fine grained, soft earthy, but some shows the striated, radiating character that suggests the replacement of manganite. The texture of the ore ranges from finely porous to cavernous, but nodular or reniform structures are also common.

Quartz is an abundant gangue mineral, and occurs both as a sugary vein quartz, and as silicified limestone. Unaltered limestone and marble are found in the ore. Small amounts of clay, more or less mixed with limonite, partly fill cavities in the ore, and a cream-colored clay locally fills late fissures in or near the ore bodies.

The presence of manganite in the ore accounts for the fact that the tailings from the mill show a higher MnO_2 content after they have been allowed to stand, than they did when they were freshly milled.

Plant Study

In the investigation of the problem of beneficiation of the manganese crude from the Philipsburg District, a study of the existing concentration facilities of the

Taylor-Knapp Mining Company, was made on November 6, 1947, and again on December 28 of the same year. At that time, the manganese ore was concentrated by gravity and magnetic methods, to yield a product suitable for use in the battery industry. Difficulty was being encountered in producing a concentrate that would meet the specifications of the battery industry; (appendix II table I) and, obtain a relatively high recovery.

The processing, by the Taylor-Knapp Mining Company is separated into two distinct stages. In the first stage, the run-of-mine ore, which has a 35 percent manganese content, is upgraded to 45 percent by gravity concentration methods. The second stage is comprised of a magnetic separation circuit, in which the concentrate from the first stage is further upgraded to give a product of approximately 68 percent MnO_2.

<u>Crushing</u>. The run-of-mine ore is crushed by a Blake-type jaw crusher, to pass a $1\frac{1}{2}$ inch grizzly. During periods when mine is not operating, tailings are used as mill feed and are bypassed around the crushing circuit.

<u>Grinding</u>. The $1\frac{1}{2}$ crusher product is fed to rod mill where it is ground to approximately minus 8 mesh. The material is classified by two cone classifiers, the first of which makes a separation at the 8 mesh screen size. The plus 8 mesh material is the feed to the second cone classifier, which makes a separation at approximately 150 mesh. Because the friable nature of the manganese oxides tend toward the over-production of fines, the

grinding circuit demands much attention.

Concentration by Tabling. The products of the second classifier are fed to two concentrating tabling circuits. In the over size circuit, a concentrate, middling and tailing product is obtained, the middling being handled as a circulating load. The slimes are fed to the slime table, where a concentrate and a tailing are obtained. The concentrate from the slime tables is of high enough grade that no further upgrading is necessary.

Dewatering. Coarse table concentrates are fed into a pool, out of which an Esperanza-type drag dewaterer operates. The pulp feed runs 8 percent solids, and is thickened to a 76 percent solid content as a product of the drag operation. This material is dumped on the ground, where the run-off and evaporation of water further reduces the moisture content.

The concentrate from the slime tables is dried by an electric drier, and is transported to the slime concentrate bins.

Rotary Drier. Concentrate from the drag dewatering device loaded into trucks by means of a slusher, and are transported to the lower mill. They are dried in a coal fired, rotary dryer. This is a critical operation in which the temperature must be closely controlled. The temperature of the dryer is kept as nearly as possible at 110°C. If this temperature is exceeded, the water of crystalization and the nacent oxygen is driven off.

Vibratory Screen. The dried concentrates from the rotary dryer, are then passed through a series of vibratory screens where the following separations are made: plus 10 mesh; minus 10, plus 20; minus 20, plus 35, and minus 35, plus 50 mesh. The products of the screens are fed into separate bins by a belt conveyer. The plus 10 and plus 20 mesh material are used as feed to one Wetherill magnetic separator, while the plus 35 and plus 50 mesh material is the feed to the second magnetic separator. The separator bins are inter-changeable, in order that one mesh size may be fed to the separators at a time.

Wetherill Magnetic Separators. Two banks of separators are used, the first bank has five separators each having six poles, using 20 amperes, and is used alternately for the plus 10 and plus 20 mesh material. The second bank has three separators, each having four poles using 12 amperes and is used to concentrate the finer plus 35 and plus 50 mesh feed. The concentrates from each separator are stored in bins, and assayed separately. The higher grade material is then blended with that of lower grade to give a product that has a 68 percent MnO_2 content, and the resulting blend is stored for shipment.

Suggested Revisions of Plant

The object of this thesis was to try to effect a method, for the beneficiation of the manganese crude, that would be more effecient then that now in use, and which would still give a product that would meet the specifications of the battery industry. The manganese dioxide is useful to the dry-cell battery industry for its free oxygen content, and paid for on that basis. The specifications demand a high free oxygen content, and that the kind and amount of impurities be controlled.

In attacking the problem many ideas and schemes had to be considered. It was decided that the work would be approached as though the material were newly discovered, using both, the work done in the Taylor-Knapp plant and that done elsewhere as a guide to methods of beneficiation. Attention also had to be given to the specifications of the battery industry, and to the elimination of the unwanted impurities.

Flotation. Although flotation has proven to be an efficient method of beneficiation of the manganese crude, it cannot be used to produce battery manganese, because fatty acid reagents contaminate the material. Flotation, however, should be considered as a means of recovery of the manganese in the tailings for possible use as metallurgical manganese.

Sink-and-Float. This method of concentration was considered and discarded as impractical, because the dense media would contaminate the product.

Jigging. Although a difference in specific gravity exists between the manganese and gangue minerals in the ore, jigging was considered impractical because of the varying physical properties of the manganese oxides. Losses of manganese to the tailings would be excessive.

Tabling. This method, followed by magnetic separators, has been successfully used to give a product suitable for use as battery manganese. Gravity concentration, supplemented by several devices, was chosen as the basis of concentration studies.

Crushing and Grinding. Microscopic studies showed that complete liberation of the manganese oxides take place approximatly in the 35 mesh range. (Appendix I, Section II) This was chosen as optimum grinding size to obtain maximum liberation of the manganese minerals, and yet get a relatively small amount of fines.

A jaw crusher was judged to be the best preliminary crushing device. Grinding was best done in a rod mill, in closed circuit with a 35 mesh vibratory screen, which was chosen as the classification device because it gives a better sizing action on the manganese oxides, than other conventional classifiers.

Following the vibratory screen, a hydro-separator was used to effect a separation between the slimes and the coarser minus 35 plus 150 mesh material. It was found that the minus 35 plus 150 mesh product was of higher grade than the slimes, and that a certain amount of upgrading accoumanied this operation. (Appendix III, test1.)

As the battery specifications demand that the product contain less than 20 per cent minus 200 mesh material, the undersize from the hydro-separator was studied for possible use as metallurgical manganese.

Tabling. The minus 35, plus 150 mesh hydro-separator product was upgraded by tabling. It was found that a better concentrate and recovery was obtained when a sized product was used as the table feed. (Appendix IV, tests 1 and 2.) The use of an unsized table feed gave a very poor recovery as well as a poor concentrate. (Appendix IV, test 3.) Table tests for the slime material from the hydro-separator overflow gave a clean, high grade concentrate and a relatively high recovery.

Dewatering. The Dreisen hydrolic cone has successfully used as a thickening device on material which is comparable to the manganese dioxide table concentrates. Use of this device as a thickener was taken into consideration, and a three inch model was constructed. Time was not available for the tests.

General Discussion.

The main contribution of this thesis is the recognition of the fact that concentration of manganese dioxide by gravity concentration methods, favors the use of a sized feed. Good control, by use of classifiers, over the grinding circuit is of utmost importance, because of the friable nature of the manganese minerals. Since the table concentrates must be further upgraded by magnetic separators, an efficient method of filtering and drying must be used. It is suggested that further investigation of the possibility of the use of the Dreisen cone as a thickening device be carried on. It is also possible that the hydrolic cone might replace the hydro-separator, and prove to be superior because of its higher capacity.

Tabling Summary

The best recovery of manganese dioxide made on a table test was 71.3 percent in a concentrate containing 64.30 percent MnO_2. This, however was made on a slime product, which would not meet battery specifications, because of the high percentage of minus 200 mesh material, and also because the manganese content was too low. The product might prove suitable, upon sintering, as metallurgical manganese.

A revovery of 69.7 percent of the manganese dioxide, in a concentrate containing 59.64 percent MnO_2 was made on a table test of a minus 35 plus 150 mesh material. This met all specifications except that of MnO_2 content. This, however, could be further upgraded by magnetic separation.

Tabling, followed by magnetic separation appears to be the best method of producing battery manganese at the present time. It is possible that further investigation will prove that the manganese that is now lost in the tailings, can be recovered and utilized.

APPENDIX I

Section 1

Specifications of Dry Cell Battery Industry.

Maximum Impurity

Fe %	Pb %	Cu %	H_2O %	As_2O_3 %	CO_2 %	Ag oz.
2.00	0.25	0.03	3.00	0.05	2.00	8.00

A minimum of 68.2% MnO_2 and a maximum of 20% minus 200 mesh material were also specified.

APPENDIX I

Section 2

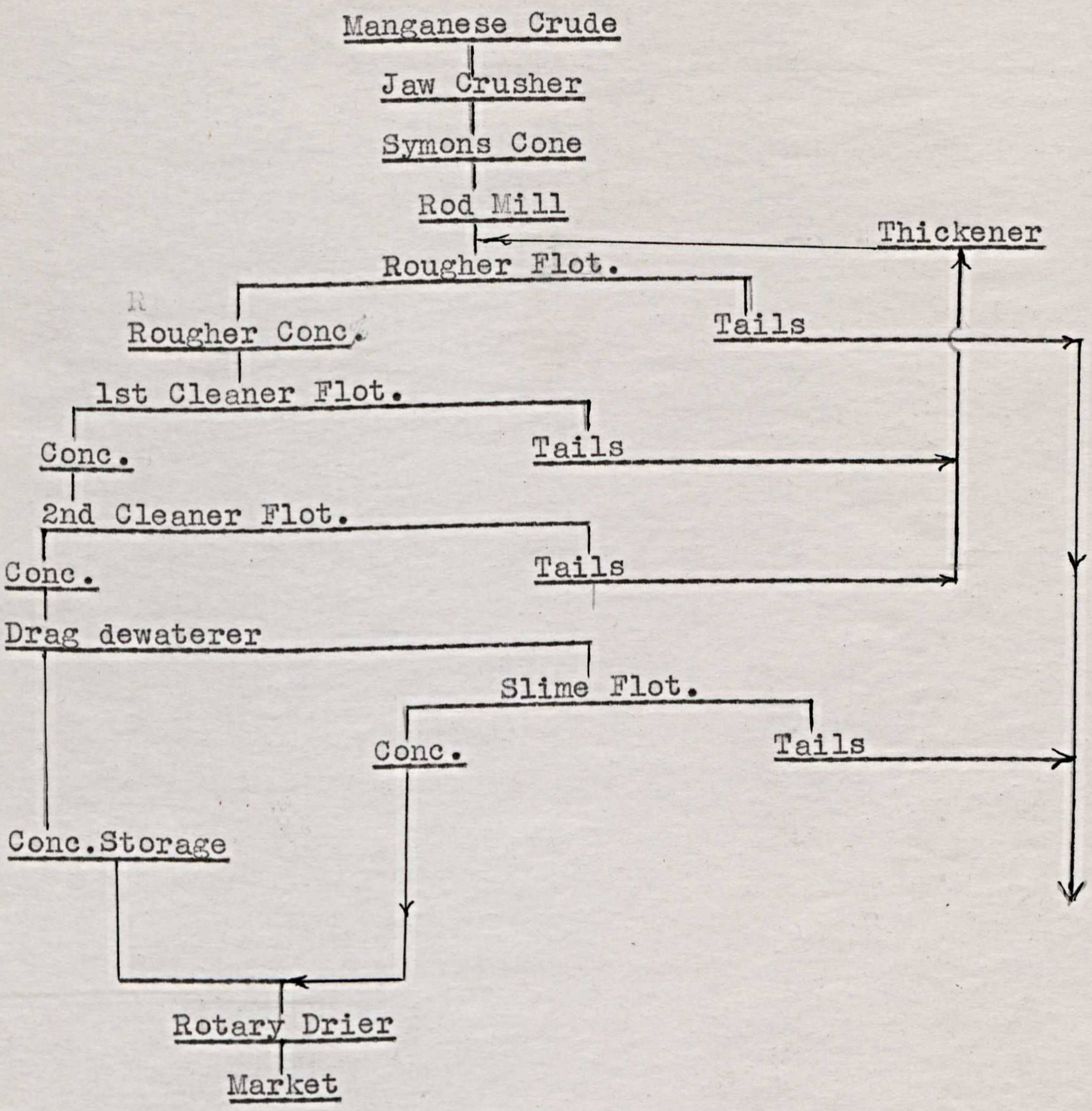
Grinding Tests on Run of Mine Ore.

All grinds were made in a rod mill, with a charge of 600 grams of feed to 600 c.c. of water. The feed was minus 8 mesh. The grinding time was varied and the following results obtained:

Mesh size	Time in Minutes 5	7	10
- 35	35.20	20.08	0.70
- 35 - 48	22.15	19.45	4.22
- 48 - 65	16.21	13.24	8.90
- 65 - 100	12.39	11.05	13.10
- 100 - 150	6.20	9.86	24.49
- 150 - 200	3.85	7.54	12.52
- 200	9.00	17.76	36.00

APPENDIX 2 SHEET 1

Flow Sheet of Cuban Plant.



APPENDIX III

Test I

All material to the hydro-separator was stage ground in a rod mill for a period of 5 minutes, resulting in a pulp, 91% plus 200 mesh. The rod mill product was screened through a 35 mesh screen, the plus 35 mesh material being returned to the rod mill.

The hydro-separator feed was the minus 35 mesh material, and was further classified into a minus 35 plus 150 mesh product and a minus 150 mesh product.

Products	Weight	MnO_2 %	% of Total MnO_2
Feed	5000	33.56	100.0
- 35 - 150	3846	35.90	81.7
- 150	1154	28.61	18.3

APPENDIX IV

Tables tests were run using various rod mill and hydroseparator products as feed. Feed to all table tests was minus 35 mesh.

All assaying was by the Montana Division of the United States Bureau of Mines.

Test I

Object: To effect a clean separation of the manganese dioxide and the gangue minerals in the minus 35 plus 150 mesh hydro-separator product.

Results:

Products	Weight	MnO_2 %	% Total MnO_2
Feed	2500 gms.	35.90	100.0
Conc.	1047 gms.	59.64	69.7
Mids	533 gms.	24.92	14.8
Tails	920 gms.	15.01	15.5

Remarks: Tails appeared dirty because slope of table necessary for a clean concentrate was too great. Finer material was washed over the riffles.

Test II

Object: To effect a separation of the manganese dioxide and the gangue material in the minus 150 mesh hydro-separator product.

Results:

Products	Weight	MnO_2 %	% Total MnO_2
Feed	1000	28.61	100
Conc.	317	64.30	71.2
Mids	158	15.22	8.4
Tails	525	11.12	20.4

Remarks: Clean concentrate was obtained.

Test III

Object: To effect a separation of the manganese dioxide and gangue minerals from the minus 35 mesh rod mill product.

Results:

Products	Weight	MnO_2 %	% Total MnO_2
Feed	1500	33.56	100.0
Conc.	403	43.73	35.0
Mids	194	40.25	15.5
Tails	893	27.86	49.5

Remarks: Tails and concentrate appeared dirty.

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Mr. W. M. McAuliffe has satisfactorily completed, this 28th day of May, 1948, all requirements prescribed by the Montana School of Mines for the degree of Bachelor of Science in Metallurgy.

Metallurgy Department

Donald W. McGlashan