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Utilization of Low Grade Alabama Manganese Ores

(Revised)

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UTILIZATION OF LOW GRADE ALABAMA MANGANESE ORES

ABSTRACT

The object of this investigation was to find a general and profitable method for the purification and concentration of low grade Alabama manganese ores, which had little, if any, value in their present form.

The various samples of Alabama manganese ore examined on a small scale were readily attacked by even weak solutions of sulphur dioxide in water and apparently a process applied by the Bureau of Mines to western manganese ores could be applied to Alabama ores.

The above process consists of the solution of the manganese ore in a solution of sulphur dioxide in water and the resulting solution of manganese sulphate is converted into an oxide of manganese by calcination.

The optimum conditions for the extraction of the manganese have been determined. These, on a plant scale, probably would be somewhat different.

The probable large scale process has been described and the cost of the process estimated. The treatment of ores containing at least twenty-five per cent manganese apparently shows considerable promise.

Utilization of Low Grade Alabama Manganese Ores

INTRODUCTION

A number of deposits of manganese ores occur in Alabama, the extent and quality of which have not been thoroughly determined². Only a few small scale and sporadic attempts have been made toward the concentration of these ores.

Most of these ores contain, in addition to manganese, considerable quantities of iron. Some contain several per cent of cobalt³. In view of the high value of cobalt, the treatment of such ores might be quite profitable.

Considering the little progress that has been made in the utilization of Alabama manganese ores, such work appeared to be of value.

BIBLIOGRAPHICAL REVIEW

C. M. Weld and others¹ discuss in detail the uses, preparation, mining costs and the production of ferro-alloys from manganese ores. This bulletin includes a discussion of the leaching of western manganese ores with Sulphur Dioxide recovered as a by-product of certain smelting operations. This is an important source of information.

Relative to Alabama ores, the extent and quality have not been thoroughly determined². At least some of these ores contain cobalt³. Various Alabama ores have been classified by Jones⁴⁻⁵. The Swann Chemical Corporation⁶ applied electric smelting to some of these ores during the war. The reaction that takes place between SO_2 solutions and MnO_2 is discussed by Vadner⁷. The use of manganous sulphate as a fertilizer is discussed by Fish⁸ and others⁹.

NATURE OF ALABAMA DEPOSITS

There are three groups of deposits of manganese ore in Alabama according to Jones⁴. They are as follows:

1. In residual clays weathered from the Weisner quartzite, of Cambrian age. The manganese-bearing parts are practically confined to Cleburne County. This ore occurs in more or less stratified layers intermixed with clay and pebbles.
2. In residual clays weathered from the Knox Dolomite of Cambro-Ordovician age. This type occurs in Cherokee, Tuscaloosa, Bibb, Shelby, Calhoun and other counties. The deposits in Calhoun and Cherokee counties occur in the form of surface pellets, or as pebbles and chunks associated with brown ore and bauxite in the limonite banks of that region. Over the rest of the area carrying this type of deposit, the association is with limonite, and might be better classed as manganiferous limonite.

3. Near the top and bottom of the Fort Payne Chert, of Mississippian Age. This type is largely confined to Blount and Etowah counties. This strip of ore is about five miles long and has produced some ore of very good quality.

Jones gives the following as typical of Alabama manganese ores:

	Ore from Stock Mills	Ore from Cleburne Co.	Ore from Woodstock (Bibb County)	
			(1)	(2)
Mn	45%	—	11.44%	13.68%
MnO ₂	—	62.55%	—	—
Fe	5.0	—	38.50	41.76
Fe ₂ O ₃	—	9.64	—	—
F	0.8	—	—	—
SiO ₂	8.0	3.67	11.45	24.65
P	0.8	—	0.27	0.55
H ₂ O	—	—	11.62	10.66

The ore is usually psilomelane, pyrolusite, and manganite and may be contaminated with brown iron ore.

VARIOUS METHODS FOR THE CONCENTRATION OF MANGANESE ORES AND THE SELECTION OF A PROCESS APPLICABLE TO ALABAMA MANGANESE ORES

Concentration processes that have been developed¹ for the concentration and purification of manganese ores can be classified as follows:

I. Simple Methods of Concentration

These include selective mining, hand picking, jigging, screening, log washing, classification, roughing table treatment, slime or vanner treatment, pneumatic separation, and combinations of two or more of the above methods.

II. Complex Methods

These include (1) magnetic separation, (2) electrostatic separation, (3) hydrometallurgical processes, including various processes of leaching with various acids and precipitation by chemical substances, or evaporation of solution and heat treatment in rotary kilns, (4) preliminary thermal process including drying to remove hygroscopic moisture, calcination to remove CO₂ or combined water, volatilization of manganese at high temperatures, and direct reduction of oxides by carbon, and (5) miscellaneous processes including flotation and the use of heavy solutions.

There is very little information concerning the application of the above processes to Alabama ores but processes under group 1 are probably the only ones that have been used to any extent at the present time.

Examination of Alabama ores shows them to be intimately mixed with impurities. These ores are mixed with limonite and it was thought that reduction to magnetic oxides of iron and subsequent magnetic separation might be practical but preliminary work indicates that the oxides of manganese

and iron are too intimately mixed. It has been found by Barneveld¹ that such ores are not usually amenable to ordinary mechanical separation.

For the above reasons, it was thought desirable to study a leaching process. Of these processes, the method¹ developed by the Bureau of Mines and applied to western ores whereby the ores are extracted with sulphur dioxide solution, appeared to be the most promising.

PRELIMINARY WORK ON SO₂ EXTRACTION

Rough preliminary work was done on representative Alabama ores. This work indicated that most of the manganese can be readily extracted from these ores with SO₂ solutions.

It further showed that these ores can be divided into two classes with respect to difficulty of extraction: (1) Soft and, (2) Hard. The soft ores are those that can be easily pulverized between the fingers. Hard ores approach hematite in hardness.

Class (1) responds to treatment without any difficulty. This work indicated that the preliminary conditions for successful extraction were as follows:

Table I

Preliminary Conditions

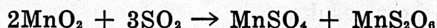
Size of Ore -----	Lumps of any size
Time of Extraction -----	Short—5 minutes
Temperature of Extn.-----	Room temperature or slightly above
Concentration of Solutions----	Concentration of about 1% were used successfully
Excess of SO ₂ -----	A fair excess of SO ₂ is required (50%)

Class (2) is somewhat refractory. The ore must be finer, the time of extraction longer and fairly high temperatures are required for good results.

ANALYTICAL PROCEDURE

Standard analytical methods were used for all analyses with the exception of the determination of manganese in the SO₂ extract. Volhard's method, with modification, was used for this analysis. On other manganese determinations, the standard Volhard method was used.

According to Vadner⁷, the reaction of SO₂ solutions and MnO₂ is represented by the following equation:



The most successful method of removing the excess SO₂ was found to be accomplished by boiling the solution to dryness and heating it to a temperature somewhat above 200 degrees C. for about five minutes. At this temperature, the dithionate was found to decompose, giving SO₂ and MnSO₄. All manganese determinations showing the amount of manganese extracted by the SO₂ were made in this way.

The manganese solution was taken up with a little water, HNO₃ and H₂SO₄, and boiled to SO₃ fumes. The nitric acid is necessary to oxidize the

iron to the ferric condition so that it will precipitate upon the addition of ZnO emulsion.

From this point the procedure is that for Volhard's standard method.

SO₂ solutions may give inaccurate results, due to the tendency of SO₂ to escape from solution, unless they are standardized daily.

EXTRACTION PROCEDURE

Extractions were made in stoppered 300-400 cc. Erlenmeyer flasks. From 0.1 gram to 1 gram of ore (size of sample was governed by manganese content) was placed in a flask, the SO₂ solution run in from a burette and the flask agitated by hand.

For temperature above room temperature a thermometer was inserted into the flask through the stopper. The flask was then placed partially in a water bath and agitated intermittently at the desired temperature. The temperature could be held within a range of about 2½ degrees of the one desired.

Filtration was done by means of a Buchner funnel and a suction flask. Ordinary filtration is very tedious.

DEVELOPMENT OF PRELIMINARY PROCESS FOR RECOVERY OF MANGANESE DIOXIDE

Examination of a number of samples, indicated that extraction of the ore was usually completed in five minutes. For good extraction of the ore an excess of sulphur dioxide over the theoretical was required. A fifty percent excess gave reasonably good results. Preliminary results indicated that a one percent sulphur dioxide solution was effective. After experience with several temperatures, a temperature of 60° C. was adopted as the proper temperature for the extraction.

As shown later, sulphur dioxide extracts were treated with fresh ore to precipitate the iron and phosphorus. The iron and phosphorus were filtered off, and the manganese sulphate solution concentrated, yielding on crystallization manganese sulphate, or on calcination, manganese dioxide.

DETERMINATION OF OPTIMUM CONDITIONS

The optimum conditions were determined on a typical hard ore such as that found at Auburn, Alabama. This ore contains 5.43% manganese. Although this sample does not represent a large ore body it is a typical Alabama ore. These conditions were then applied to six other ores and modified to give the best results.

Fineness of the Ore:

The fineness of the ore was varied, holding the other variables constant. The other variables were previously determined by preliminary work. They were as follows:

Time	-----	5 Minutes
Amount of SO ₂	-----	50% excess over the theoretical
Concentration	-----	1% Solution
Temperature	-----	60 degrees C.

The results obtained with ores of different degrees of fineness are given in Table II.

TABLE II

Degree of Fineness

	Percent of Manganese Extracted
20-40 Mesh-----	14.04 %
60-100 Mesh-----	34.32 %
Through 200 Mesh-----	43.68 %

A degree of fineness beyond 200 mesh was not considered practical from an economic standpoint. For this reason 200 mesh was accepted as the best degree of fineness for this ore, since materially better results were secured than at 60-100 mesh.

Excess of SO₂ Necessary:

The theoretical amount of SO₂ necessary for extraction was determined from the following reaction:



Preliminary results indicated that a moderate excess over the theoretical was necessary. Consequently, results were secured with 50, 100, 200, and 300 percent excess sulphur dioxide.

In the above determination the optimum degree of fineness previously determined was employed. The other conditions were the preliminary conditions previously determined.

The results secured with various quantities of sulphur dioxide are given in Table III.

TABLE III

Excess of Sulphur Dioxide

Percent Excess	Percent of Manganese Extracted
50 -----	43.68
100 -----	60.8
200 -----	70.69
300 -----	74.0

Little increase in extraction being secured above 200%, this amount was adopted for future work.

Temperature:

The temperature employed in the preliminary work was 60° C. Employing the optimum degree of fineness and the optimum excess of sulphur dioxide (the other conditions being the preliminary conditions discussed on page 7), runs were made at the temperature (60° C.) and at 45° C. and 30° C. (room temperature). In view of the volatility of the sulphur dioxide, temperatures exceeding 60° C. did not seem to be practical. The results are given in Table IV.

TABLE IV

Temperature

	Percent of Manganese Extracted
30° C (room temperature) -----	54.60
45° C -----	69.95
60° C -----	70.06

These results indicate that there is little advantage gained by employing temperatures exceeding 45° C. Consequently this temperature was adopted at the optimum temperature for extraction.

Concentration of Sulphur Dioxide:

Employing optimum conditions determined above and the other preliminary conditions the concentration was varied on both sides of the preliminary figure of 1 per cent. The results are given in Table V.

TABLE V

Concentration of Sulphur Dioxide

Percent of Sulphur Dioxide by Weight	Percent of Manganese Extracted
0.5 -----	70.37
1.0 -----	70.45
2.5 -----	70.29

According to the above table, one per cent solutions were as satisfactory as much stronger solutions (2.5 per cent) as far as extraction of the manganese is concerned. However, in order to reduce the cost of the evaporation of the solution of manganese sulphate as much as possible, on a large scale much stronger extraction solutions, as recommended by the Bureau of Mines, are desirable. See Probable Large Scale Apparatus.

Time:

Using the other optimum conditions previously determined as shown above, the time of extraction was varied from one minute to ten minutes. The results as given in Table VI follow:

TABLE VI

Time of Extraction

1 Minute -----	56.4 %	Extraction
5 Minutes -----	70.20 %	Extraction
10 Minutes -----	70.93 %	Extraction

This table indicates that there is little advantage in an extraction time above five minutes. Consequently this was adopted. A one gram sample was used in this work.

BEST CONDITIONS FOR EXTRACTION OF SIX REPRESENTATIVE ALABAMA ORES

Recognizing that there was considerable variation in the various ores in the State, the best conditions for six ores varying in manganese from 5.43 percent to 43.47 percent were determined. The results are given in Table VII.

TABLE VII
Optimum Conditions for Six Alabama Ores

Source of Ore	Physical Characteristics	Mn Content % Mn	Size of Ore (Mesh)	Temp. of Soln. Deg. C	Time of Extn. Minutes	% Excess SO ₂ used	% Mn Recovered
Auburn, Ala.	Hard	5.43	200	45	5	200	71
Alexander City, Ala.	Hard	43.47	200	45	10	250	96
Childersburg, Ala.	Soft	36.38	Lumps*	30° C	5	150	93
Walnut Grove, Ala.	Soft	28.29	Lumps	45	2	250	91
Sulligent, Ala.	Soft	1.91	Lumps	45	2	150	95.5
Heflin, Ala.	Soft	9.61	Lumps	30° C	5	125	81

*These ores are so soft that large lumps are reduced to a pulp when mixed with SO₂ solutions and agitated.

1% solutions were used on all these ores.

Note that the hard ores are more difficult to extract.

Iron and Cobalt in SO₂ Extracts:

Qualitative work was done on ores from Walnut Grove, Childersburg and Alexander City. The extract obtained by use of the optimum conditions were first tested. It was found that relatively large quantities of iron are contained in these solutions.

Further work on precipitation of iron from these solutions indicated that practically all of the iron is removed by the addition of fresh manganese ore. Other groups of elements were found only in very small amounts.

The ore from Alexander City contained small amounts of cobalt, the most of which apparently is soluble in SO₂ solutions.

Quantitative Determination of Iron and Phosphorus in the SO₂ Solutions:

Quantitative analyses of iron and phosphorus were made on a soft ore from Childersburg, a hard ore from Childersburg and a soft ore from Walnut Grove.

Iron, phosphorus and silica are the most objectionable impurities found in Manganese ores. Alabama ores examined ranged from 0.27 to 0.8 percent phosphorus and from five to twenty-five per cent iron oxide⁵. Of these three constituents, only iron and phosphorus are capable of being dissolved to any extent in SO₂ solutions.

SO₂ extracts obtained by application of the optimum conditions were treated with an excess of fresh ore to precipitate iron and phosphorus as indicated by qualitative work. The iron and phosphorus were filtered off, and the manganese solution concentrated whereby the manganese was precipitated as MnSO₄ which on calcination gives MnO₂. The results of these analyses are given in the following table:

TABLE VIII
Purity of Precipitated Manganese Oxides

Ore	Iron in MnO ₂ obtained by Ppt. (Percent by weight)	Phosphorus in MnO ₂ obtained by pt. (Percent by weight)	Mm in Mm Oxides (Percent)
Childersburg Hard Ore	0.02	Trace	60 (approx.)
Childersburg Soft Ore	0.008	Trace	60 (approx.)
Walnut Grove	0.017	Trace	60 (approx.)

No precipitate of phosphorus was obtained with ammonium molybdate. The iron content seems low enough to meet the specifications for any manganese product used commercially.

Sulphur Dioxide Lost in the Process:

An excess SO₂ is required to cause the solution to go to completion. Some SO₂ is tied up as dithionate. A portion of this SO₂ is precipitated by lime and other compounds and cannot be recovered. Work was done on three ores to determine just how much cannot be recovered by roasting.

The procedure was to dissolve the ores using the optimum conditions, additions of fresh ore, filtering, concentrating and driving the SO₂ off at about 400° C. into NaOH solution. The excess NaOH was titrated with standard HCl. At 400° C. all dithionate is decomposed into SO₂ and MnSO₄. From these determinations the percentage excess of SO₂ above that required to produce MnSO₄ was calculated. The results are given in the following table.

TABLE IX
Sulphur Dioxide not Recoverable

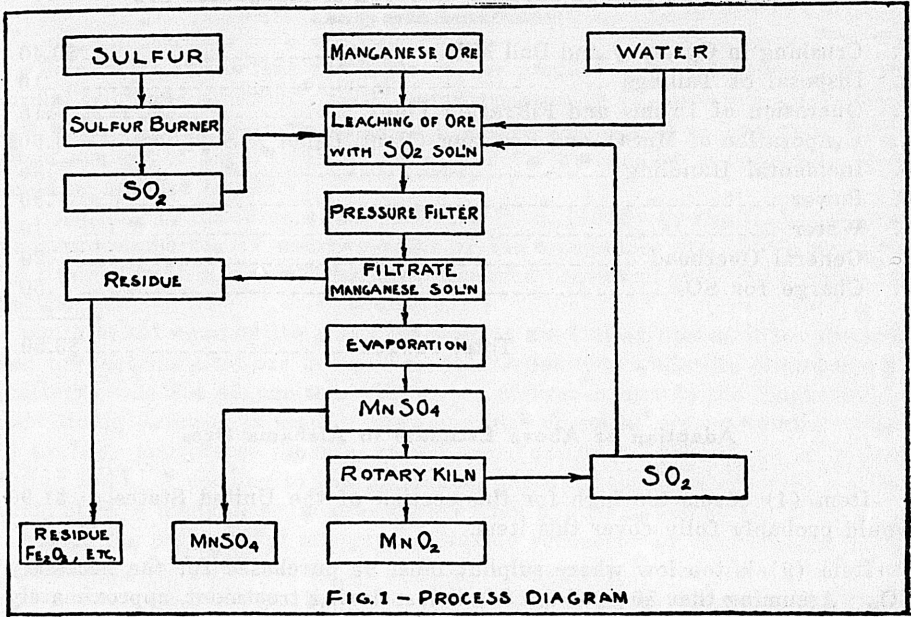
Ore From	Excess SO ₂ Not Recoverable (Percent by weight)
Walnut Grove -----	24
Childersburg -----	31
Alexander City -----	27

This work indicates that about 30% of the amount of SO₂ required to produce MnSO₄ cannot be recovered. This work gives a fairly good idea of the excess required, and of the amount of SO₂ which cannot be recovered.

PROBABLE LARGE SCALE APPARATUS

Barneveld¹ gives a discussion of a plant of 250 tons capacity located in Arizona. Corrosion and filtration are discussed. A flow sheet and an idea of equipment needed is given. He estimates that a 250 ton capacity plant would cost approximately \$100,000 in Arizona. Such a plant, being nearer the production center for the equipment needed, might not be as expensive in Alabama.

In carrying out the above process, a suitable storage would be necessary for the sulphur and manganese ore. A satisfactory source of water also would be required. A rotary type of improved sulphur burner, or a vertical



type frequently employed for small chamber sulphuric acid plants might be used. The leaching might be carried out in two stages employing two tandem drums for the purpose, as suggested by the Bureau of Mines. After the leaching is completed, the resulting solution might be filtered through a pressure filter and the nearly saturated solution concentrated in evaporators. The resulting manganese sulphate might be calcined in a rotary kiln giving solid manganese oxides.

For complete details see Bureau of Mines Bulletin 173, by C. M. Weld and others entitled "Manganese", pages 57-69. This bulletin was published in 1920.

A flow sheet in its simplest form is given in Figure 1. This does not include the grinding of the ore.

COST OF MANUFACTURE OF MANGANESE DIOXIDE FROM ALABAMA ORES

In the following cost estimates, the cost of production of MnO_2 is considered. Although $MnSO_4$ can be produced, MnO_2 is the product of most importance. An ore containing 25% manganese is assumed.

Barneveld¹ gives the following cost estimate for SO_2 treatment of an ore of approximately this manganese content as follows:

Estimated Cost of Treating One Ton of Manganese Ore

1.	Crushing in Gyratory and Ball Mill -----	\$0.40
2.	Disposal of Tailings -----	.10
3.	Operation of Drums and Filtration Plant -----	.15
4.	Evaporation of MnSO ₄ and Roasting (Heat, Labor & Supplies) ---	1.50a
5.	Incidental Handling -----	.25
6.	Power -----	.30
7.	Water -----	.10
8.	General Overhead -----	.20
9.	Charge for SO ₂ -----	.50
Total -----		\$3.50

Adaption of Above Estimate to Alabama Ores

Item (4) seems too high for this section of the United States. \$1.00 would probably fully cover this item.

Item (9) is too low where sulphur must be purchased for the necessary SO₂. Assuming that 50% of the SO₂ is lost during treatment, approximately 0.3 unit sulphur is required for each unit of manganese. According to information available, one ton of sulphur delivered to Birmingham would cost approximately \$29.00. In 300 ton lots, the cost would be about \$26.00. On this basis, the charge for the SO₂ would be \$2.00.

Correcting for the SO₂, the cost of treating one ton of ore (25%) would be \$4.50. To this must be added the cost of mining, haulage to the railroad, loading, sampling, general handling, and railroad freight to smelter. Barneveld allows \$2.00 per ton for mining, \$3.00 for haulage to the railroad, i.e., 30c per ton mile, \$1.00 for loading, sampling, etc., and \$2.50 for railroad freight to smelters.

These figures would require considerable modification in Alabama. Much of the Alabama ore is on or near a railroad, and the ore might be refined at the mines. Hence the figure of \$3.00 per ton for haulage to the railroad, and \$2.50 per ton for railroad freight to the smelters should be considerably reduced. Probably these combined figures could be reduced to \$1.50 which at 30c per ton mile permits a haulage of five miles. The cost of mining (\$2.00 per ton) also seems too high. If the cost of mining does not exceed that of coal, \$1.00 per ton would be a more reasonable figure.

On the basis of the above figures, then the complete cost for treating one ton of Alabama ore containing 25 percent manganese would be as follows:

1.	Cost of treating one ton of ore-----	\$4.50
2.	Cost of mining-----	1.00
3.	Cost of loading, sampling, etc.-----	1.00
4.	Cost of haulage to railroad and railroad freight-----	1.50
Total -----		\$8.00

VALUE OF MnO_2 FROM ONE TON OF ALABAMA ORE (25% MANGANESE)

Barneveld¹ states that the value of the product from this process under normal economic conditions would be approximately 75 cents per unit. One unit is 22.4 lbs. of manganese dioxide or one percent of a long ton. This would amount to \$29.68. If 95 percent of the manganese is recovered, this would be reduced to \$28.19.

According to the Mineral Resources of U. S. (1928) by the U. S. Department of Commerce, the average value of the manganese ore (35% or more Mn.) mined in U. S. was \$27.50. The so called "chemical ore" had a value of \$41.40.

In the 1931 issue of Mineral Resources of the United States, it is reported that the metallurgical ore dropped to \$13.23 per ton, while the chemical ore (battery) was \$35.40 per ton. The same reference quotes the Engineering and Mining Journal as stating that domestic chemical ore containing from 70 to 72% manganese dioxide, varied from \$40.00 to \$60.00 per long ton during 1931. According to this reference, imported chemical ore with a minimum of 80% manganese dioxide, varied from \$50.00 to \$60.00 per ton.

Since the precipitated manganese dioxide recovered by this process is of high purity, its value should be strictly comparable to the high grade imported ore. On this basis, the value of the manganese dioxide recovered from one ton of ore containing 25% manganese assuming 90% recovery should be from \$17.78 to \$21.95.

According to the Mineral Resources of the United States the domestic plus the imported ore, exceeds 600,000 tons per year.

COMMERCIAL POSSIBILITIES OF THE PROCESS

It will be noted that the total cost of treating one ton of ore (25% manganese) is estimated to be \$8.00 while the total value of the manganese dioxide recovered from one ton of ore is from \$17.78 to \$28.19. Deducting the estimated cost of treating the ore, the estimated profit is from \$9.78 to \$20.19 per ton of ore treated.

The margin of profit might be increased under the following conditions:

- (1) Where the manganese content of the ore exceeds twenty-five percent. Several of the ores examined (see page 11) contained from 28 to 43 percent.
- (2) Where the ore contains considerable cobalt in a form where it might be recovered to advantage, Barneveld shows that this process is applicable to such ores.

Under any of the above conditions, the treatment of Alabama ores should offer considerable promise.

USES OF MANGANESE COMPOUNDS

Manganese compounds are used in a variety of ways. Probably the greatest use is in the manufacture of ferroalloys for steel manufacture. All steels carry some manganese, while high manganese steels carry up to 12 or 14 percent.

Manganese dioxide is used in considerable quantities in glass manufacture. It is also used as a laboratory reagent and in the manufacture of other

manganese compounds. Considerable quantities are employed in the battery industry.

Recently manganous sulphate has been found to be of value as a fertilizer. F. H. Fish⁸ states that specifications for fertilizers in the next few years may call for small percentages of manganese.

SUMMARY

The effect of sulphur dioxide dissolved in water on a number of Alabama ores has been studied on a small scale and the apparent suitability of these ores for the application of the process applied by the Bureau of Mines to western ores, has been determined.

The optimum conditions for extraction of the manganese have been determined. These conditions probably would be somewhat different on a plant scale.

The probable large scale equipment has been discussed and the cost of the process estimated.

It appears from the work completed on a small scale that where the ores average at least 25 per cent manganese the process offers considerable promise.

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