

[72] Inventor **David Weston**
Suite 500 129 Adelaide Street, Toronto,
Ontario, Canada
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 and a continuation-in-part of Ser. No.
564,033, July 11, 1966. This application
Apr. 24, 1967, Ser. No. 633,241

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[54] **FLOTATION OF METALLIC OXIDES III**
51 Claims, 5 Drawing Figs.

[52] **U.S. Cl.**..... **241/16,**
209/5, 209/166, 241/24
 [51] **Int. Cl.**..... **B02c 17/00,**
B03d 1/02
 [50] **Field of Search**..... **209/166,**
167, 5; 241/20, 24, 16

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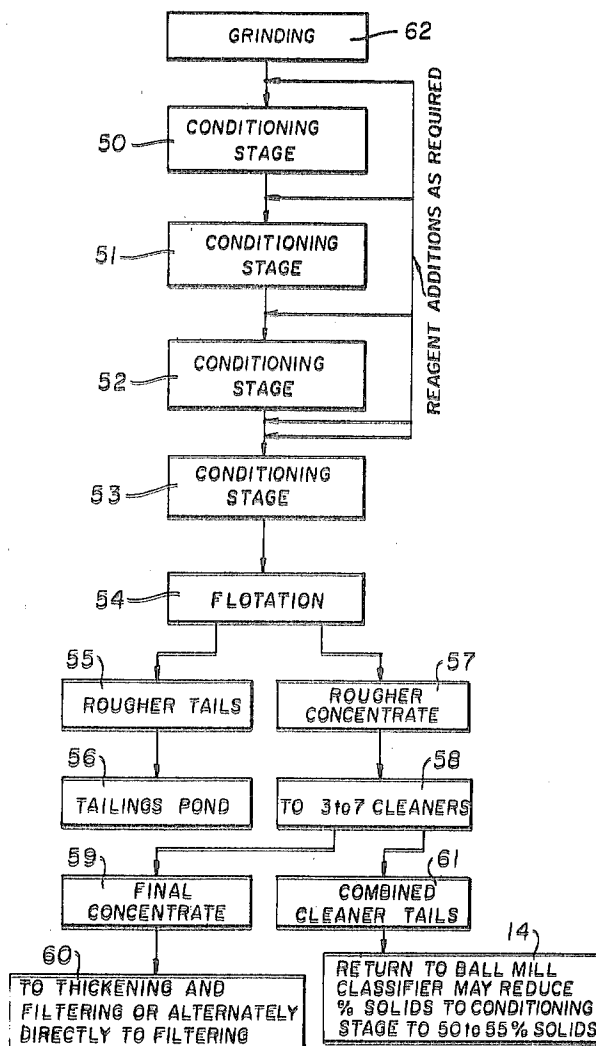
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Primary Examiner—Frank W. Lutter
 Assistant Examiner—Robert Halper
 Attorney—Smart & Biggar

ABSTRACT: A process for the flotation of metallic oxides in which a dispersed pulp is conditioned in a number of stages to achieve differential flocculation of the metallic oxides and dispersion of the gangue materials prior to flotation.



SHEET 1 OF 4

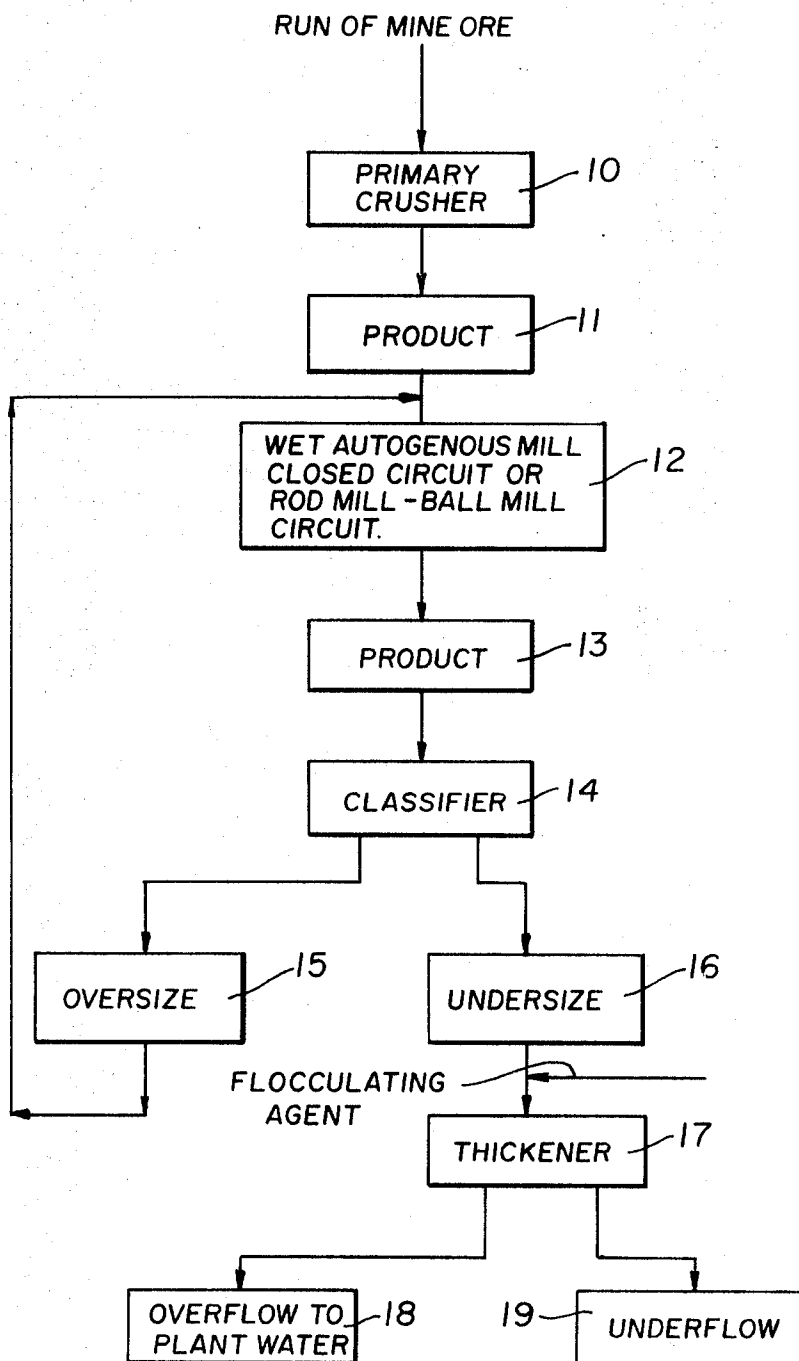


FIG. 1

INVENTOR
DAVID WESTONBY *Smart & Biggar*
ATTORNEYS

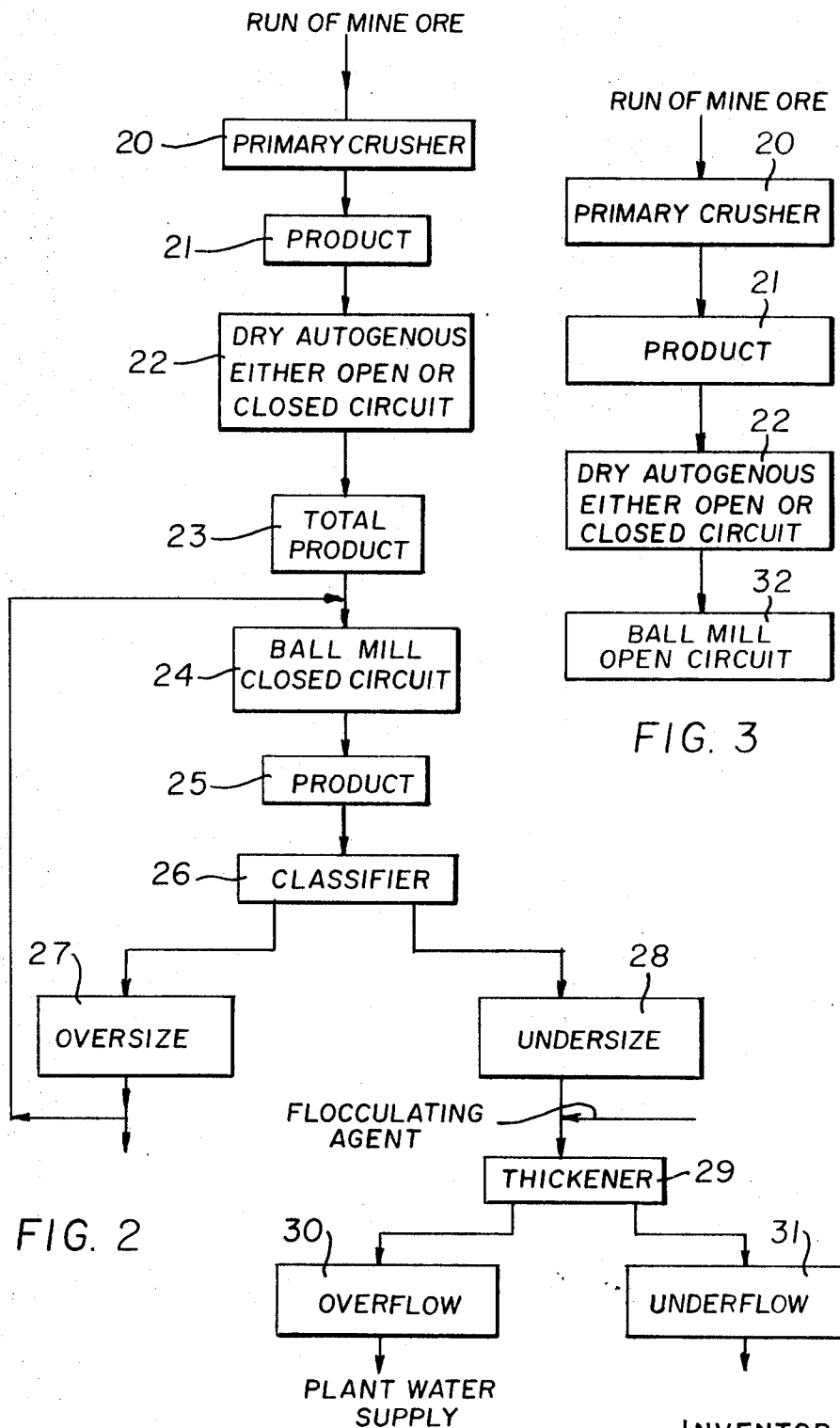
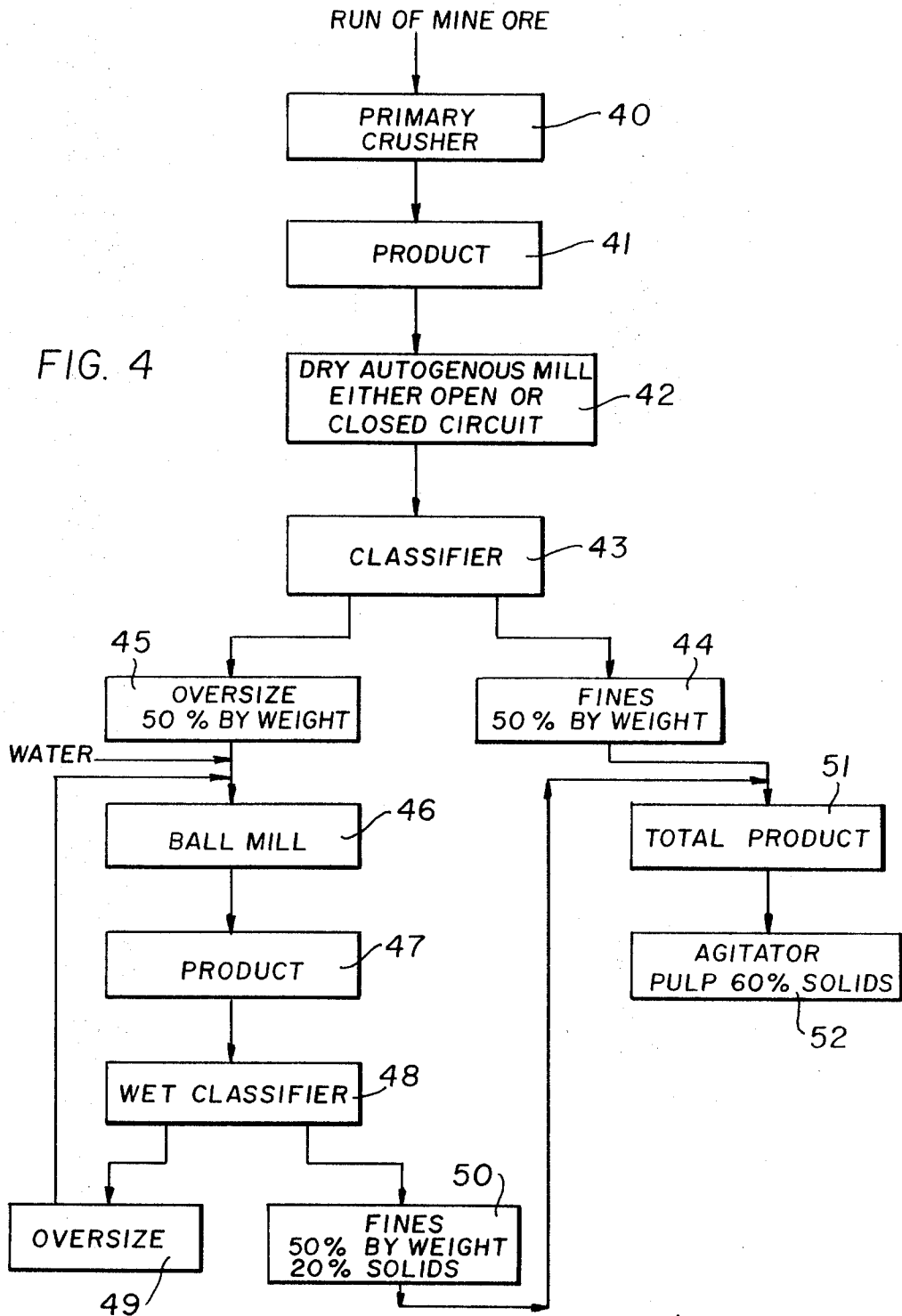


FIG. 3

FIG. 2

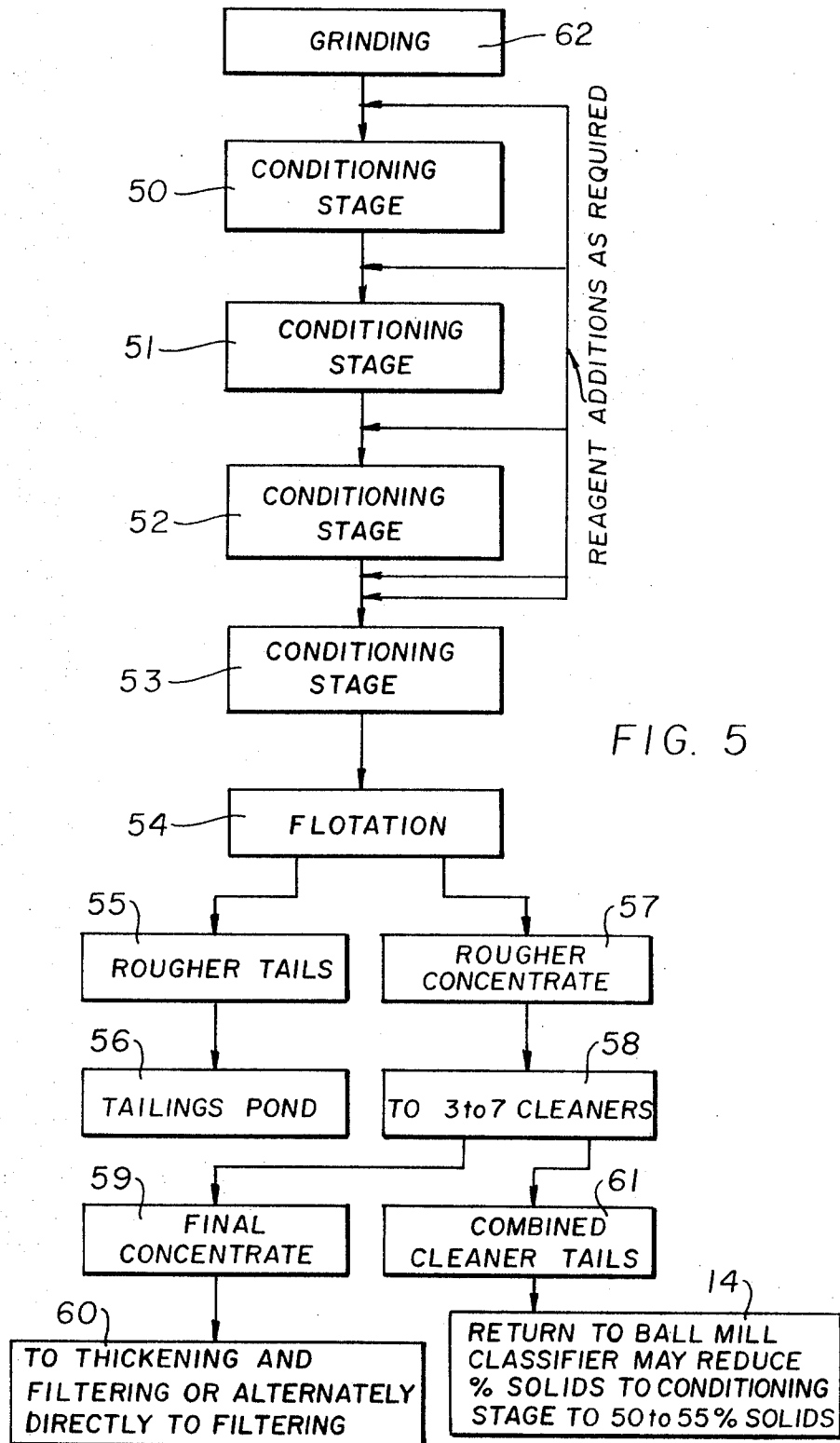
INVENTOR
 DAVID WESTON
 BY *Ernst & Biggs*
 ATTORNEYS

FIG. 4



INVENTOR
DAVID WESTON

BY- *Emmett & Biggs*
ATTORNEYS



INVENTOR
DAVID WESTON
BY *Smart & Biggar*
ATTORNEYS

FLOTATION OF METALLIC OXIDES III

This application is a continuing-in-part of my prior applications Ser. No. 550,922, filed May 18, 1966 now abandoned and 564,033 filed July 11, 1966.

This invention relates to a process for the flotation of metallic oxides and metallic oxides with water of crystallization in chemical combination.

Although much has been written in the technical literature concerning the flotation of this class of materials and a vast amount of money has been spent on research for many years particularly in respect to iron ores of this class, these materials remain for the most part refractory to economic flotation.

I have now found a process by which these materials may be readily and effectively concentrated by flotation without the need for any desliming prior to flotation.

My invention does not involve the employment of new or unusual reagents nor does it depend upon the use of special apparatus nor of techniques beyond the skill of those currently versed in the flotation art. My invention on the other hand, consists in the employment of a particular sequence of carefully controlled process steps which I have found to be necessary to obtain a controlled differential flotation of minerals of the above class. I have found that metallic oxides tend to flocculate with the gangue materials in the ore, this tendency being aggravated by the high degree of sliming which normally results from the high proportion of natural slimes in the ores and the fine grind necessary to achieve mineral liberation. Where the mineral and gangue materials are flocculated together prior to flotation it becomes impossible to obtain any useful degree of concentration by floating the thus flocculated material. It is the solution to this problem which enables my new process to be effective.

GENERAL DESCRIPTION OF THE INVENTION

According to my invention the comminuted ore in a pulp is initially caused to become dispersed to a sufficient degree that minimum flocculation is present. The achieving of such dispersion (which I herein call effective dispersion) in some instances with particular ores requires careful control of pH, conditioning time and addition of reagent. In other cases it is readily achieved without the addition of reagent over a substantial range of pHs and conditioning times. When effective dispersion has been achieved a beaker sample of the pulp will, while standing, usually give a visual indication in that the minerals settle leaving a supernatant liquid in which the gangue materials can be seen to be suspended. The effectively dispersed pulp is then conditioned with a collecting agent for the mineral using a quantity of reagent and a conditioning time determined experimentally for optimum results and the pH is adjusted to a value at which the mineral constituents of the pulp will flocculate in the presence of the collecting agent used. This value is usually on the acid side and is generally quite critical for any particular mineral or combination of minerals present in the ore. The pulp is conditioned at this new pH for a period sufficient to initiate flocculation of the mineral constituent, which flocculation may again be observed visually in a beaker sample. The pH of the pulp is then raised to within the optimum range of pHs for flotation of the mineral constituents concerned and the pulp is further conditioned for a predetermined period to bring about conditions of dispersion which will keep the gangue materials effectively dispersed with the obtaining of final heavy flocculation of the metallic oxide and hydrated metallic oxide constituents of the ore. The pulp is then subjected to flotation to produce a concentrate of the metallic oxides and hydrated metallic oxides.

The collecting agent used does not appear to be critical to the success of the process. Fatty-acid-type collectors have been found to be effective as have combinations of fatty acid and petroleum sulfonate fatty acid and fuel oil as well as combinations of fatty acid, petroleum sulfonate and fuel oil. Where fuel oil has been used in conjunction with the collectors a surprisingly small amount of 1 to 3 pounds per ton of ore

is required for optimum results. This is in contradistinction to reported processes employing fuel oil in conjunction with fatty acid where from a minimum of 7 pounds to upwards of 200 pounds per ton have been reported as having been necessary.

Where a dispersing agent is necessary I prefer to use sodium silicate which is the most readily available and economical of those commonly employed in flotation, however, conventional dispersants such as sodium hydroxide, lignen sulfonate and silicon tetrachloride can be used, as the general effectiveness of the process does not appear to depend upon the selection of a particular dispersing agent but rather upon the achieving of the correct degree of dispersion.

Where the initial dispersion stage is conducted in an alkaline circuit the use of an electrolyte under some conditions of operation has shown appreciable improvements in metallurgical results. The selection of the electrolyte does not appear important so long as it does not produce ions detrimental to flotation of the particular minerals present. Sodium fluoride, sodium sulfate and sodium chloride have all been successfully used although sodium chloride in some cases produces a froth condition which is less easily controlled.

The flotation circuit used may be of conventional design and will normally include an appropriate number of cleaning stages either without or with appropriate conditioning between some or each of the stages. In most cases the conditioning between cleaning stages will be for the purpose of producing conditions of additional dispersion employing controlled amounts of dispersing agent for this purpose. I have found surprisingly however, that in the later stages of cleaning some of the carbonates which tend to float in the process with the metallic oxides are effectively depressed by lowering the pH of the pulp to the acid side of the scale and conditioning with controlled amounts of sodium fluoride.

DESCRIPTION OF THE MANNER IN WHICH THE INVENTION MAY BE CARRIED OUT

The manner in which the various steps of my new process may be carried out can vary to a substantial extent and will depend in most instances upon the nature of the ore being treated and the equipment employed in the particular plant concerned for the preparation of the pulp. In plants where a wet grinding circuit precedes the flotation circuit it is, with some ores, possible to employ the grinding circuit to produce the effective dispersion necessary in the first step of the process. This may be accomplished by adding dispersing agent directly to the grinding circuit. With some ores which readily achieve effective dispersion the nature of the ore and of the water used may render the use of a dispersing agent unnecessary. In a batch-type laboratory mill it is possible on certain ores to add the dispersing agent and/or the collecting agent in a grinding circuit so that the mill product may be passed substantially directly to the initial flocculation stage of the process. What appears most important is that the pulp be in effectively dispersed condition when the collecting agent is added to it, regardless of whether this condition results from a natural condition of the ore and plant conditions so that the minerals which are released during grinding inherently have this characteristic or whether it is necessary to bring about this condition with a dispersing agent as already described.

For the flocculation stage in some cases where the nature of the ore and water used are such that a neutral or slightly acid pulp result from the dispersion stage of the process the addition of a fatty acid collector may alone be sufficient to lower the pH to within the optimum range for initial flocculation. In most cases however, the pH will be adjusted with an acid reagent. Any acid reagent which does not introduce undesirable ions into the pulp is suitable. I prefer sulfuric acid because of its relative cheapness and availability. Where an alkaline agent is required similar considerations apply. I prefer to use sodium silicate as this acts both as a dispersing agent and an alkaline agent, but sodium hydroxide and other chemicals conventionally used may be used in place of sodium silicate or together with it.

The initial pH to which the pulp is lowered for the initial flocculation stage depends upon the particular mineral or minerals in the ore and may be neutral or near neutral or in some cases as low as pH 5.0.

In raising the pH for purposes of carrying out the final preflotation conditioning stage an alkaline agent may be added. However, in many cases the ore itself contains acid-consuming constituents and in some cases these may be sufficient to return the pulp to near neutral or slightly alkaline. I have found that the preferable range for optimum flotation is from approximately 7.2 to approximately 8.5 although in certain cases a slightly lower or slightly higher pH can produce satisfactory results. With some ores it is most important in the final preflotation conditioning stage to control closely quantities of dispersing agent and the time of conditioning, the optimum quantity and time being different for each ore and requiring predetermination by experiment. With other ores the quantity of dispersing agent and the time of conditioning are more flexible.

Importance of conditioning Times:

As will be apparent, in the process of the invention great importance attaches to the order and concentration of reagent addition at the beginning or during each of the various cycles. The time period of each cycle is of prime importance and may vary within limited periods dependent on the reagent balance used, the condition of the pulp either during or following comminution, and the density of the pulp in the various cycles.

Considering it as a four-stage conditioning cycle, the grinding stage may in suitable cases be regarded as the first cycle. During the grinding stage we may have the following conditions:

Example (a) of Four-Stage Conditioning Cycle:

Sufficient dispersion of the pulp may result due to the characteristics of both the solution used and the soluble salts in the ore, and the pH of the pulp both during and after comminution. In such a case, no reagent addition is necessary to the grinding stage. The second stage consists of the addition of the collecting, and if required, modifying agent such as fuel oil wherein the pH may be either on the alkaline or acid side. Alternately, it may be necessary to adjust the pH, either alkaline or acid, with reagents that do not affect the floatability characteristics of the metallic oxides, or alternately, the final depression effect on the waste host rock materials. The acid reagent normally used is sulfuric acid, and the alkaline reagent, sodium hydroxide. These reagents are comparatively cheap and normally readily available. This second stage of conditioning in the four-cycle circuit has been illustrated as between a minimum of 4 minutes to a maximum of 16 minutes with 8 minutes being generally optimum. The pH of this second stage is in the range of a maximum of 8.9 to a minimum pH of 6.5. The optimum is usually in the range of 6.8 to 8.2.

The third-stage conditioning is the lowering of the pH, normally with the use of sulfuric acid. Where the initial pH is towards the high end of the alkaline range, the pH for this third stage need only be lowered to close to neutral. Where the first stage is near the middle of the range the pH may be lowered to as low as 5.0. Where the pH is lowered towards the lower end of the acid range, normally acid-consuming constituents in the ore will bring the pH back towards neutral by the end of the conditioning cycle. The time period of this cycle is normally quite critical and is in the range of a minimum of 8 minutes to a maximum of 16 minutes with the optimum being approximately 12 minutes.

The fourth cycle consists in raising the pH either to slightly acid, i.e., a pH of approximately 6.7—6.95, or to as high as a pH of 8.5. This may be accomplished by the use of an alkaline agent such as sodium hydroxide, or alternately, with an alkaline-dispersing agent such as sodium silicate, or alternately, with the combined use of an alkaline agent such as sodium hydroxide and sodium silicate, although depressing agents other than sodium silicate may be used, such as silicon tetrachloride, lignen sulfonate or the like. Where sodium silicate is used comparatively large concentrations may be required. The amount of sodium silicate necessary may be as

high as 15 pounds per ton. The time cycle required for this stage is critical for the type or ore, the density of the pulp and the combination of reagents used in the previous three cycles. It may vary from a minimum of 4 minutes to a maximum of 24 minutes. The normal optimum time is 10—20 minutes.

Example (b) of Four-Stage Conditioning Cycle:

On some ores, to obtain optimum results it may be necessary to add a dispersing agent either to the grinding circuit, or alternately, following the grinding circuit. Where it is necessary to add the dispersing agent, in each case the other three cycles remain the same. Where the dispersing agent is added to the grinding circuit, or alternately, following the grinding circuit, the amount used is within a critical range and must be closely controlled. Where it is added following the grinding stage, the conditioning time differs for different conditions of operation such as pulp density and the pH, the minimum time required for optimum results has been found to be 3 minutes with a maximum of 15 minutes. The optimum time has been found to be approximately 10 minutes. Where it is added following the grinding stage we can consider, with the grinding stage forming the first cycle, that we now have five cycles instead of four. Where the dispersing agent is added to the grinding circuit it is normally unnecessary to follow with a conditioning cycle prior to the addition of the collector.

Example (c) of Four-Stage Conditioning Cycle:

In some cases the collector and/or modifying agent may be added to the grinding circuit with or without the dispersing agent. The optimum condition is normally with the addition of both dispersing agent and collector, rather than the collector alone. Where the collecting and/or modifying agent is added to the grinding or No. 1 cycle, normally a short conditioning period of up to 8 minutes must still be required in the second cycle, although this cycle with some ores and operating conditions may be eliminated and the pH adjusted to what would normally be the third stage of conditioning. This time of conditioning is normally a maximum of 16 minutes.

Example (d) of Four-Stage Conditioning Cycle:

There is an exceptional condition, wherein in the grinding circuit, optimum conditions of dispersal are achieved, and the surfaces of the minerals remain in readily activated condition. Considering the comminution stage as Cycle 1 and no reagent added to the grinding circuit other than the dissolved reagents that are returned in the mill solution water, the second conditioning stage can be eliminated as a separate stage. In such as case the collector and/or modifying agents are added following the grinding circuit and the pH adjusted to the optimum either before or after the addition of the collecting and/or modifying agent. The conditioning time under such conditions will vary between 4 minutes and 20 minutes with the optimum being normally in the range of 8 to 16 minutes. The final cycle of dispersion may be carried out at the end pH of the pulp, or alternately, at a raised pH as noted in the previous examples. Again, for each condition of pH, density of the pulp and characteristics of the ore, the concentration of the dispersing agent and the time-cycle are critical. The minimum time is normally 4 minutes and the maximum 25 minutes with the optimum being in the range of 10—15 minutes. The prolonged period of conditioning is normally used where the dispersing agent is added over various periods of time in the time-cycle period. The shorter time of the conditioning cycle may be used where the reagent is all added at the beginning of the cycle or, say, in two stages. In the latter case the optimum period is normally in the range of 10—15 minutes.

EXAMPLES OF LABORATORY TESTS EMPLOYING THE PROCESS OF THE INVENTION

My invention is illustrated by the following examples of direct flotation of iron ores.

EXAMPLE I

In this example a number of tests were made on an iron ore in which approximately 70 percent of the iron was present in

the form of magnetite and approximately 30 percent in the form of specular hematite. The ore also contained varying minor amounts of iron silicates. Samples of this ore were prepared in a laboratory rod mill and then subject to conditioning and flotation in accordance with the process of the invention in a Fagergren flotation cell. The following two tests, number 513 and 517, give a comparative illustration of the importance of obtaining effective dispersion during the initial stage of the process. In these tests the sodium silicate was added to the grinding stage followed by addition of the same amount of fatty acid and fuel oil in each case with conditioning. Sulfuric acid was then used to bring the pH down to approximately 6.2 and after a period of conditioning the pulp due to acid-consuming constituents therein had returned to a pH of approximately 6.8. The flotation was carried out in this slightly acid circuit.

In test 513, 2.77 pounds per ton of sodium silicate was added to the grinding circuit and the rougher tailing produced was 9.7 percent by weight analyzing 5.21 percent iron. In test 517, where 3.89 pounds per ton of sodium silicate was added the rough tailing was 15 percent by weight analyzing only 4.97 percent iron, illustrating a greater rejection of waste host rock and also better activation of the iron minerals as shown by the lower tailings iron analysis. In test 518 a further increase in the sodium silicate to 4.4 pounds per ton only slightly increased the tailings rejection to 16.8 percent by weight but the iron flotation was seriously affected with the tailings analysis increasing to 7.93 percent iron.

The foregoing tests indicate that in the case of the ore tested the quantity of dispersing agent is a critical factor in obtaining effective dispersion of the pulp permitting effective tailings rejection in subsequent flotation.

EXAMPLE II

A sample of ore from the same area as that used in example I containing 68 percent of the iron as specular hematite, 30 percent as magnetite and 2 percent as iron silicate was brought to a state of effective dispersion by grinding for 18 minutes in a laboratory rod mill at a pulp density of 60 percent solids in the presence of 3.9 pounds per ton of sodium silicate as dispersing agent and 0.275 pounds per ton of sodium fluoride was added as an electrolyte. The pulp was then conditioned for 8 minutes with 1.04 pounds per ton of fatty acid, 1.53 pounds per ton of petroleum sulfonate and 2.16 pounds per ton of fuel oil as collecting agent. The pH was then lowered to slightly on the acid side by the addition of 2.14 pounds per ton of sulfuric acid and the pulp subjected to 16 minutes of conditioning to initiate flocculation of the mineral constituents. The pH of the pulp was then raised to within the optimum range for flotation by the addition of 2.77 pounds per ton of sodium silicate with a further conditioning time of 6 minutes.

The thus conditioned pulp was then subjected to flotation employing five stages of cleaning with 10 minutes of conditioning after the third stage with the addition of 0.42 pounds per ton of sodium fluoride and 6.9 pounds per ton of sulfuric acid to drop the carbonates. The resulting concentrate was 44.8 percent of the heads by weight, contained 63.53 percent iron representing a recovery of 81.57 percent. The acid soluble iron in the tailings analyzed 1.29 percent iron.

EXAMPLE III

Another sample of the same ore as that used in example II was subjected to the same conditioning procedure followed by flotation with the difference that instead of conditioning with sodium fluoride and sulfuric acid after the third cleaner the pulp was conditioned for 5 minutes after the second cleaner with 0.42 pounds per ton of sodium fluoride and 6.9 pounds per ton of sulfuric acid and after the third cleaner the pulp was conditioned for 8 minutes with 0.28 pounds per ton of sodium fluoride and an additional 6.9 pounds per ton of sulfuric acid. The concentrate produced was 41.8 percent by weight of the

head sample and contained 64.92 percent iron with a recovery of 75.96 percent of the total iron.

Comparing the metallurgical results in this example with those in example II it will be apparent that the more drastic use of sodium fluoride dipping more deeply into the acid side has resulted in an improvement of the concentrate grade by depressing more carbonates and combined particles. The head sample of the ore analyzed 1.40 percent CaO and 1.76 percent MgO with siliceous material being predominant in the gangue. The concentrate analyzed 0.75 percent CaO, 0.79 percent MgO and 3.93 percent SiO₂. Thus the use of sodium fluoride at a low pH has not only effectively depressed the silica as would be expected but has in addition surprisingly depressed the calcium and magnesium carbonates. It is also evident that the iron minerals once floated can withstand this low pH of initially about 4.0 to 4.5 without their floatability being adversely affected.

The reagent balance employed in this example approached the optimum for the particular ore concerned. Both the quantity of dispersing agent and the time of conditioning in the final conditioning stage proved to be quite critical. The use of lesser quantities of sodium silicate than that indicated produced less efficient tailings rejection in the rougher tailing while the use of higher quantities resulted in depression of the iron minerals and thus a higher loss of values in the rougher concentrate. Shorter conditioning times tended to produce lower grade and recovery in the concentrate whereas longer conditioning times had an adverse effect upon tailings rejection and tended to depress the iron minerals producing a bad effect both upon the recovery and the grade in the final concentrate.

In this example the fuel oil appears to act as a modifier and it proved necessary closely to control the quantity. Amounts greater than about 3½ pounds per ton tended to depress the iron minerals whereas quantities smaller than 1 pound per ton, did not produce any measurable improvement over cases where fuel oil was not used at all.

The fatty acid may be used in moderately higher amounts than that indicated without any deleterious result, however, when the amount used is decreased the effect of "starvation" is surprisingly a drop in recovery accompanied by a drop in grade. This is in contravention to conventional flotation where normally starvation of the pulp with respect to collecting agent produces an increase in the grade accompanied by a drop in the recovery. The petroleum sulfonate appears to act as a substitute for fatty acid and equivalent results can be achieved by reducing the fatty acid and increasing the petroleum sulfonate. This substitution is desirable mainly because of the relatively low cost of petroleum sulfonate compared to that of fatty acid. The extent to which this substitution may safely be made without adversely affecting the metallurgy has not yet been determined.

The effect of the initial pH during the initial flocculation stage is quite marked. Too low an initial pH results in poor activation of the metallic oxides wherein during the cleaning stages these minerals tend to drop too rapidly resulting in low recovery and low-grade concentrates, while too high an initial pH results in depression of the metallic oxides.

In this example the initial pH of the initial flocculation stage was approximately 6.45 rising at the end to 6.6. During the final conditioning stage the pH was at 7.35 which was the pH at which initial flotation was carried out. Departure up or down from this last pH was found to have a deleterious effect, giving a lower grade concentrate when lower and a reduction in both recovery and grade when appreciably higher.

EXAMPLE IV

In this example and in example V the ore tested was an iron ore in which the mineral constituent was principally martite with approximately 11 percent of the total iron as magnetite, a minor amount of hematite, and up to about 5 percent in the form of iron silicate.

A sample of the ore (test 118) was ground for 15 minutes in a laboratory ball mill at 60 percent solids in the presence of 4.7 pounds per ton of sodium silicate to produce a pulp in which the ore was effectively dispersed. 2.2 pounds per ton of fatty acid (Acintol, a product of the Arizona Chemical Company) was added and the pulp was conditioned for 10 minutes to activate the mineral constituents thereof. Then 1.33 pounds per ton of sulfuric acid was added in four equivalent portions over 16 minutes of conditioning time at 4 minute intervals. The initial pH before the addition of the sulfuric acid was 7.7 and thereafter the pH after each addition of sulfuric acid was 7.4, 7.1, 7.0 and 6.8. The final pH of the pulp after 16 minutes of conditioning was 6.9 at which time the mineral constituents were effectively flocculated.

Then 1.83 pounds per ton of sodium silicate was added bringing the pH to 7.1. The pulp was conditioned for 9 minutes with the addition of one-half pound per ton of sodium silicate after 3 minutes and another one-half pound after 6 minutes. The final pH after 9 minutes of conditioning was 7.25 and the pulp was differentially flocculated in that the gangue materials were effectively dispersed and the desired mineral constituents were flocculated. The pulp was then subjected to flotation in a Fagergren flotation cell with four stages of cleaning and the addition of one-half pound per ton of sodium silicate between the first, second and third cleaners and one-fourth pound per ton between the third and fourth cleaners.

The flotation resulted in a concentrate having a grade of 65.71 percent iron at a recovery of 74.80 percent in the open circuit. The rougher tailing combined with the first cleaner tailing represented 35.36 percent by weight and contained 5.38 percent of the total iron. The cleaner concentrate analyzed 4.07 percent SiO_2 , 0.12 percent CaO and 0.38 percent MgO . On an ore of this type where no previous desliming had been carried out the metallurgy is to be considered as outstanding.

EXAMPLE V

A sample of the same ore as that used in example IV was employed in test no. 123. In this case the conditions and procedure were the same as those of example IV except that the grinding time was 22 minutes and instead of 2.2 pounds per ton of fatty acid as a collecting agent there was used 0.79 pounds per ton of fatty acid together with 2.78 pounds per ton of petroleum sulfonate (899 American Cyanamid) following the activation cycle the pulp was subjected to the same acid flocculation cycle and differential dispersion and flocculation cycle as in example IV.

The flotation produced a concentrate assaying 65.96 percent iron at a recovery of 74.12 percent in the open circuit. The rougher tailing combined with the first cleaner tailing was 32.66 percent by weight containing 5.81 percent of the total iron content.

This test and that described in Example IV approached the optimum metallurgy for this particular ore. This ore was characterized by being difficult to disperse effectively, it having been found advantageous to add the dispersing agent to the mill in order to avoid long conditioning times to achieve dispersion. The quantity of dispersing agent employed was quite critical, larger quantities than that indicated tending to depress the mineral constituents and lower quantities resulting in poor activation of the iron minerals.

The ore was further characterized by the fact that under the conditions of operation effective activation could not be achieved until the ore had been brought into an effectively dispersed condition. Consequently the collecting agent when added to the mill along with the dispersing agent resulted in substantially poorer metallurgy with higher tailings losses, lower grade concentrate and a lower recovery. Similarly where the dispersing agent and the collecting agent were both added together in a conditioning stage following grinding the same poor results were obtained. The initial pH during the differential dispersion and flocculation cycle was shown to be

critical and where the initial pH was dropped lower than that indicated both grade and recovery suffered. With this ore the quantities of fatty acid and petroleum sulfonate were found to be optimum at the values indicated but minor variations in quantity either up or down did not seriously affect the metallurgy achieved.

EXAMPLE VI

10 Ore Description

This ore had a head value of approximately 32 percent iron with approximately 66 percent of the iron minerals present as hematite and 34 percent present as goethite.

The grinding cycle of the testing program in all cases was carried out in a laboratory rod mill.

At a grinding time of 28 minutes and 60 percent solids the following was the screen analysis of the product produced.

	Percent weight
20 +325 mesh-----	11.45
-325+400 mesh-----	4.20
-400+500 mesh-----	20.10
-500 mesh-----	64.25
Total-----	100.00

25 Effect of Conditioning Time in the Second Cycle of the Four-Cycle Circuit

The grinding time in tests 153, 154 and 158 was 32 minutes using 8.3 pounds of sodium silicate per long ton of ore added at the beginning of the grinding cycle. In the terminology used the grinding cycle would correspond to the No. 1 cycle in the effective dispersion of the ground pulp.

The collector-modifier reagents to the second cycle were in all cases 1.04 pounds Acintol FA2, 1.53 pounds 899 and 3.0 pounds of fuel oil respectively per long ton of solids.

The third stage, in all cases, consisted of stage addition of 1.08 pounds sulfuric acid per ton and a conditioning period of 16 minutes.

The fourth stage used 5.56 pounds of sodium silicate per ton, using 8 minutes conditioning in 153 and 154, and 6 minutes in 158.

All test used five cleaner stages following the rougher float.

The pH by the beginning of stage 2 was 8.45. The lowest pH recorded in stage 3 was 6.85 and ended at pH 7.0.

The pH in stage 4 was 8.45.

In test 158 with 8 minutes conditioning in the second stage, the concentrate grade was 6.94 percent iron, containing approximately 73 percent of the total iron. In test 153 with 12 minutes conditioning in the second stage, the concentrate grade was 61.75 percent iron, containing approximately 74 percent of the total iron. In test 154 with 16 minutes conditioning in the second stage, the concentrate grade was 58.56 percent iron, containing approximately 85 percent of the total iron.

For effective cleaning of the rougher concentrate there was little difference between the 8 minutes and 12 minutes in this activation stage. However, the curve broke between 12 minutes and 16 minutes with the activation extending to a higher range of the iron particles together with part of the host rock materials.

EXAMPLE VII

Effect of Excessive Dispersion in Final Cycle

In comparative tests 195 and 205 the reagents and conditioning times were the same with the exception of the amount of sodium silicate used in the last cycle of the four cycle circuit. The grinding time was 28 minutes with 8.3 pounds of sodium silicate and 0.7 pounds of sodium sulfate respectively per ton of ore added to the beginning of the grinding cycle. Stage 2 consisted of 10 minutes conditioning using 1.83 pounds fatty acid (Acintol FA2,) 1.11 pounds of petroleum sulfonate (899) and 3 pounds of fuel oil respectively per ton of solids. Stage No. 3 used 16 minutes conditioning with stage addition of 1.94 pounds sulfuric acid per ton. Stage 4 con-

sisted of 8 minutes conditioning with half of the sodium silicate added at the beginning and the other half added at the end of 2 minutes conditioning for a total of 8 minutes conditioning time. In test 195, 6.66 pounds of sodium silicate per ton was used and in test 205 7.77 pounds sodium silicate per ton was used.

In test 195 the rejection in the rougher and first cleaner tailings was 42.8 percent by weight, analyzing 5.76 percent iron. In test 205 the rougher tailings rejection alone was 34.4 percent by weight, analyzing 6.13 percent iron, showing a higher iron loss at a lower percent rejection and illustrating the detrimental effect of excess dispersion in this cycle.

EXAMPLE VIII

The Effect of the Use of a Wetting Agent in the Grinding Circuit

In tests 229 and 230 the grinding cycle was 45 minutes and in test 229 Dow Benax 2A-1 which is described as a wetting agent was added at the beginning of the grinding cycle using 0.10 pounds per ton of solids. In test 230 no reagent addition was made to the grinding cycle, depending entirely upon the natural dispersion of the pulp in both cases. The second cycle in both cases consisted of 8 minutes conditioning using 2.62 pounds of fatty acid (Acintol FA2) 1.11 pounds of petroleum sulfonate (899) and 3 pounds of fuel oil respectively per ton. The third cycle in both cases was 16 minutes using stage addition of 1.94 pounds per ton of sulfuric acid. In addition, in test 229 sodium fluoride was added at the beginning of this cycle at the rate of 0.84 pounds per ton. Stage 4 consisted of 11 minutes conditioning in both cases with stage addition of 9.15 pounds per ton of sodium silicate. In test 229 the tailings rejection was 36.5 percent by weight analyzing 3.78 percent iron while in test 230 the tailings rejection was 36.4 percent by weight analyzing 6.29 percent iron.

Previous testing had indicated that the use of the electrolyte in the acid circuit had very little effect on the iron activation, indicating that the use of a wetting agent in the grinding circuit where no dispersal agent was used resulted in a surprising increase in the iron activation and in the host rock depression.

EXAMPLE IX

Effect of Using Two Stages only Following the Grinding Stage

In test 243 a 28 minute grinding cycle was used with no added reagents. In the cycle following grinding 20 minutes conditioning was used with the addition of 3.66 pounds of fatty acid (Acintol FA2) and 3 pounds of fuel oil respectively per ton. The water pH was 7.05, and 5 minutes after the addition of the reagents the pH of the pulp was 6.8. At the end of the 20 minutes conditioning cycle the pH was 7.3. Following this conditioning stage 2.78 pounds of sodium silicate per ton was added, bringing the pH to 8.1 and the conditioning time was 8 minutes.

After six cleaners the concentrate grade was 60.7 percent iron at a recovery of 79.8 percent.

In comparing this test to test 254 wherein the grinding time was 28 minutes without reagent addition, the second stage was 8 minutes with the same fatty acid and fuel oil addition followed by the normal third stage using 0.7 pounds per ton of sulfuric acid and the fourth stage using a 21 minute conditioning cycle and 5 pounds per ton of sodium silicate. After six cleaners the concentrate grade was 61.1 percent iron with 91 percent of the total iron in the concentrate showing a marked improvement in metallurgy over the two-cycle stage following grinding. However, considering that in test 243 further research, particularly with the last cycle, could improve these results on some ores and under certain plant conditions of water supply and soluble salts, it could result in acceptable metallurgy.

EXAMPLE X

The Use of Sodium Hydroxide for Controlled pH and Final Dispersal of the Pulp

In test 244 a 28 minute grind was employed without any reagent addition to the grinding stage. The cycle following grinding was 8 minutes with the addition of 3.66 pounds of fatty acid (Acintol FA2) and 3 pounds of fuel oil respectively per ton. The pH after the addition of the reagents was 6.8. The next cycle was 12 minutes conditioning with the stage addition of 1.67 pounds of sulfuric acid. The lowest pH recorded was 6.00. The next cycle consisted of 7 minutes conditioning with the addition of 0.833 pounds of sodium hydroxide per ton. The pH at the end of this cycle was 8.05. Seven cleaners were employed following the rougher float resulting in a final concentrate analyzing 61.41 percent iron and containing 88.3 percent of the total iron, showing excellent metallurgy in the open circuit work. However, the froth was on the tough side and without a further modifier could result in a problem in plant practice in final grade control.

EXAMPLE XI

The following test used the four-cycle circuit wherein an ultrafine grind was used with a grinding cycle time of 45 minutes at 60 percent solids with no reagent added to the grinding cycle. The second stage was 8 minutes conditioning with 3.66 pounds of Acintol FA2 and 3 pounds of fuel oil respectively per ton of solids. The pH during this cycle was 6.8. The third cycle was of 12 minutes duration and used 1.11 pounds of sulfuric acid per ton in stage addition. The lowest pH recorded was 6.05. The fourth stage was 7 minutes conditioning with stage addition of 8.88 pounds sodium silicate per ton. The following metallurgy was attained:

TEST 235

Product	Percent weight	Analysis, percent total Fe	Percent Fe distribution
Cleaner concentrate.....	42.8	62.15	82.1
No. 6 cleaner tails.....	3.2	49.51	4.7
No. 5 cleaner tails.....	2.1	32.95	2.1
No. 4 cleaner tails.....	2.8	26.44	2.3
No. 3 cleaner tails.....	4.4	18.49	2.5
No. 2 cleaner tails.....	6.9	10.29	2.2
No. 1 cleaner tails.....	12.4	5.47	2.1
Rougher tails.....	25.4	2.59	2.0
Total.....	100.0		100.0

NOTE: Calculated heads=32.53% total Fe.

It will be noted that a cleaner concentrate grade of 62.15 percent iron was obtained with a recovery of approximately 82 percent of the iron in this concentrate with open circuit. The loss of ignition of this concentrate after pelletizing and due to the goethite would be approximately 3 percent, which would mean after loss of ignition this concentrate would increase in grade to approximately 64 percent iron. Ahead of the fifth and sixth cleaners the concentrate was conditioned for short periods with the addition of lignen sulfonate for additional rejection of middling and host rock minerals.

EXAMPLE XII

An appreciably coarser grind was used in this test than in example XI as the grinding period was only 28 minutes. No reagents were added to the grinding circuit and the second cycle of fatty acid (Acintol FA2) and fuel oil addition was identical to example XI. In the third cycle the conditioning time was 12 minutes as in example XI. However, sulfuric acid was reduced to 0.7 pounds per ton. The major change was in the fourth cycle in which the conditioning time was increased to 21 minutes and the sodium silicate added in stages for a total of 5 pounds per ton as against the larger amount used in example XI for the shorter conditioning period of 7 minutes. The rougher concentrate was water cleaned only using seven cleaners. It will be noted that the final concentrate analyzed 61.67 percent higher for the recovery of 89½ percent of the total iron in the concentrate. With the loss of ignition of approximately 3 percent on this concentrate after pelletizing this concentrate grade would increase to approximately 63.6 percent iron, which is an outstanding grade for the recovery in-

volved in the open circuit test. It will further be noted that in test 235 the original tailings rejection was 37.8 percent by weight carrying 4.1 percent of the total iron. In test 254 the same rougher tailings plus first cleaner rejection was 40.3 percent by weight, carrying only 3.4 percent of the total iron. This comparative metallurgy basically extending the time of the last conditioning cycle in test 254 with a smaller amount of the dispersing agent, that is, sodium silicate, illustrates the high importance of the time factor of this last cycle.

TEST 254

Product	Percent weight	Analysis, percent total Fe	Percent Fe distribution
Cleaner concentrate.....	46.6	61.67	89.5
No. 7 cleaner tails.....	1.2	40.84	1.5
No. 6 cleaner tails.....	0.9	33.65	1.0
No. 5 cleaner tails.....	1.3	28.89	1.2
No. 4 cleaner tails.....	1.7	18.56	1.0
No. 3 cleaner tails.....	2.9	13.72	1.2
No. 2 cleaner tails.....	5.1	7.67	1.2
No. 1 cleaner tails.....	12.1	4.44	1.7
Rougher tails.....	28.2	1.94	1.7
Total.....	100.0		100.0

NOTE: Calculated heads=32.10% total Fe.

EXAMPLE XIII

A sample of uranium ore from Blind River Ontario was ground for 14 minutes at 60 percent solids in a laboratory rod mill with the addition of 2.5 pounds per ton of sodium chloride as an electrolyte. The grind screen analysis was 60 percent minus 200 mesh. The ground ore was then placed in a 600 gram Fagergren laboratory flotation cell and the density was decreased to approximately 40 percent solids. The pH of the water was 7.15. 1.23 pounds per ton of sodium silicate was added and the pulp was conditioned for 10 minutes to achieve effective dispersion. 2.73 pounds per ton of fatty acid (Acintol FA2) and 3.9 pounds per ton of fuel oil were added and the pulp was conditioned for 20 minutes at the end of which time the pH was 6.95 and the pulp was in final condition for flotation and was floated.

The head value of the ore was 0.17 percent U_3O_8 and the weight of the tailings was 62.4 percent by weight analyzing 0.039 percent U_3O_8 for a tailings loss of 14.3 percent.

An identical test carried out using only 0.615 pounds per ton of sodium silicate produced a tailings weight of 78.5 percent analyzing 0.112 percent U_3O_8 for a tailings loss of 51.7 percent. In a third identical test 1.845 pounds per ton of sodium silicate was used producing a tailings weight of 84.1 percent analyzing 0.137 percent U_3O_8 for a tailings loss of 67.7 percent.

This testing indicated the criticality of the quantity of dispersing agent with respect to the achievement of effective dispersion of this ore, too much or too little dispersing agent resulting in a high tailings loss.

Application of the Process to Typical Plant Flow Sheets

The process of the invention may be applied to plant flow sheets in a number of different ways depending upon the comminution circuit which is used. The application of the process will be described in connection with four basically different comminution circuits.

Several examples of plan flow sheets are illustrated in the accompanying drawings wherein:

FIG. 1 is a block flow sheet illustrating a typical grinding circuit;

FIG. 2 is a block flow sheet illustrating another typical grinding circuit;

FIG. 3 is a block flow sheet of a further grinding circuit;

FIG. 4 illustrates still another comminution circuit; and

FIG. 5 is a block flow diagram illustrating the four conditioning cycles of the present invention together with a typical flotation circuit.

Description of Comminution Circuits

A. One comminution circuit is illustrated in FIG. 1 and consists of feeding the run of mine ore to a primary crusher 10 the

product 11 of which is fed to a wet autogenous mill 12, operating in closed circuit. Alternatively the wet autogenous mill may be replaced by a conventional rod mill, ball mill in closed circuit with its classifier in which case an appropriate number of additional stages of crushing would of course be necessary. The product 13 from the wet autogenous mill 12 or rod mill-ball mill circuit is fed to a classifier 14 from which the oversize 15 is returned to the inlet side of the ball mill circuit. The undersize 16 which will generally be a pulp containing from about 12 to 25 percent solids is treated with a flocculating agent and passed to a thickener 17 from which the overflow is passed to the plant water supply system 18. The underflow 19 from the thickener 17 which will be at between 50 and 70 percent solids serves as the feed for the flotation plant.

B. In a second type of comminution circuit (see FIG. 2), the run-of-mine ore is passed to a primary crusher 20 the product 21 of which is feed to a dry autogenous mill 22 which is either in open or closed circuit with a dry classifier, the product 23 of which is fed to a closed circuit ball mill 24 the product 25 of which is fed to a classifier 26 from which the oversize 27 is recirculated to the ball mill inlet. The undersize 28 from the classifier 26 which is at approximately 20 percent solids is treated with a flocculating agent and fed to a thickener 29 from which the overflow 30 is returned to the plant water supply and the underflow 31 which is in the form of a pulp containing from about 50 to 70 percent solids is used as the flotation plant feed.

C. As an alternative to B using a dry autogenous mill in either open or closed circuit for primary grinding the product of the dry autogenous mill 22 (see FIG. 3) may be fed to an open circuit ball mill 32. The output of the open circuit ball mill consists of a pulp containing approximately 50 to 70 percent solids and is used as the flotation plant feed. Alternatively with certain ores it is possible and in many cases advantageous to employ the open circuit ball mill for purposes of carrying out one or more of the stages of conditioning which would normally be carried out in the flotation plant:

Alternative 1

On certain ores it would be advantageous to add dispersing agent to the feed end of the ball mill and to maintain pH control arranged to maintain a controlled pH at the mill outlet. This alternative is advantageous in cases where the ore has a slime factor which is particularly deleterious to the flotation unless effectively dispersed in a controlled pH range prior to activation of the desired recoverable constituents.

Alternative 2

In certain cases it may be desirable to add both dispersing agent and a collecting agent to the feed end of the mill again while maintaining pH control. This procedure is advantageous in cases where activation of the desired mineral constituents can be obtained at the same time that dispersion of the waste host rock materials is taking place. This is generally so when the desired mineral constituents are not readily depressed by the dispersing agent within the pH range used, and where it is employed the conditioning cycles of effective dispersion, partial activation and partial flocculation occur within the ball mill during the grinding process to produce a grinding plant product which is already in partially activated and flocculated condition.

Alternative 3

In some cases where the nature of the ore and other conditions are such as to produce effective dispersion without the addition of dispersing agent it is advantageous merely to add collecting agent to the feed end of the ball mill and to maintain the appropriate pH control, which is preferably slightly on the acid side. Where this alternative procedure applies the ball mill product is again a conditioned partially activated and flocculated pulp.

D. Another alternative for ore preparation is illustrated in FIG. 4 where a dry closed circuit autogenous mill is used for primary grinding and the mill product is passed through a dry classifier to produce a fines fraction 44 consisting of approximately 50 percent by weight of the total material which is of a

size range suitable for flotation and an oversize fraction 45 consisting of approximately 50 percent by weight of the total material which is then fed to a secondary closed circuit ball mill 46, the product 47 of which is passed to a classifier 48 from which the oversize 49 is returned to the inlet side of the ball mill, and the undersize 50 which consists of a pulp containing about 50 percent by weight of the total primary mill product at approximately 20 percent solids is combined with the dry classifier fines to form product 51 which is passed to an agitator 52 to produce a grinding plant product at about 60 percent solids. Under some conditions this may be too high and dilution of the pulp down to 35 to 50 percent solids may be desirable.

Description of Conditioning and Flotation Circuits and Their Application to Flotation Plant Feed Prepared as Above

a. Where the flotation plant feed is prepared in a wet autogenous or conventional closed grinding circuit according to (A) above, it will contain the flocculating agent which was added ahead of the thickener and there are three general applications of the process of the invention, the selection of which will depend upon the nature of the flocculating agent which has been used and the nature of the ore and plant water supply.

I. Where the flocculating agent which has been used prior to the thickener is a persistent flocculating agent which forms a flock difficult to break up by mechanical means such as passage through a pump, it will be necessary to employ a dispersing agent and to condition the pulp to achieve effective dispersing thereof prior to the addition of a collector. This conditioning cycle will normally require the adjustment of the pH by the addition of sodium hydroxide or sulfuric acid to within the range of from about 7.0 to 8.9 with a conditioning time of from about 3 minutes to about 16 minutes in conditioning stage 50 (see FIG. 5). The pulp is then subjected to an activation cycle in conditioning stage 51 wherein the collecting agent is added and a further period of conditioning of from about 3 minutes to about 16 minutes is carried out at a pH of from about 6.7 to 8.7. The pH of the pulp will then be reduced if necessary to bring it within the range of from about pH 5.0 to 7.2 and a further period of conditioning in conditioning stage 52 of from about 8 minutes to about 16 minutes is carried out. Upon completion of the foregoing cycle the pH will be adjusted if necessary to bring it within the pH range of from about 6.5 to 8.5 with the addition of dispersing agent and the pulp is conditioned in conditioning stage 53 for a period of from about 3 minutes to about 25 minutes to bring about differential flocculation by dispersing the gangue materials while bringing about final heavy flocculation of the desired mineral constituents. The conditioning stages 50, 51, 52 and 53 may employ conventional flotation plant equipment and may each consist of several conditioning units.

The pulp is then subjected to flotation which is basically the same for all of the different conditioning procedures and consists of a rougher float 54 to produce a final rougher tailing 55 which is passed to the tailings pond 56 and a rougher concentrate 57 which may be followed by cleaners 58 which may contain up to seven cleaning stages with or without conditioning between some or all of the stages to produce a final concentrate 59 which is passed to further processing 60 such as thickeners prior to filtering or alternatively directly to filtering and combined cleaner tailings 61 which will normally be returned to the classifier 14 of mill 12 (see FIG. 1). In some cases the first or second cleaner tailing may be of sufficiently low value to be rejected with the rougher tailing as a final tailing.

II. Where the ore is such that the mineral constituents are not depressed within the concentration of dispersing agents required to effectively disperse the host rock, the first two cycles 50 and 51 described in (I) may be carried out together. In this case the dispersing agent and the collector will be added to the pulp for a combined dispersing

and activation cycle which will be carried out at a controlled pH of from about 6.7 to 8.9 for a period of from about 4 minutes to 16 minutes. This cycle will be followed by a flocculating cycle 52 in which the pH of the pulp is reduced if necessary by the addition of e.g. sulfuric acid to within the range of pH 5.0 to 7.2 and a period of conditioning from about 8 to about 16 minutes will be carried out to achieve heavy flocculation. Following the flocculation cycle the pH of the pulp will be adjusted if necessary with, e.g. sodium hydroxide to bring it within the range of from about 6.5 to about 8.9 and conditioning stage 53 is carried out for a period of from about 3 to 25 minutes with or without the addition of additional dispersing agent to bring about differential flocculation by effective dispersion of the gangue materials with final heavy flocculation of the desired mineral constituents. This last cycle is then followed with flotation similar to that described in (I).

(III) Where the flocculating agent used in advance of the thickener is a nonpersistent flocculating agent which produces flocks which are readily broken down by mechanical agitation such as a guar gum in slightly acid circuit, the pulp by the time it arrives at the flotation plant may be in effectively dispersed condition. In this case the pulp will be passed directly from the grinding plant 61 to an activation stage 52 where with the pH adjusted if necessary to a pH range of from about 5.0 to about 7.2 the pulp is subjected to from about 4 minutes to 20 minutes conditioning in the presence of the collecting agent. In this case the activation and flocculation stages may be combined, since the addition of collecting agent will tend initially to lower the pH while acid-consuming constituents in the ore will tend to slightly raise the pH as the conditioning proceeds. Thus, at the end of the conditioning period the mineral constituents will be in an effectively flocculated condition. The pH of the pulp will then be adjusted if necessary to within the range of from about 7 to about 8.9 with the use of an alkaline agent and the pulp will be conditioned in a conditioning stage 53 to bring about partial dispersion in the presence of a dispersing agent. In some cases the dispersing agent may also be used as an alkaline agent, e.g. in the case of sodium silicate alone, sodium hydroxide alone or sodium silicate and sodium hydroxide. The pulp is then subjected to flotation which will suitably be of the same type as described in (I).

b. Where the flotation plant feed is prepared with a grinding circuit as described in B, the flotation plant feed will contain the flocculating agent added prior to the thickener 29. Conditioning procedures I, II or III will be applied to this material in accordance with the same principles as those outlined in (a).

It should be pointed out that in some cases it may be possible to operate the thickeners referred to in (A) and in (B) without the use of flocculating agents in which case partial flocculation of the material takes place in the thickener due to the conditions of the mill solution and the otherwise normal or adjusted pH in the thickener. In such a case the particular plant may find it more economic to eliminate the use of the flocculating agent and take a comparatively small loss in the valued mineral constituents that will occur in the overflow in the thickener overflows which in this case would be taken to the tailings pond or a tailings thickener where it would be flocculated probably with the plant tailings and sent directly to waste. The flotation plant feed produced by thickening in the foregoing manner would be treated by any one of (I), (II) or (III) depending upon the degree of flocculation and the strength of the flocs initially formed.

c. Where the flotation plant feed is prepared by a dry autogenous mill followed by an open circuit ball mill as in C above in the first instance where no reagents are used in the open circuit ball mill, the conditioning procedure followed will be (I), (II) or (III) depending upon the condition of dispersion and flocculation which obtains in the flotation plant feed on its arrival.

Where alternative 1 has been applied and the dispersing agent added with the feed to the ball mill under controlled pH

the flotation plant feed will arrive in effectively dispersed condition and depending upon the nature of the ore, procedure (III) may be used (where the concentration of dispersant in the pulp at the pH concerned does not interfere with flocculation of the mineral constituents).

Where alternative ore preparation procedure 2 is used with the ball mill in open circuit and both a dispersing agent and a collecting agent are fed to the ball mill while maintaining pH control the flotation plant feed will arrive at the flotation plant in flocculation condition, the conditioning stages of effective dispersion, activation and flocculation (i.e. 50, 51 and 52) having been accomplished during the progress of the material through the open circuit ball mill. In this case, the flotation circuit feed arriving from grinding plant 61 will be subjected directly to a stage of conditioning 53 in the presence of a dispersing agent, preferably sodium silicate, for a period of from about 3 to about 25 minutes at a pH within the range of from about 6.7 to about 8.5 for purposes of bringing about differential flocculation wherein the gangue materials are effectively dispersed while the mineral constituents are effectively maintained in flocculation condition. The pulp is thereafter subjected to a flotation 54 with subsequent stages as described in (I).

Where the feed to the flotation plant has been prepared in accordance with alternative 3 and only collecting agent has been added to the ball mill fed under pH control the flotation plant feed will also arrive at the flotation plant under conditions of flocculation. Once again the conditioning cycles of effective dispersion, activation, and flocculation will have been accomplished in the ball mill where slightly acid conditions will have prevailed. In this instance the flotation plant feed will again be subjected directly to a stage of conditioning 53 to bring about differential flocculation by maintaining the gangue effectively dispersed with final heavy flocculation of the desired mineral constituents. However, conditions may be such that it may not be necessary to add a specific dispersing agent since the conditions already present in the pulp may permit the desired effect to be achieved at the initial pH of the pulp. It is unlikely however that such conditions will prevail over any substantial period of operation in any particular plant because of variations in the nature of the ore and in the water supply. Consequently it will normally be desirable to add sufficient alkaline agent to elevate the pH to the appropriate value (the preferred range being from about 7.2 to 8.5) and it will usually be desirable either that the alkaline agent used by itself be a dispersing agent, e.g. sodium silicate or sodium hydroxide or to add a controlled amount of dispersing agent as well as an alkaline agent. Following conditioning in the above controlled conditions for a period of from about 3 to 25 minutes the pulp will be subjected to flotation 54 and the subsequent stages described in (I). d. Where the flotation plant feed is prepared in accordance with the comminution procedure described in C above, the flotation plant feed will arrive at the flotation plant without any reagents having been added to it during the comminution cycle with the exception of dissolved reagents in the normal mill water supply. In this case depending upon the nature of the ore and of the mill water supply any one of procedures (I), (II) or (III) would be appropriate.

The above-described comminution procedures are the most commonly met with in plants where the present invention is likely to find application. It will be apparent however that the process of the invention involving the four cycles of effective dispersion, activation, flocculation and differential dispersion and flocculation is readily adaptable to other plant flow sheets with which it may be integrated where conditions permit with the comminution circuit employed. It is immaterial to the effectiveness of carrying out of the process whether one or more of the conditioning cycles are carried out within the grinding circuit or whether all four are carried out externally to the grinding circuit in the flotation plant. Generally speaking savings in plant equipment and operating costs are achieved by integrating the conditioning process into the comminution circuit to the extent that it is possible to do so under any given conditions with any particular ore.

In addition to cases where the invention is applied to the product of a grinding plant there are many instances where it may be applied to the treatment of materials which are already in comminuted or naturally finely divided condition e.g. tailings piles or slimes from an existing plant that uses desliming procedure ahead of the normal recovery plant as well as in the case of some beach sands and natural occurring clays containing desired mineral constituents such as vanadium oxides. In such cases the pulp formed from the material to be treated will usually be similar in condition to one the various pulps produced by the grinding circuits already described, and the conditioning procedures to be employed according to the invention will be the same for pulps of similar type.

In the foregoing description of the application of the invention the various optimum conditioning times and reagent additions are based on the pulp being at approximately 40 percent solids, at temperatures of from 7°C to 21°C with high impeller speeds in the conditioning cycles. Increases or decreases in the pulp density in one or more of the conditioning cycles and increases or decreases of the conditioner impeller speeds, will affect the optimum time and reagent balance in any particular case. Furthermore temperatures outside the above range can be expected to have an effect upon the optimum times of the various conditioning cycles and upon the optimum reagent balance. The operating conditions employed throughout the test work reflected in the examples are generally representative or normal operating plant conditions and no difficulty should be experienced in adapting the process of the invention to conditions of temperature, plant water conditions and different forms of conditioning equipment which are within the range of what is generally accepted as normal plant practice. Changes in operating conditions may bring about changes in the optimum procedure according to the invention and it is desirable according to the invention, in plant practice to control the conditions so as to avoid major fluctuations within the circuit. Water temperature, pulp density and the pH in each conditioning cycle may be automatically controlled within the effective limits required for optimum plant operation employing conventional plant control devices.

40 What I claim as my invention is:

1. A process for the flotation of metallic oxides and metallic oxides with water of crystallization in chemical combination occurring in ores as finely disseminated minerals requiring fine grinding for the liberation of said minerals from essentially undeslimed partially dispersed pulps thereof comprising: conditioning a pulp of an ore containing one or more of said minerals in a state of subdivision where the mineral content thereof is substantially liberated with a predetermined amount of a collecting agent for the desired mineral values thereof at a substantially neutral pH; then at a pulp pH within the range of from substantially neutral to about 5.0, conditioning the pulp until substantial flocculation of said mineral values has occurred; then in a final conditioning stage at a pH of from about 6.5 to about 8.9 conditioning the pulp with the addition of a dispersing agent to bring about effective dispersion of the gangue materials while the mineral values remain flocculated; and then subjecting the thus conditioned pulp to flotation to produce a concentrate of the mineral values therein.

2. A process as defined in claim 1 in which the pulp initially contains a controlled amount, predetermined by experiment, of an electrolyte which is free of ions deleterious to flotation.

3. A process as defined in claim 2 wherein the electrolyte is sodium sulfate.

4. A process as defined in claim 2 wherein the electrolyte is sodium chloride.

5. A process as defined in claim 2 wherein the electrolyte is sodium fluoride.

6. A process as defined in claim 1 wherein the pulp initially contains a controlled amount, predetermined by experiment, of a wetting agent which is free of ions deleterious to flotation.

7. A process as defined in claim 1 wherein the pulp initially contains a controlled amount, predetermined by experiment, of a dispersing agent.

8. A process as defined in claim 7 wherein the dispersing agent is sodium silicate.

9. A process as defined in claim 1 wherein the mineral values are iron.

10. A process as defined in claim 1 wherein the mineral values are uranium.

11. A process as defined in claim 1 wherein the mineral values are iron and the flocculation stage of conditioning is carried out at an initial pH, predetermined by experiment, of from about 6.2 to about 7.2 for a period of approximately 12 minutes.

12. A process as defined in claim 1 wherein the final stage of conditioning is carried out at a controlled pH predetermined by experiment, within the range of from about 7.2 to about 8.5 in the presence of a controlled amount, predetermined by experiment, of sodium silicate for a period predetermined by experiment of from about 3 to about 25 minutes.

13. A process as defined in claim 1 wherein the mineral values are iron and the final stage of conditioning is carried out at a controlled pH, predetermined by experiment, of from about 6.7 to 8.5 in the presence of a controlled amount, predetermined by experiment of a dispersing agent for a period, predetermined by experiment of from about 4 to about 24 minutes.

14. A process for the flotation of metallic oxides, and metallic oxides with water of crystallization in chemical combination occurring in ores as finely disseminated minerals requiring fine grinding for liberation of said minerals from essentially undeslimed pulps thereof, comprising; bringing about, in a pulp of an ore containing one or more of said minerals in a state of subdivision where the mineral values thereof are substantially liberated, effective dispersion of said ore; then in an activation conditioning stage conditioning said effectively dispersed ore in said pulp with a predetermined amount of a collecting agent for the mineral values thereof; and then in a flocculating conditioning stage at a pulp pH within a range in which the mineral values thereof will flocculate in the presence of the collecting agent used, conditioning the pulp until substantial flocculation of said mineral values has occurred; then in a final conditioning stage at a pulp pH within the optimum range of pH for flotation of the mineral values thereof, conditioning the pulp with the addition of a dispersing agent to bring about effective dispersion of the gangue materials without deflocculating the mineral values; and then subjecting the thus conditioned pulp to flotation to produce a concentrate of the said mineral values.

15. A process as defined in claim 14 wherein said effective dispersion of said ore is brought about in the presence of a controlled amount, predetermined by experiment of at least one reagent selected from the group consisting of wetting agents, electrolytes and dispersing agents which are free from ions deleterious to flotation.

16. A process as defined in claim 14 wherein the mineral values are iron and said effective dispersion of said ore is brought about in the presence of a controlled amount, predetermined by experiment, of sodium silicate.

17. A process as defined in claim 14 wherein the mineral values are iron and said effective dispersion of said ore is brought about in the presence of a controlled amount, predetermined by experiment, of sodium sulfate.

18. A process as defined in claim 14 wherein the mineral values are iron and said effective dispersion of said ore is brought about in the presence of a controlled amount, predetermined by experiment, of a wetting agent which is free from ions deleterious to flotation.

19. A process as defined in claim 14 wherein the activation conditioning stage is carried out at an initial pH, predetermined by experiment, within the range of from about 6.7 to about 7.2 for a period of time, predetermined by experiment, of from about 4 to about 16 minutes.

20. A process as defined in claim 14 wherein the mineral values are iron and the activation conditioning stage is carried out at an initial pH, predetermined by experiment, within the range of from about 6.7 to about 7.2 for a period of time, predetermined by experiment, of from about 4 to about 16 minutes.

21. A process as defined in claim 14 in which the flocculating conditioning stage is carried out at an initial pH, predetermined by experiment of from about 5.0 to about 6.5 for a period of time, predetermined by experiment, of from about 8 minutes to about 16 minutes.

22. A process as defined in claim 14 in which the mineral values are iron and the flocculation conditioning stage is carried out at an initial pH, predetermined by experiment, of from about 5.0 to about 6.5 for a period of time, predetermined by experiment, of from about 8 minutes to about 16 minutes.

23. A process as defined in claim 14 wherein the final conditioning stage is carried out at an initial pH, predetermined by experiment, within the range of from about 7.2 to about 8.5 with the addition to the pulp of a controlled amount, predetermined by experiment, of sodium silicate and for a period of time, predetermined by experiment of from about 6 to about 21 minutes.

24. A process as defined in claim 14 wherein the mineral values are iron and the final conditioning stage is carried out at an initial pH, predetermined by experiment, within the range of from about 7.2 to about 8.5 with the addition to the pulp of a controlled amount, predetermined by experiment, of sodium silicate and for a period of time, predetermined by experiment of from about 6 to about 21 minutes.

25. A process for the flotation of metallic oxides, and metallic oxides with water of crystallization in chemical combination occurring in ores as finely disseminated minerals requiring fine grinding for the liberation of said minerals from essentially undeslimed pulps thereof, comprising; bringing about, in a pulp of an ore containing one or more of said minerals in a state of subdivision where the mineral values thereof are substantially liberated, effective dispersion of said ore; then in an activation conditioning stage conditioning said effectively dispersed ore in said pulp with a predetermined amount of a collecting agent for the mineral values thereof; then in a flocculation conditioning stage at a pulp pH within a range in which the mineral values thereof will flocculate in the presence of the collecting agent used, conditioning the pulp with the addition of a dispersing agent until substantial flocculation of said mineral values and effective dispersion of the gangue materials without deflocculating the mineral values has occurred; and then subjecting the thus conditioned pulp to flotation to produce a concentrate of the said mineral values.

26. A process as defined in claim 25 wherein said effective dispersion of said ore is brought about in the presence of a controlled amount, predetermined by experiment of at least one reagent selected from the group consisting of wetting agents, electrolytes and dispersing agents which are free from ions deleterious to flotation.

27. A process as defined in claim 25 wherein the mineral values are iron and said effective dispersion of said ore is brought about in the presence of a controlled amount, predetermined by experiment, of sodium silicate.

28. A process as defined in claim 25 wherein the mineral values are iron and said effective dispersion of said ore is brought about in the presence of a controlled amount, predetermined by experiment of sodium hydroxide.

29. A process as defined in claim 25 where the mineral values are iron and said effective dispersion of said ore is brought about in the presence of a controlled amount, predetermined by experiment, of sodium sulfate.

30. A process for the flotation of metallic oxides and metallic oxides with water of crystallization in chemical combination occurring in ores as finely disseminated minerals requiring fine grinding for the liberation of said minerals from essentially undeslimed pulps thereof comprising; preparing a pulp of an ore containing at least one mineral selected from the class consisting of metallic oxides and metallic oxides with water of crystallization, which has been finely ground to substantially liberate the minerals thereof for the process of flotation, in which pulp ground ore is effectively dispersed for differential activation; conditioning said pulp with collector

agent used for flotation of said minerals to differentially activate said minerals and then adjusting the pH of the pulp to within the optimum range for flotation of said minerals and depression of the gangue and conditioning said pulp for flotation in a final stage of conditioning with the addition of dispersing agent whereby gangue materials in said ore are effectively dispersed while the said minerals remain flocculated; and then subjecting the thus conditioned pulp to flotation to produce a concentrate of said minerals.

31. A process according to claim 30 in which the pH range within which said final stage of conditioning is carried out is from about 6.7 to about 8.9 and the predetermined period of time of said final stage of conditioning is a time predetermined by experiment within the range of from 3 to 25 minutes.

32. A process as defined in claim 30 wherein the pulp contains a controlled amount, predetermined by experiment, of an electrolyte which is free of ions deleterious to flotation.

33. A process as defined in claim 32 wherein the electrolyte is sodium sulfate.

34. A process as defined in claim 30 wherein the pulp contains a controlled amount, predetermined by experiment of a dispersing agent.

35. A process as defined in claim 34 wherein the dispersing agent is sodium silicate.

36. A process as defined in claim 30 wherein the mineral values are iron.

37. A process as defined in claim 30 wherein the mineral values are uranium.

38. A process as defined in claim 30 wherein the ore is an iron ore wherein the mineral values in the ore are selected from the group consisting of hematite, specular hematite, magnetite, martite and goethite.

39. A process as defined in claim 30 wherein the flotation reagent is selected from the group consisting of fatty acid, mixtures of fatty acid and petroleum sulfonate and mixtures of fatty acid and petroleum sulfonate and fuel oil wherein the fuel oil is present in amounts not exceeding 5 pounds per ton of solids.

40. A process as defined in claim 30 wherein the conditioning with collector agent is in an acid circuit and wherein said conditioning within the optimum range for flotation of said minerals and depression of the gangue is in an alkali circuit.

41. A process as defined in claim 40 in which the ore is a readily dispersible iron ore wherein the dispersion of the ore for differential activation and the conditioning of the pulp with a collector agent to differentially activate the minerals is combined in a single stage.

42. A process for the flotation of metallic oxides and metallic oxides with water of crystallization comprising; wet grinding an ore containing one or more of said minerals in the presence of a quantity predetermined by experiment of a dispersing agent and a quantity predetermined by experiment of a collecting agent for said minerals to produce a pulp of said ore wherein liberated mineral content thereof is activated; then at a pulp pH within a range in which the mineral values thereof will flocculate in the presence of the collecting agent used, in a flocculation stage of conditioning, conditioning the pulp until substantial flocculation of said mineral values has occurred; then at a higher pulp pH within the optimum range of pH for flotation of the mineral values thereof, in a final conditioning stage, conditioning the pulp to bring about effective dispersion of the gangue materials without deflocculating the mineral values.

43. A process as defined in claim 42 wherein the ore is an

iron ore wherein the mineral values in the ore are selected from the group consisting of hematite, specular hematite, magnetite, martite and goethite.

44. A process as defined in claim 42 wherein the flotation reagent is selected from the group consisting of fatty acid, mixtures of fatty acid and petroleum sulfonate and mixtures of fatty acid and petroleum sulfonate and fuel oil wherein the fuel oil is present in amounts not exceeding 5 pounds per ton of solid pulp.

45. A process for the concentration of iron ore occurring as finely divided minerals of the class consisting of hematite, martite, goethite, magnetite and specular hematite requiring fine grinding for the liberation of said minerals from essentially undeslimed pulps, which comprises subjecting the ore to wet comminution in the presence of a predetermined amount of sodium silicate to produce an effectively dispersed pulp of said ore; conditioning said ore in the presence of a collecting agent for the iron mineral present to cause flocculation of said mineral; lowering the pH to neutral or slightly lower by the addition of a suitable acid reagent; further conditioning the pulp in the presence of a predetermined amount of sodium silicate for a predetermined period of time to maintain the gangue materials dispersed while the pH of said pulp rises to within the optimum range for flotation of the said iron mineral to effect and bring about flocculation of said iron mineral favorable for flotation; and then subjecting the thus conditioned pulp to flotation to produce a concentrate of said iron mineral.

46. A process for the concentration of iron ore occurring as finely divided minerals of the class consisting of hematite, martite, goethite, magnetite and specular hematite requiring fine grinding for the liberation of said minerals from essentially undeslimed pulps, which comprises subjecting the ore to wet comminution, adding to said comminuted ore a predetermined amount of sodium silicate to produce an effectively dispersed pulp of said ore; conditioning said ore in the presence of a collecting agent for the iron mineral present to cause flocculation of said mineral; lowering the pH to neutral or slightly lower by the addition of a suitable acid reagent; further conditioning the pulp in the presence of a predetermined amount of sodium silicate for a predetermined period of time to maintain the gangue materials dispersed while the pH of said pulp rises to within the optimum range for flotation of the said iron mineral to effect and bring about flocculation of said iron mineral favorable for flotation; and then subjecting the thus conditioned pulp to flotation to produce a concentrate of said iron material.

47. The improved process of claim 46 wherein the collecting agent is a fatty acid collecting agent.

48. The improved process of claim 47 wherein in further conditioning the pulp the pH thereof is adjusted to within the optimum range for flotation by the addition of a reagent selected from the group consisting of a dispersing agent, a dispersing agent combined with an alkali or an alkali dispersing agent.

49. The improved process of claim 46 wherein the collecting agent consists of a combination of fatty acid collecting agent and petroleum sulfonate collecting agent.

50. The improved process of claim 46 wherein the collecting agent is a combination of fatty acid collecting agent, petroleum sulfonate collecting agent and fuel oil.

51. The improved process of claim 46 wherein the collecting agent consists of a combination of fatty acid collecting agent and fuel oil.