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SOUTH AUSTRALIAN GOVERNMENT DEPARTMENT OF MINES

Amdel Report

No. 517

PREPARATION OF IRON OXIDE AS PIGMENT

by

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SUMMARY

History

Deposits of micaceous hematite ore near Williamstown, SA were formerly exploited for iron flux, and, more recently for cement and paint-filler material.

However, mining closed due to flooding of the site under the present South Para water reservoir.

Other deposits in the same general area were taken up under Mineral Lease and a compnay, Micor Pty Ltd was formed in 1965 to exploit the deposits.

Previous work at laboratory scale and at pilot scale had shown that beneficiation of the ore by froth flotation using fatty-acid collectors produced concentrates closely approximating British and American Standards for paint pigment material.

Objectives

Further investigation was desired to determine:

a. the physical and chemical properties of material which should be aimed for in any beneficiation process, to compete with currently marketed pigments for paints, concrete masonary and ceramic ware,

b. the quality requirements for other uses, such as welding-rod coatings and ceramic magnet powders,

and to produce bulk samples of material as indicated by "a" and "b" for consumer evaluation.

Summary of Work Done

Laboratory milling and micronising of Malcolm Creek Ore, and ore beneficiated by flotation, gave powders of high specific surface area. The powder was of a plum-red colour, and the colour of synthetic red iron oxide could not be matched by roasting Malcolm Creek material in an oxidizing atmosphere. Reduction roasting however gave a black powder closely approaching the colour of a synthetic black iron oxide sample of imported origin. Samples of prepared natural colour and black pigments were submitted to ceramic and concrete manufacturers.

Laboratory and pilot-scale dry-milling and size classification tests on Malcolm Creek ore failed to produce the desired product grade. Wear on milling equipment was excessive due to the abrasive nature of the siliceous fraction of the ore. Further beneficiation of dry-milled and size-classified ore by flotation proved difficult, and low-grade concentrates were produced from continuous pilot-scale operation.

Wet milling and size classification of ore provided a suitable feed for flotation, and satisfactory concentrate grades and recoveries were obtained in continuous operation. A bulk sample of approximately 9 tons of concentrate containing 82.9% Fe_2O_3 was produced for consumer evaluation. The highest concentrate grade produced in one eight-hour period from continuous operation was 93.7% Fe_2O_3; calculated recovery at a concentrate grade of 88.5% Fe_2O_3 was 91%.

Conclusions

The following conclusions were drawn from the test work:

1. A micaceous hematite concentrate can be produced at satisfactory recovery from ground ore by froth flotation.
2. The grade of concentrate obtainable from a feed containing approximately 55% Fe_2O_3 is up to 95% Fe_2O_3, and thus meets the grade requirements of material for welding-rod coatings and structural paint filler.
3. Further size-classification or size reduction of flotation concentrate may be required to meet the size specifications of paint-filler grade material: however, the bulk sample produced meets the chemical and other physical requirements of most known paint-filler specifications.
4. Production of a black pigment of high surface are from a micaceous concentrate is feasible by reduction roasting and fine size reduction; however, the density of colour obtained will not match that of synthetic oxide powders of much higher purity.

Recommendations

It is recommended that portions of the bulk micaceous hematite concentrate sample produced be submitted to consumers for evaluation.

Should the market demand for such material warrant the establishment of a treatment plant, the basic flowsheet shown in Figure 3 is recommended. The data obtained from the pilot-scale operations described in this report can be used for specification of some of the major items of plant equipment.

1: INTRODUCTION

Deposits of micaceous hematite near Williamstown were worked as the Mt Bessemer Mine, mainly as a source of iron flux, and have been reported by Ward (1913) and Jack (1922, 1928). Mining at Mt Bessemer from 1935 to 1952 was carried out on a limited scale for use in cement and as a pigment (Miles 1950). Mining ceased at Mt Bessemer due to flooding of the area under the present South Para water reservoir; however, other areas some miles away and possibly an extension of the same ore body were taken up under Mineral Lease, and beneficiation investigations for the production of high-grade micaceous pigment conducted at Amdel (Bollen 1947, 1964). Subsequently a company, Micor Pty Ltd was formed to exploit the deposits.

The investigations reported here were aimed initially on a laboraty scale to produce three basic grades of material:

a. Micaceous oxide filler for structural paints.

b. Red iron oxide pigment for paints, concrete and ceramics.

c. Black iron oxide for paints, concrete and ceramics.

2: MATERIAL EXAMINED

2.1 Imported Pigments

Two samples produced by Farbenfabriken Bayer, East Germany, were received:

Iron Oxide,	Red	10F
Iron Oxide,	Black	318F

A sample of imported micaceous hematite as used by W. P. Crowhurst Pty Ltd, in structural paints was also received.

2.2 Malcolm Creek Ore

A sample of approximately 30 tons of ore from the Malcolm Creek deposit was received for beneficiation tests. The ore consisted of mainly fine, friable material, with some lumps up to approximately 5 inches in size.

The ore contained approximately 55% Fe_2O_3 as micaceous hematite, the remainder being principally quartz. Microscopic examination of the ore has been previously reported (Amdel Report 200).

3. EQUIPMENT

3.1 Laboratory Tests

The following equipment was used in batch tests:

British Standard Screens (BSS)	
Crushing rolls	6 x 10 inch diameter
Microniser	8 inch diameter
Laboratory vertical tube furnace	
Laboratory batch rod mills	7 inch dia. x 10 inch
	11 inch dia. x 13 inch
Fagergren laboratory flotation machine	3 litre capacity
Agitair flotation machine	40 litre capacity
Laboratory attriter	500-g capacity
Wemco attritioner	10 Kg capacity
Raymond laboratory hammer mill	
Gayco centrifugal air classifier	2 ft diameter
Raymond centrifugal air classifier	3 ft diameter
Pelletising drum	2 ft diameter

3.2 Pilot-Scale Tests

The following equipment was used in continuous tests:

Raymond Model 0000 impact mill	
Raymond centrifugal air classifier	3 ft diameter
Boulton gyratory screen	
Ball mill	2 ft diameter x 4 ft
Denver No. 15 flotation cells	
Agitair No. 8 flotation cells	

Accessory equipment, including ore feeders, reagent feeders, slurry pumps, vacuum filters, etc.

4. ANCILLARY MATERIALS

The fatty-acid collector mixture used in flotation was made up of the following components:

Tall oil, semi-refined (PAMAK 4) supplied by A.C. Hatrick Pty Ltd.

Fuel Oil, light grade furnace oil.

Triton X-100, a non-ionic emulsifier, supplied by Robert Bryce and Co. Ltd.

Naphthenic acid, grade SP180, supplied by Shell Chemical Co.

5. EXPERIMENTAL PROCEDURE AND RESULTS

5.1 Examination of Imported Pigments

5.1.1 Chemical Analysis

Analyses of samples of imported synthetic iron oxides for acid insoluble material were used as a guide to purity.

Sample	Acid insoluble %
Iron oxide red 10F	1.06
Iron oxide black 318F	1.61

5.1.2 Sizing

The samples were sized using a Fisher sub-sieve sizer and Cyclosizer. The average particle diameter for both samples was 0.5 microns. The size distribution is shown in Table 1.

5.2 Examination of Imported Micaceous Hematite

5.2.1 Chemical Analysis

Fe_2O_3 88.7%

5.2.2 Sizing

Sizing of material by screening is as below:

Nominal Aperture µ	Mesh BSS	Weight %
+355	+ 44	Nil
-355 +250	- 44 + 60	Nil
-250 +150	- 60 +100	Trace
-150 + 75	-100 +200	2.6
- 75	-200	97.4
		100.0

5.3 Laboratory-Scale Beneficiation of Malcolm Creek Ore

A sample of lump ore received was crushed to minus 10-mesh, and representative samples cut out for tests.

5.3.1 Micronising

A sample of crushed ore was fed to an 8-inch diameter microniser at a feed rate of approximately 20 pounds per hour. The milled product was a deep plum-red colour. The major silica content of the ore remained in the microniser grinding chamber as coarse particles; it may be possible to bleed of this material in a continuous milling operation to allow simultaneous milling and beneficiation of the iron oxide product. Average particle diameter of the mirconised product was 1.1 microns.

Size distribution of the micronised product was:

Particle Size μ	Weight %
+25.4	0.2
-25.4 +19.2	1.7
-19.2 +13.5	5.0
-13.5 + 8.9	10.6
- 8.9 + 6.9	10.0
- 6.9	72.5
	100.0

5.3.2 Roasting of Micronised Ore

Samples of micronised ore were agglomerated by adding water, and mixing by hand to form pellets which were then oven dried.

Oxidation Roasting. Pellets of material were held in porcelain crucibles in a muffle furnace at a temperature of 1000°C for 1 hour and 4 hours, with free air access, and also in an atmosphere of steam.

On cooling, the material showed some slight reddening in colour from the micronised material, but with no difference between 1 and 4 hours treatment.

Reduction Roasting. Pellets of material were held in a stainless-steel mesh basket in a vertical tube furnace at a temperature of approximately 600°C for 2 hours. An atmosphere of coal-gas or hydrogen was maintained during the roasting and cooling stages.

The resultant material was black in colour, but not of comparable colour-density to synthetic black oxide pigment samples.

5.3.3 Flotation Testing.

Preliminary batch tests indicated that a grinding time of 10 minutes gave a satisfactory feed size, and good flotation results were obtained using 2 pounds of tall-oil collector per ton of ore.

The collector mixture used was of the following composition:

	%
Pamak 4	18.9
Fuel oil	79.4
Naphthenic acid	3.8
Triton X-100	1.9
	100.0

Results of a test in which the froth concentrate was cleaned once are shown in Table 2.

Further tests were done, employing two stages of cleaning, and the resultant concentrate samples were combined to form a bulk sample for testing.

The bulk concentrate assayed 90.9% Fe_2O_3.

The sizing of the bulk concentrate by screening was:-

Nominal Aperture μ	Mesh BSS	Weight %
+355	+ 44	Nil
-355 +250	- 44 + 60	Trace
-250 +150	- 60 +100	1.0
-150 + 75	-100 +200	10.8
- 75	-200	88.2
		100.0

5.3.4 Milling and Micronising of Flotation Concentrate

A sample of bulk flotation concentrate was treated in a laboratory Raymond hammer mill in preparation for micronising.

Sizing of the milled material was:

Nominal Aperture μ	Mesh BSS	Weight %
+150	+100	Trace
-150 +125	-100 +120	0.2
-125 + 90	-120 +170	2.2
- 90 + 75	-170 +200	1.4
- 75 + 63	-200 +240	5.4
- 63 + 45	-240 +350	14.2
- 45	-350	76.6
		100.0

The average particle diameter of the micronised material was 0.5 microns. Size distribution of the micronised material was:

Particle Size µ	Weight %
+25.3	0.2
-25.3 +18.8	Trace
-18.8 +13.2	0.4
-13.2 + 8.7	0.4
- 8.7 + 6.7	0.6
- 6.7	98.4
	100.0

5.3.5 Reduction Roasting of Micronised Flotation Concentrate

Samples of micronised concentrate were pelletised by hand, and held in a vertical tube furnace for 4 hours at temperatures of approximately 550 and 650°C in both coal gas and hydrogen atmospheres. Samples reduced in coal gas were of a lower intensity black colour than those reduced in hydrogen.

5.3.6 Milling and Air Classification

A sample of approximately 140 lb of lump ore was rolls crushed to nominally 10 mesh, and fed to a laboratory hammer mill fitted with an $^1/_8$ inch aperture perforated screen. The milled material was fed to a centrifugal air classifier and separated into coarse and fine fractions. The coarse fraction consisted essentially of free siliceous material. The fine fraction represented 62% of the weight of feed to the unit and assayed 70.0% Fe_2O_3. The coarse fraction was not assayed.

Flotation of Milled and Classified Ore. Samples of 30 lb of the fine fraction from air classification were conditioned at approximately 70% solids with tall oil collector emulsion for 20 minutes in an attrition-conditioner. It was found necessary to add up to approximately 8 lb of collector per ton to obtain satisfactory flotation. The conditioned material was floated, and the froth concentrate was cleaned twice.

Final concentrate from three such flotation separations was combined, filtered, dried, weighed and sampled.

The concentrate assayed 97.0% Fe_2O_3.

Sizing of the concentrate was:

Nominal Aperture µ	Mesh BSS	Weight %
+150	+100	0.3
-150 +125	-100 +120	0.3
-125 + 90	-120 +170	2.4
- 90 + 75	-170 +200	2.0
- 75 + 63	-200 +240	3.5
- 63 + 45	-240 +350	11.3
- 45	-350	80.2
		100.0

Micronising. A sample of approximately 30 lb of flotation concentrate was fed to an 8-inch diameter microniser unit.

Average particle diameter of the micronised product was 1.3 microns.

Reduction. A sample of approximately 15 lb of micronised material was agglomerated in a pelletising drum at approximately 20% moisture content, oven dried, and held in a reduction container in a muffle furnace for 4 hours. A hydrogen atmosphere was maintained in the reduction chamber during both heating and cooling cycles.

The reduced material was then milled in a laboratory hammer mill fitted with an 0.25 mm slotted screen.

Average particle diameter of the milled material was 1.5 microns.

5.4 Pilot-Scale Beneficiation of Malcom Creek Ore

Following laboratory batch tests to produce samples for evaluation as filler for structural paints, welding rod coatings, and pigments for ceramic and masonry ware, tests were done at pilot scale to produce bulk samples from a 30 ton ore parcel.

The tests were done within the following programme:

a. Dry milling of feed, and size classification of the milled ore to reject siliceous material as a coarse fraction. Flotation concentration of the hematite fraction, to produce a froth concentrate suitable as a filler for structural paint without further size reduction.

b. Wet milling of ore and flotation to produce a froth concentrate suitable for structural paint filler.

c. Further up-grading of concentrate from (b) by flotation to produce material suitable for welding rod coatings, and possible uses, after further treatment, for ceramic and masonry pigment, and ceramic magnetic iron oxides.

5.4.1 Feed Preparation

The ore received in drums was passed through a rolls crusher set of ½ inch, and the crushed product re-drummed.

The ore was considered to be fairly uniform in grade from drum to drum, and no attempt was made to blend the total sample or obtain a representative head sample.

5.4.2 Dry Milling

Drums of ore were tipped into a portable bin fitted with a constant-weight belt-feeder, and fed to a dry milling circuit comprising Raymond impact mill, cyclone product collector and Raymond centrifugal air classifier.

Preliminary tests were done at varying feed rates and classifier settings, aiming for a coarse siliceous product low in hematite content which could be rejected, and a fine product containing most of the hematite, and a minimum of fine quartz. The conditions that most-closely met these requirements were a feed rate of 800-900 pounds per hour, and coarse classifier settings. However, sampling and examination of classifier products showed that hematite recovery into the fine product was low:

Product	Weight %
Coarse	67
Fine	33
	100

Sizings of the products are tabulated below:

Screen Sizing Mesh BSS	Weight %	
	Coarse	Fine
+ 8	0.1	Nil
- 8 + 12	0.5	Nil
- 12 + 16	1.0	Nil
- 16 + 22	2.0	Nil
- 22 + 30	3.2	Nil
- 30 + 44	8.1	Nil
- 44 + 60	13.1	Nil
- 60 + 85	14.2	Nil
- 85 +120	11.0	Nil
-120 +170	8.0	Nil
-170 +200	2.1 (a)	Nil
-200	36.7	100.0 (b)
	100.0	100.0

(a) Quartz, some hematite. 90% Hematite, 10% Quartz.
(b) 70% Hematite, 30% Quartz.

The Raymond centrifugal classifier could not be adjusted to make a classification at coarser than 200 mesh which would have been required to obtain high recovery of hematite in the fine product.

Attempts were made to further classify the coarse product from the Raymond unit. Samples were treated in a Gayco centrifugal classifier at various conditions, but only an additional 2% by weight was recovered as a fine hematite-rich fraction.

A sample of the coarse product was screened at 60 mesh on a continuous Boulton gyratory screen. A good separation was obtained at high throughput, the oversize product being rejectable because of an unsignificant hematite content. (See Table 3).

It was therefore decided to treat the whole sample by milling, air classification and dry-screening of the coarse product as shown in Figure 1. A screen size of 60 mesh was selected as one giving sufficiently high screening rate, and giving an undersize product suitable for flotation separation.

Results of the complete separation are shown in Table 1.

5.4.3 Flotation of Dry-Milled Ore

Samples of the two hematite-bearing fractions from dry milling and classification were treated in batch flotation tests. A tall-oil collector mixture was added at the rate of 4 to 8 pounds per ton of flotation feed, either to a high-density conditioning stage, or to the flotation cell. Satisfactory flotation was obtained with both samples after conditioning in the flotation cell at approximately 25% solids for 10 minutes with 7 lb of collector per ton of feed. Results of the tests are shown in Tables 4 and 5.

Treatment of Classifier Fine Product. This material was treated by flotation to aim for a concentrate grade of 80% Fe_2O_3 minimum which would require no further milling to meet the size requirements for structural paint filler.

The material was fed at the rate of 250 pounds per hour to the circuit as shown in Figure 2. Initially, 6 lb of reagent per ton was added, but flotation conditions were poor, and reagent dosage was increased to 9, and then to 12 pounds per ton. Flotation conditions were generally poor, partly due to lack of control over the induced aeration on the cells used - this allowed cleaner cells to operate much faster than desirable. However, tailing grades were very high in Fe_2O_3, and all reject material was saved for possible re-treatment. The flotation circuit was sampled near the conclusion of the run:

Product		Assay Fe_2O_3%
Rougher concentrate:	1st cell	68.4
	2nd cell	63.7
	3rd cell	70.4
	4th cell	51.6
Cleaner concentrate		69.4
Re-cleaner concentrate		70.5
Rougher tailing		45.9

The bulk concentrate from the continuous operation was sampled and assayed 72.6% Fe_2O_3.

Because of the unsatisfactory grade of this product, it was air-dried, and stored for further treatment.

Treatment of Screen Undersize Fraction. Because of the unsatisfactory results from treatment of classifier fine fraction in the large-scale flotation cells, treatment of the screen undersize fraction was commenced in the small flotation plant at the rate of 120 pound per hour, using a flowsheet similar to that shown in Figure 2.

Treatment was started using 8 pounds of reagent per ton of feed, but conditions were poor, and this was increased to 12 pounds per ton. Good flotation could be obtained on start-up of the plant, but conditions progressively deteriorated with continuous operation. At this point it was established that samples of conditioned feed pulp from the plant required 5 to 10 minutes further conditioning, without additional reagent, in the laboratory flotation cell to give satisfactory flotation. Preliminary batch tests had given satisfactory results after 10 minutes conditioning (see Table 4) and calculated mean retention time in the plant conditioner was approximately 60 minutes; however, the mechanism of conditioning in the two cases was quite dissimilar so that the two units can not be compared.

A second conditioner tank of similar capacity was installed in series with the original one, and flotation conditions improved sufficiently to allow the production of a batch of high grade concentrate for submission overseas.

Consistent operating conditions were obtained after replacing the agitator-sump with a 2-stage high density conditioner. The dry feed was pulped with water to 70% solids and conditioned with reagent in this unit, diluted to 15-20% solids and pumped to the two low-density conditioners in series, and gravitated to flotation.

The following concentrate was obtained from the screen undersize fraction of dry-milled ore:-

Batch No.	Assay Fe_2O_3%
1	95.1
2	93.3
3	92.5

Re-Treatment of Flotation Concentrate From Classified Fines. Low-grade concentrate obtained from the large-scale circuit, using a 2-stage high density conditioner for re-pulping and the addition of further reagent (approximately 3 lb/ton).

The grade of the resultant froth concentrate was 76.9% Fe_2O_3, and was thus unsuitable for paint filler grade material.

5.4.4 Wet Milling and Flotation of Ore

Preliminary Testing. The plant was set-up as shown in Figure 3.

The flotation circuit was modified to allow a low-grade scavenger froth concentrate to be re-cycled to the head of the circuit.

The ore was ground using a ball-mill in closed-circuit with a hydrocyclone. A sieve-bend screen was installed to treat cyclone underflow material in anticipation that a coarse quartz fraction low in hematite may be rejected from the circuit by the screen. Sizings of samples taken after bedding-in of the grinding circuit at a feed rate of 500 pound per hour are shown in Table 6.

The sieve-bend screen oversize product, although a relatively small proportion of total ore, contained sufficient hematite to indicate re-cycling the product to ball mill.

The plant was run for 2 days at a feed rate of approximately 500 pounds per hour and a reagent dosage of approximately 12 pounds per ton.

Samples taken over the second day's run gave the following results:

Sample	Weight %	Fe_2O_3% Assay	Fe_2O_3% Distribution
Final concentrate	73.2	76.6	95.7
Final tailing	26.8	9.32	4.3
	100.0	58.6 (a)	100.0

(a) Calculated grade.

Reagent addition was reduced to approximately 8 pounds per ton of ore, to increase the concentrate grade, and the plant was prepared for continuous operation on a 24 hours per day basis to treat the main ore sample at a feed rate of approximately 800 pounds per hour.

Continuous Operation. The plant was operated for seven consecutive 8-hour shifts. Ore feed-rate ranged from 900 to 1100 pounds per hour on a shift-average basis, although some feeder difficulties with moist ore caused short-term fluctuations of from 800 to 1400 pounds per hour in feed rate. Average reagent addition was 8 pounds per ton of feed.

Under steady feed conditions, the circuit operated satisfactorily, and concentrates were filtered on batch vacuum filters. Flotation concentrates of known or suspected unacceptable grade (80% Fe_2O_3) were stored in drums without filtering, for further processing.

Filtered concentrates were sampled on an 8 hour shift basis for assay:

Shift No.	Concentrate Assay Fe_2O_3%
1	93.7
2	78.4
3	74.7
4	79.5
5	88.5
6	74.5
7	82.4

Batches of low grade, from shifts 3 and 6, produced during conditions of reduced feed rate and consequent increased reagent dosage per ton of feed, were set aside. The remaining lots were combined, air-dried and sampled. The bulk sample, of approximately 9 tons, assayed 82.9% Fe_2O_3.

Sizing of the bulk concentrate sample was:

Nominal Screen Aperture μ	Equivalent Mesh BSS	Cumulative Weight Passing %
250	60	100.0
180	85	99.0
125	120	95.0
90	170	86.2
75	200	82.7
63	240	69.1
53	300	64.1
45	350	24.7

Analysis of the material, relevant to paint filler specifications is shown in Appendix D.

Samples from the whole circuit were taken under conditions of satisfactory operation. These were weighed wet, filtered and the solids dried and re-weighed, then sampled for assay and, in some cases, sized.

Data obtained from the circuit is shown in Tables 7 and 8.

The recovery of Fe_2O_3 calculated from the data in Table 7 was approximately 91%.

Upgrading of Concentrate. Concentrate slurry from the main ore treatment, estimated to average 75-80% Fe_2O_3 content, was pumped to the small flotation plant for re-treatment. No further reagent addition was made, and the concentrate was upgraded by three stages of flotation. Tailing from re-treatment was rejected to waste.

Results of the re-treatment of approximately 2 tons of material were:

Batch No.	Assay Fe_2O_3%
1	94.5
2	91.9

6. DISCUSSION

6.1 Laboratory Tests

6.1.1 Comparison of Beneficiated Malcolm Creek Ore with Imported Pigments

Standard and typical consumer specifications for micaceous hematite for use as welding rod coating and for filler in structural paints are shown in Appendices A and B.

The material produced by laboratory batch flotation of ground ore met the specifications for welding rod coating material both for chemical analysis and size distribution. Flotation concentrate satisfied the grade requirements both of a structural paint filler manufacturer's specification and a paint filler sample supplied, but contained 2.8 and 1.2% respectively excess material coarser than 200 mesh. However, it is expected that this sizing specification could be met by control of the milling operation.

Hammer milled flotation concentrate contained some red undertone in the steel grey colour of the material but it is not expected that this would be deleterious in a structural paint.

Micronising of ore as received produced a plum-red coloured material which was roasted under reducing conditions to give a grey-black coloured pigment. Although a significant amount of free silica was rejected during micronising, the final product would still have a relatively high silica content and thus could not be compared with the high purity or dense black colour of the imported Bayer pigment.

Micronised flotation concentrate was of a similar sizing to the Bayer pigment. Reduction roasting gave a good black colour, which however was not as dense as the imported material. However, it is likely that both micronised, and micronised and reduced material, may be suitable as pigments in concrete, masonry and ceramic products.

6.1.2 Dry Milling and Size Classification

Air classification of milled ore did not give sufficient up-grading, at high weight rejection, to produce material suitable for use in welding rod-coatings or structural paints. However, the material produced by such a preliminary beneficiation step was considered to have possible application as a filler in asphalt, or concrete and ceramic masonry products.

Flotation of the fine product from air classification gave a high grade concentrate which was readily micronised to produce pigment of high specific surface area.

6.2 Pilot-Scale Tests

6.2.1 Dry Milling and Size Classification

It was not possible, in the equipment available, to size classify milled ore in one step to give satisfactory rejection of a barren quartz fraction. Screening at 60 mesh rejected approximately 20% of the ore with little hematite loss, and although dry screening at this size would be possible economically, the dry milling process is undesirable because of excessive abrasion on equipment. In the test work reported, two sets of impact hammers were consumed in treating approximately 10 tons of ore, and other moving parts of the impact mill were severely worn.

6.2.2 Flotation of Dry Milled Ore

Flotation treatment of the fine classified fraction from dry-milled ore was unsatisfactory.

Batch testing of this fraction had indicated relatively low recovery, and the necessity for a high middlings weight recirculation to obtain satisfactory concentrate grade. Low concentrate grade was obtained in initial continuous flotation treatment, but this may be partly attributed to difficulties in obtaining satisfactory conditioning time with reagents, and to mechanical problems with the flotation equipment. However, re-treatment of concentrate in small continuous equipment failed to produce a satisfactory concentrate grade.

Treatment of the coarser hematite fraction from dry-milled ore in the continuous flotation plant gave satisfactory results as predicted by preliminary batch tests. The highest concentrate grade obtained in continuous tests was 95.1% Fe_2O_3.

6.2.3 Wet Milling and Flotation

Wet milling, wet size classification and flotation was successfully carried out on a continuous basis at the rate of approximately ½ ton of feed per hour.

The grade of concentrate obtained varied, on an 8-hour shift basis from approximately 75 to 90% Fe_2O_3 and a bulk sample of approximately 9 tons of material produced contained 82.9% Fe_2O_3.

Re-treatment of concentrate from the ½ ton per hour pilot plant in a small flotation circuit using three stages of flotation gave a concentrate grade of 91.9% Fe_2O_3. Such material meets the grade requirements of welding-rod coatings, but does not meet the requirements for ceramic magnetic powder.

7. CONCLUSIONS

7.1 Laboratory Testing

Material suitable for uses in welding-rod coatings can be produced by flotation of ground Malcom Creek ore.

Material suitable as a filler in structural paints can be produced by milling of flotation concentrate.

Further size reduction by micronising produces a pigment which may find application in concrete or ceramic products. A black pigment can be produced by roasting micronised material in a reducing atmosphere.

7.2 Continuous Pilot-Scale Testing

The following conclusions have been reached from pilot-scale tests:

1. Dry milling of ore to the desired product size followed by flotation is not an attractive treatment method. Although up to 20% of feed can be rejected with low hematite loss by size classification of milled ore, wear rate on milling equipment is high, and subsequent flotation concentration of the finest fraction of the milled ore is difficult.

2. Wet milling of ore followed by flotation with two stages of cleaning is capable of maintaining a concentrate grade of 85-90% Fe_2O_3 at a recovery of approximately 90%. However, it is important to operate the mill with closed-circuit size classification equipment which will allow a suitable maximum flotation feed size to be maintained without producing excessive fine material, which lowers the flotation selectivity. The concentrate produced by wet milling and flotation will thus be coarser than that desired for some end uses, and will require further size reduction with a consequent partial break-down of the grey-black colouration to a reddish tone. This is acceptable, within limits, for paint-filler grade material.

3. The maximum grade of concentrate practicably obtainable by flotation of hematite with fatty-acid collectors, even with numerous cleaning stages, is considered to be approximately 95% Fe_2O_3.

8. REFERENCES

BOLLEN, L., (1947), "Malcolm Creek Iron Ore - Beneficiation Tests", Amdel Report 200.

BOLLEN, L., (1964), "Preparation of Iron Oxides as Pigment", Amdel Report 350.

JACK, R. L., (1922), "The Iron Ore Resources of South Australia", Geol. Survey of South Aust. Bull. 9, pp 15-20.

MILES, K. R., (1950), "The Geology of the South Para Dam Project", Geol. Survey of South Aust. Bull 24, pp 45-46.

WARD, L. K., (1913), Mining Review 17 pp 24-27.

APPENDIX A

TYPICAL AUSTRALIAN CONSUMER SPECIFICATION

Iron Oxide, Micaceous, for Welding-Rod Coating

Screen Analysis:

Grade 100 mesh

Retained on 44 mesh BSS sieve	Nil
Retained on 60 mesh BSS sieve	2% max.
Passing 60 mesh - retained 100 mesh BSS sieve	30% max.
Passing 200 mesh	50% max.

Chemical Analysis:

Silica,	SiO_2	7% max.
Alumina,	Al_2O_3	0.4% max.
Ferric Oxide,	Fe_2O_3	90% max.
Ferrous Oxide,	FeO	Nil
Sulphur		0.05% max.

Tests Required:

Spectrographic comparison.

Chemical analysis - Fe_2O_3.

Screen Analysis.

APPENDIX B

TYPICAL AUSTRALIAN CONSUMER SPECIFICATION

Micaceous Iron Oxide, Paint, Filler Grade

General Description:

A naturally occurring foliated micaceous iron oxide with characteristic steel-grey (graphitic) appearance and red undertone. Supplied as a fine dry powder.

Safety Precautions:

This pigment contains siliceous material and must be considered as toxic. Dust respirators should be worn when handling it.

Specification:

A. Routine Control

Test	Limits	Method
Colour (straight)	Close to standard	ASTM 101.1 or 101.2
Fineness	Equal to standard	121.1
Coarse particles	1% retained on 200 BS mesh sieve - maximum	122.1
Oil absorption	8.5 to 11.5 (a)	551.1
Moisture	0.5% maximum	260.1 or 260.2

B. Additional Control Tests to be carried out Periodically

Test	Limits	Method
Iron oxide content	80% minimum as Fe_2O_3	ASTM 405.2
Water soluble	0.3% maximum	553.1

General Information:

This material contains an average of 17% SiO_2 and siliceous matter. Approximately 0.7% TiO_2 and 1.5% CaO, MgO and Alkalies. It is used as the prime pigment in Dark Metallic Grey Roof Paints. The specific gravity varies from 4.39 to approximately 5.0.

(a) The determination of the end point of the oil absorption is a difficult matter with this pigment.

APPENDIX C

EXTRACTS FROM BRITISH STANDARD 3981 : 1966

Specification for Iron Oxide Pigments for Paints

Classification of Pigments:

Colour	Black
Class	8
Former BS description	None (micaceous iron oxide)
Fe_2O_3 content percent by weight (minimum)	85
Grades and types	Single grade and type

Properties of Pigments:

Class	8
Description of oxides	Micaceous iron
Residue on sieve of nominal aperture 63μ	10.0
Normal range of oil absorption values	Not required
Water covering area	300
Volatile matter at 105°C	1.0
Matter soluble in water	0.5
pH of extract	6
Soluble chlorides	0.1
Content of carbonates	0.5
Content of sulphur and sulphur compounds other than insoluble sulphates	3.5 (a)

(a) By agreement between vendor and purchaser, this limit may be reduced.

APPENDIX D

REPORT ON ANALYSIS OF MICACEOUS HEMATITE

Commonwealth of Australia
Department of Supply

Address all Communications to
Branch Superintendent, and quote
File No. 1/988
TS66-826
BW

Defence Standards Laboratories
South Australian Branch
Box 1935 P, GPO Adelaide
South Australia

23rd September, 1966

Submitted by:	Micor Pty Ltd 100 Currie Street, Adelaide, S. A.
Reference:	Order No. 4 dated 8/9/66.
Description:	Micaceous Hematite submitted for examination as a paint pigment.
Date Received:	8/9/66.
RESULTS OF ANALYSIS:	The results of chemical and physical analysis are set out below:

Iron as ferric oxide (Fe_2O_3)	82.3 per cent
Matter insoluble in hydrochloric acid	15.3 per cent
Sulphate (SO_4)	0.01 per cent
Water soluble matter	0.10 per cent
Water at 105^0C (moisture)	0.10 per cent
pH	6.5
Specific gravity	4.48
Oil absorption (ASTM D281-31)	13lb/100lb pigment

(J. F. McNeil)
BRANCH SUPERINTENDENT

Report No. TS66-826

TABLE 1: SIZE DISTRIBUTION OF IMPORTED PIGMENTS

Red 10F		Black 318F	
Particle Size μ	Weight %	Particle Size μ	Weight %
+25.2	1.4	+23.5	3.0
-25.2 +18.7	1.7	-23.5 +18.2	2.1
-18.7 +13.1	2.9	-18.2 +12.8	3.5
-13.1 + 8.7	4.0	-12.8 + 8.4	4.0
- 8.7 + 6.7	3.6	- 8.4 + 6.5	2.6
- 6.7	86.4	- 6.5	84.8
	100.0		100.0

TABLE 2: PRELIMINARY FLOTATION TESTING

Product	Weight %	Fe_2O_3%	
		Assay	Distribution
Cleaner concentrate	49.3	87.6	70.9
Cleaner tailing	20.3	63.8	21.2
Rougher tailing	30.4	15.9	7.9
	100.0	63.0	100.0

TABLE 3: DRY MILLING AND CLASSIFICATION

Product	Weight % approx.
Air classifier fines	36
60-mesh screen undersize	42
Total hematite product	78
60-mesh screen oversize (quartz reject)	22
	100

TABLE 4: BATCH FLOTATION OF PRODUCTS FROM DRYMILLING

Fraction	Product	Weight %	Fe_2O_3% Assay	Fe_2O_3% Distribution
Classifier fines:	Flotation concentrate	48.1	88.0	64.3
	Combined middlings	40.8	51.6	32.0
	Flotation tailing	11.1	21.8	3.7
		100.0	65.9(a)	100.0
Screen undersize: (from classifier coarse fraction)	Flotation concentrate	56.2	94.0	81.3
	Combined middlings	16.8	63.1	16.3
	Flotation tailing	27.0	5.8	2.4
		100.0	64.9(a)	100.0

(a) Calculated values.

TABLE 5: SIZE DISTRIBUTION OF BATCH FLOTATION PRODUCTS

Screen Mesh BSS	Weight %	
	Concentrate from classifier fines fraction	Concentrate from Screen undersize fraction
+ 60	Nil	Trace
- 60 + 80	Trace	2.6
- 80 +120	Trace	9.8
-120 +170	0.6	13.9
-170 +240	1.0	17.5
-240 +350	1.1	18.2
-350	97.3	38.0
	100.0	100.0

TABLE 6: SIZE DISTRIBUTION OF GRINDING AND CLASSIFICATION FRACTIONS

Mesh BSS	Weight %			
	Ball-mill Discharge	Cyclone Overflow	Sieve-bend Undersize	Sieve-bend Oversize
+ 22	Trace	Nil	Nil	Trace
- 22 + 30	0.2	Nil	0.2	0.2
- 30 + 44	0.8	Nil	1.2	1.0
- 44 + 60	2.2	Trace	3.6	3.0
- 60 + 85	4.1	0.3	5.7	5.6
- 85 +120	8.4	1.1	10.3	11.4
-120 +170	18.1	5.8	18.7	22.2
-170 +240	17.1	13.6	16.3	17.4
-240 +350	12.3	10.0	10.2	11.2
-350	36.8	69.2	33.8	28.0
	100.0	100.0	100.0	100.0

TABLE 7: DATA FROM WET MILLING AND FLOTATION

Product	Pulp Solids %	Weight [(a)] %	Assay Fe_2O_3%
Grinding and Classification			
Mill discharge	55	100	53.1
Cyclone underflow	69	33	56.7
Cyclone overflow	35	67	49.3
Sieve-bend undersize	43	16	65.3
Sieve-bend oversize	65	17	53.6
Flotation			
Rougher concentrate	55	70	78.4
Scavenger concentrate	15	70	78.4
Cleaner concentrate	56	66	83.0
Re-cleaner concentrate	53	62	88.0
Scavenger tailing	15	38	14.4

(a) Approximate weight % of Mill discharge.

TABLE 8: SIZE DISTRIBUTION OF SAMPLES FROM WET MILLING AND FLOTATION

Mesh BSS	Weight %									
	Ball-Mill Discharge	Cyclone U/f (a)	Sieve-Bend		Cyclone O/f (b)	Ro Conc	Scav Conc	Clnr Conc	Final Conc	Final Tail
			Oversize	Undersize						
+ 22	Trace	0.5	0.6	nd (c)	nd	Nil	Nil	Nil	Nil	nd
- 22 + 30	Trace	0.9	0.9	nd	nd	Nil	Nil	Nil	Nil	nd
- 30 + 44	0.5	3.8	4.5	nd	nd	Nil	Nil	Nil	Nil	nd
- 44 + 60	4.1	8.8	10.2	4.9	5.4	0.3	0.1	0.1	0.2	4.2
- 60 + 85	11.0	14.0	17.0	6.0	9.6	1.5	1.5	1.0	1.4	13.0
- 85 +120	16.0	16.0	20.2	9.5	11.0	4.4	3.7	4.0	4.6	22.2
-120 +170	16.1	13.4	15.5	14.0	11.0	8.3	8.1	8.5	9.0	20.0
-170 +240	11.6	9.0	8.6	13.8	9.6	10.4	12.8	11.2	12.5	11.6
-240 +350	3.6	3.0	2.5	4.6	3.7	5.0	6.0	5.2	5.0	25.8
-350	37.1	30.5	20.0	47.2	49.7	70.1	67.8	70.0	67.3	25.8
	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

(a) U/f - Underflow.
(b) O/f - Overflow.
(c) nd - not determined.

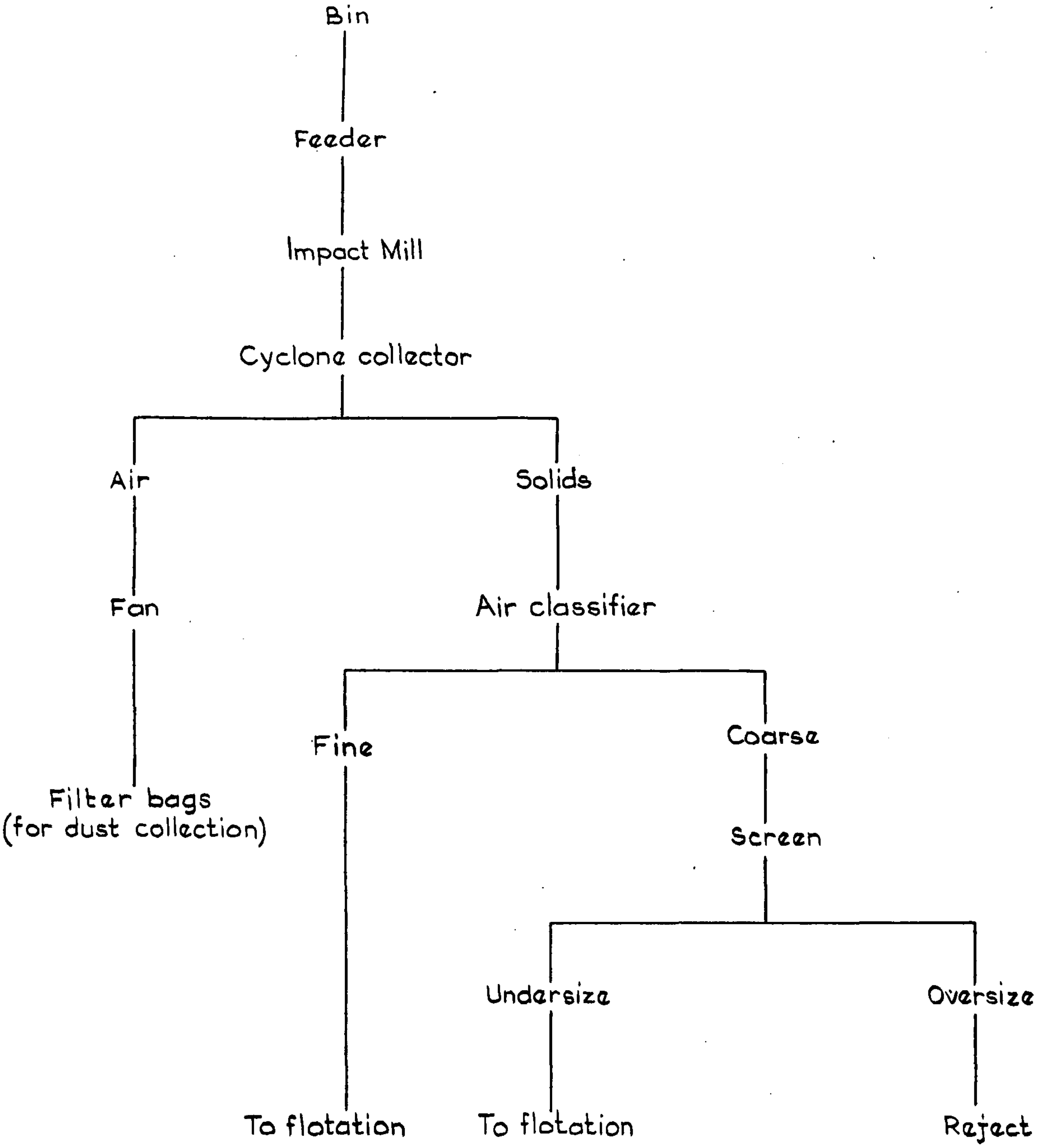

FIG. 1: FLOW SHEET FOR PILOT-SCALE
Dry milling and classification

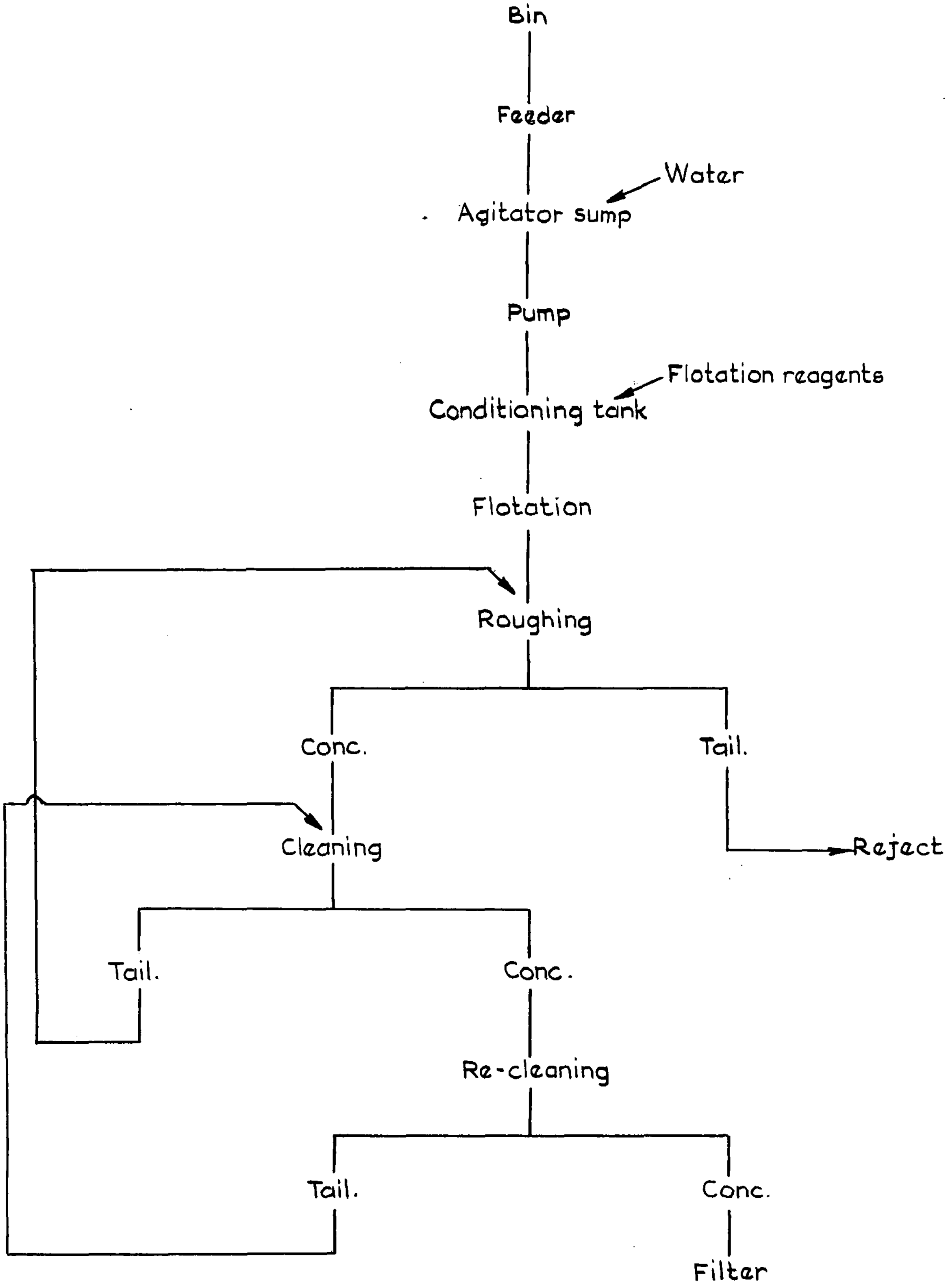

FIG. 2: FLOW SHEET FOR PILOT-SCALE
Flotation of dry milled classifier fines

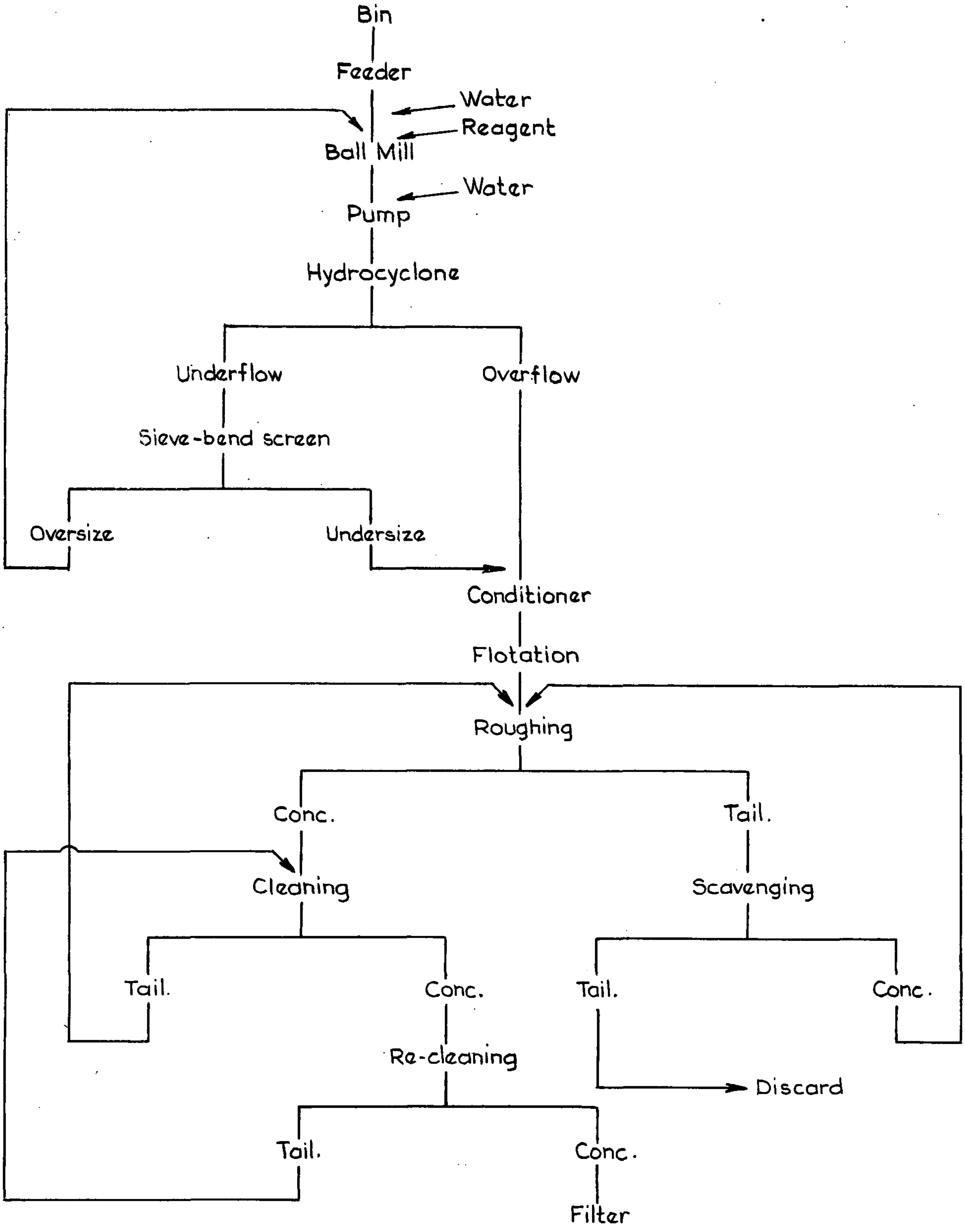

FIG. 3: FLOW SHEET FOR PILOT-SCALE
Wet milling and flotation of ore