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A PILOT SCALE DEMONSTRATION OF THE IMC/CLDRI/FIPR FLOTATION PROCESS FOR FLORIDA HIGH-MgO PEBBLE

Prepared by IMC Phosphates Company

under a grant sponsored by



August 2001

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A PILOT-SCALE DEMONSTRATION OF THE IMC/CLDRI/FIPR FLOTATION PROCESS FOR FLORIDA HIGH-MgO PEBBLE

FINAL REPORT

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JACOBS ENGINEERING GROUP INC. and LIANYUNGANG DESIGN AND RESEARCH INSTITUTE as subcontractors of IMC PHOSPHATES COMPANY

Prepared for

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PERSPECTIVE

Patrick Zhang, Research Director - Beneficiation & Mining

With the depletion of the higher grade, easy-to-process Bone Valley deposits, the central Florida phosphate industry has moved into the lower grade, more contaminated ore bodies from the Southern Extension. The phosphate deposits in the Southern Extension may be divided into two zones: an upper zone and a lower zone. The upper zone is readily processable using the current technology, but the lower zone is highly contaminated by dolomite.

Separation of dolomite from phosphate has been one of FIPR's top research priorities since its inception.

In 1997, the FIPR Board of Directors approved funding for the IMC proposal "Development of New Beneficiation Technology for Florida Dolomitic Phosphate Resources" (FIPR #97-02-129). The Chinese Lianyungang Design and Research Institute (CLDRI) was the major subcontractor of the project for laboratory development of a processing flowsheet for high-dolomite pebbles in Florida. As a result, a fine flotation process, the CLDRI process, was developed. In this process, the pebble sample is ground to suitable particle sizes for liberating dolomite and other impurities from phosphate. The ground slurry is then subject to dolomite flotation by using a mixture of H_3PO_4 and H_2SO_4 as pH modifier and PA-31 (a proprietary reagent) as dolomite collector. The sink product of dolomite flotation can be beneficiated by either silica or phosphate flotation. The table below summarizes the lab testing results.

Sample	Grind Size	Feed	Concentrate A	Analysis (%)	$%P_2O_5$
	-200 Mesh (%)	% MgO	BPL	MgO	Recovery
FLA-1	55	1.85	67.1	0.87	82.2
FLA-2	29	1.19	67.7	0.76	90.1
FLA-3	70	9.40	68.6	0.96	60.6
FLA-4	32	2.04	68.4	0.73	81.2
FLA-5	63	2.88	68.8	0.91	83.6

Summary of Lab Flotation Results on Five Dolomitic Pebble Samples.

Since the above lab testing results were so encouraging, with 20% higher recovery than most previously developed processes, the FIPR Board of Directors approved funding for pilot testing of the CLDRI fine flotation technology. As the next table indicates, the pilot testing achieved similar results to those from lab testing.

Sample	Feed % MgO	Concentrate Analysis (%) %P ₂ Reco			
	/0 10150	BPL	MgO	Recovery	
FLA-6	3.54	64.9	1.14	76.5	
FLA-7	2.81	63.4	0.81	81.8	

Summary of Pilot Testing Results on Two Dolomitic Pebble Samples.

Based on a preliminary economic analysis conducted by Jacobs Engineering, the total operating cost for the CLDRI flotation process is about \$15.60 per ton of final product, which is a few dollars less than that for processing the low-dolomite deposits in current Florida operations. This translates to an approximate profit of \$4 per ton of waste dolomitic pebble processed. It is therefore fair to say that FIPR has developed a viable process for the dolomitic phosphate resources in Florida.

ABSTRACT

The beneficiation process developed by the China Lianyungang Design and Research Institute was proven by pilot plant tests on two samples of high-MgO reject pebble using either laboratory tap water or recycled process water from a local beneficiation plant. Key components of the process include fine grinding and inverse flotation to remove the liberated gangue minerals. For the pebble samples tested, flotation to remove dolomite was followed by flotation to remove silica. The pebble samples were washed to remove clays prior to grinding and flotation, but were not deslimed after grinding.

In addition to the collectors, phosphoric acid, sulfuric acid, soda ash, and diesel fuel were used as flotation reagents. The dolomite collector was proprietary fatty acid soap developed by CLDRI. The silica collector was an amine, currently used in the cleaner flotation step of the Crago Process.

For Pebble #1 the 30-ton pilot plant sample had a higher MgO content than the small sample used for laboratory development tests (3.5 vs. 3.1%). Consequently dolomite flotation was more difficult in the pilot plant and both grade and recovery suffered slightly. The opposite situation occurred for Pebble #2 (2.8 vs. 4.3%), and the pilot plant performance was improved over the laboratory. The CLDRI beneficiation process produced concentrates with MgO contents less than one-third the MgO contents of the pebble samples treated in the pilot plant. More than 75% of the phosphate contained in the pebble was recovered as concentrate analyzing 63 to 66% BPL.

A preliminary study was performed to develop estimates of capital cost and operating cost for a battery limits beneficiation plant employing the CLDRI beneficiation process. The study was based on a plant capacity of 300 tph pebble containing 54% BPL and 2.00% MgO and 200 tph concentrate containing 66% BPL and 0.82% MgO. The constructed cost of the battery limits beneficiation plant was estimated to be 32 million dollars. Operating costs were estimated at \$15.62 per ton of concentrate.

ACKNOWLEDGMENTS

This research project was co-funded by the Florida Institute of Phosphate Research (FIPR) and IMC Phosphates Company (IMC) under contract number FIPR #99-02-133S, for which the authors are gratefully appreciative. The successful completion of the pilot plant program by the China Lianyungang Design and Research Institute (CLDRI) and Jacobs Engineering would not have been possible without the sponsorship, cooperation, and leadership provided by both the Florida Institute of Phosphate Research (FIPR) and IMC Phosphates Company (IMC).

CF Industries and IMC each provided a large sample of low-grade pebble and loaded the pebble onto dump trucks. FIPR made available laboratory facilities for CLDRI to produce and evaluate carbonate collectors from USA feedstock. IMC allowed access to their operations so that the chemicals and plant flotation reagents as well as the beneficiation plant "process" water used for the pilot plant tests could be obtained on a timely basis. An additional service provided by IMC was the independent evaluation of the pilot plant data to establish a formal material balance for each test run.

CLDRI and Jacobs gratefully acknowledge the assistance and excellent cooperation provided by CF Industries, the Florida Institute of Phosphate Research, and IMC Phosphates Company.

CLDRI gives special thanks to Dr. Clifford and Dr. Zhang of FIPR for their confidence in CLDRI and unwavering support since the laboratory development stage of the project. CLDRI also thanks Dr. Hwang of IMC and the Jacobs pilot plant personnel for their help in bringing the project to a successful conclusion.

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EXECUTIVE SUMMARY

The removal of dolomite from phosphate rock has been a high priority for investigations funded by the Florida Institute of Phosphate Research (FIPR). High-grade phosphate ore in the Bone Valley formation of Central Florida is being depleted and dolomite contamination is increasing as the mines advance to the South. Two mines exploiting the southern ore have installed heavy media plants to treat low-grade pebble; however, these plants have not been operated recently. Currently the dolomite problem is addressed by selective mining, blending, and by rejecting low-grade pebble.

In October 1997, the FIPR Board of Directors approved funding for a one-year program to develop a flotation process for beneficiating Florida dolomitic phosphate pebble. That program, managed by IMC Phosphates Company (IMC), involved laboratory research and testing by the China Lianyungang Design and Research Institute (CLDRI). The encouraging results of that program were presented in FIPR Publication No. 02-129-67. Five low-grade pebble samples containing from 1.2 to 9.4% MgO were tested by CLDRI. The fine particle flotation technology used by CLDRI recovered more than 80% of the phosphate as concentrate containing 0.7 to 1.0% MgO.

SCOPE

The FIPR Board of Directors consequently approved funding for a second oneyear program (FIPR #99-02-133S) to demonstrate the CLDRI Process in a pilot plant. IMC, the prime contractor and a sponsor for this program, subcontracted CLDRI and Jacobs Engineering Group, Inc (Jacobs) to perform the work. The contractual scope of work involved various tasks, as outlined below.

Sample Collection and Laboratory Flotation Tests

Four pebble samples were collected by IMC and dispatched to CLDRI in China for further laboratory work prior to pilot plant testing. CLDRI performed bench scale verification tests in China to determine preliminary reagent dosages and conditions for pilot plant operation.

Jacobs collected 50 tons of low-grade pebble, 15,000 gallons of plant process water, and various reagents for use in pilot plant testing. CLDRI provided 55 gallons of proprietary reagent (PA-31), and prepared similar reagents from locally available feed stocks.

Pilot Plant Design and Assembly

CLDRI provided a process diagram and design parameters. Jacobs sized and selected process equipment to achieve the process objectives. Jacobs made the equipment arrangement and plant layout, and erected the pilot plant.

Pilot Plant Operation

About four weeks of informal testing was conducted to establish procedures for operating, sampling, and safety training. Five weeks of formal testing was conducted on Pebble #1 and three weeks of formal testing was conducted on Pebble #2. CLDRI identified test conditions and operated the flotation circuits. Jacobs operated the remainder of the pilot plant and provided flotation reagents at the desired concentration and flow rate. Sampling, sample preparation, and sample analyses were performed by Jacobs. After review and approval by Jacobs and CLDRI, the measured data for each formal run were submitted to IMC for independent determination of process efficiencies.

Report Writing

CLDRI and Jacobs prepared the report describing the pilot plant program and test results. Jacobs estimated capital and operating costs for a commercial plant utilizing the CLDRI Process.

METHODOLOGY

Eleven formal pilot plant runs were completed on Pebble #1 from IMC's Four Corners mine. The use of plant process water for dolomite and silica flotation was tested in two runs. Other parameters examined were reagent dosage, fineness of grind, and mechanical modifications to the carbonate flotation cells. Eight formal pilot plant runs were completed on Pebble #2 from the Hardee Complex II, owned and operated by CF Industries. The use of plant process water for dolomite and silica flotation was tested in one run, and a dolomite collector made locally was tested in one run. Other parameters examined were reagent dosage, fineness of grind, and mechanical modifications to the carbonate flotation cells.

RESULTS

The CLDRI Process is characterized by fine grinding without subsequent desliming, followed by dolomite flotation and an optional silica flotation. A desliming step before grinding was incorporated in the pilot plant; however, CLDRI considered this as optional for Pebble #1 and mandatory for Pebble #2. Two stages of desliming were

performed on Pebble #2 because of high clay content. Flotation results were improved when the ground flotation feed contained more than 90% passing 100 mesh.

Dolomite flotation required three reagents: sulfuric acid for pH control, phosphoric acid for phosphate depression, and PA-31 for dolomite collection. These three reagents accounted for about 80 to 90% of the total reagent cost. Dolomite flotation was not particularly sensitive to the use of plant water. The proprietary PA-31 dolomite collector is not currently commercially available in the USA. This Chinese reagent was synthesized from vegetable fatty acids, a base, and a surfactant, all available in the USA. A pilot plant test with FA-#4, the most promising of the synthesized collectors produced a concentrate with higher grade but lower recovery than PA-31.

Silica flotation also required three reagents; soda ash for pH adjustment, amine for silica collection, and diesel fuel (No. 2 D.O.) as an extender. The use of plant water reduced the amount of silica floated, unless the amine dosage was increased.

Laboratory test results are reliable indicators of pilot plant results. The CLDRI Process recovered 76 to 82% of the phosphate, which is improved over other dolomite rejection processes that require desliming of the ground flotation feed. The BPL and acid insoluble (A.I.) contents of the recovered concentrates were acceptable. Even though the MgO contents of the concentrates were reduced to less than one third of the pebble MgO, the overall concentrate quality was marginal because of iron and aluminum oxides (I&A). The minor element ratio and the ratio of calcium oxide to P_2O_5 are elevated for both concentrates.

Reagent costs, assuming a delivered price of \$0.30 per pound of PA-31, were in the range of four to five dollars per ton of concentrate. A preliminary study of a beneficiation plant using the CLDRI Process provided estimates of constructed cost and operating cost. For a battery limits plant capable of producing 1.6 million tpy concentrate, the estimated constructed cost is 32 million dollars (\$20 per annual ton). The estimated operating cost of \$15.62 per ton of concentrate is lower than the average cost of phosphate rock.

The pilot plant program demonstrated the technical and economic suitability of the CLDRI Process for beneficiating dolomitic pebble. Additional work to develop a Florida version of PA-31 is warranted. This key reagent controls process performance and contributes 8% of the operating cost.

Pilot plant results, selected from one test run for each low-grade pebble sample treated, are summarized on the following page.

	Pebble #1, Run 18			Pebble #2, Run 9B				
	% Weight	% BPL	% MgO	% A.I.	% Weight	% BPL	% MgO	% A.I.
Washings ⁽¹⁾	-	-	-	-	22.0	11.4	5.86	51.0
Clay O'flow	4.7	36.2	7.71	6.8	2.7	30.2	7.95	13.1
Dolomite Tail	27.2	32.9	9.07	4.0	9.8	32.0	8.66	9.8
Silica Tail	7.8	17.4	0.29	74.0	11.4	10.3	0.13	83.9
Concentrate	60.3	64.9	1.14	4.6	54.1	63.4	0.81	9.3
Composite	100.0	51.2	3.54	9.9	100.0	41.9	2.81	27.2
% Recovery to								
Concentrate	60.3	76.5	19.4	27.8	54.1	81.8	15.6	18.6
Concentrate								
MER ⁽²⁾	0.125				0.116			
Concentrate								
$CaO:P_2O_5^{(3)}$	1.558				1.550			
Reagent Cost								
\$/t Concentrate	4.32				2.94			

CLDRI Process Pilot Plant Results Summary

(1) Pebble #2 was deslimed twice. "Washings" were removed by the first desliming, and "Clay O'flow" was removed by the regular log washing step.

- (2) MER = minor element ratio $(MgO+Fe_2O_3+Al_2O_3)/P_2O_5$
- (3) $CaO:P_2O_5 = CaO/P_2O_5$

INTRODUCTION

RESEARCH OBJECTIVE

The main objective of this project is to demonstrate, on pilot scale, the technical and economical feasibility of the fine particle flotation technology developed by CLDRI (Chinese Lianyungang Design and Research Institute). Encouraging bench scale tests previously conducted by CLDRI, funded by FIPR, and led by IMC prompted the pilottesting program.

The previous work (IMC-Agrico 1999) demonstrated the technical feasibility of the CLDRI fine particle flotation technology. A concentrate analyzing greater than 66% BPL and less than 1% MgO was obtained from dolomitic phosphate pebble with an overall phosphate recovery of more than 80%.

The CLDRI fine particle flotation technology for beneficiation of high magnesium phosphate ore has the following features:

- **Grinding for liberation:** The low-grade pebble must be subjected to grinding to liberate phosphate from carbonate. The fineness of grinding depends on the grain sizes of the interlocked phosphate and carbonate minerals. Since the flotation concentrate can be directly used as feed to the chemical plant, the grinding cost at the acidulation plant is avoided.
- **Non-desliming after grinding:** The process flowsheet has been made relatively simple by eliminating the desliming step after grinding but prior to dolomite flotation. This improves flotation recovery and lowers production costs relative to other flotation processes which incorporate the desliming step.
- **High performance:** High-quality phosphate concentrates are achieved because both carbonates and silicates are rejected. High phosphate recovery is maintained because desliming losses are minimized and flotation performance is high.

HISTORICAL PERSPECTIVE

In 1997, the FIPR Board of Directors approved funding for the proposal "Development of New Technology for Beneficiation of Florida Dolomitic Phosphate Resources" (FIPR contract #97-02-129), jointly submitted by IMC and CLDRI. In February 1998, CLDRI received five dolomite pebble samples collected by IMC. Extensive laboratory development work was carried out at CLDRI through the end of the year. The chemical components and flotation test results on the as-received samples are given in Tables 1 and 2 respectively.

Sample ID	Sample Source	% BPL	% MgO	% A.I.
FLA-1	Ft. Green pebble #1	54.17	1.85	19.48
FLA-2	Ft. Green pebble #2	56.68	1.19	19.20
FLA-3	Ft. Green pebble #3	32.67	9.40	11.06
FLA-4	Clear Springs pebble	52.12	2.04	20.20
FLA-5	Kingsford pebble	55.46	2.88	12.24

Table 1. Analyses of Test Pebble Samples.

Table 2.	Summary	of Flotation	Results.
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	Process ⁽¹⁾	Concentra		
Sample ID		% BD I	% MgO	BPL %
		70 DF L		recovery
FLA-1	G-Df-S-Pf	67.1	0.87	82.2
FLA-1	G-Df-S-Sf	68.4	0.91	81.7
FLA-2	G-S-Df-Sf	67.7	0.76	90.1
FLA-2	G-Df-S-Sf	58.4	0.78	90.8
FLA-3	G-Df-Sf	68.6	0.96	60.6
FLA-4	G-Df-S-Pf	68.4	0.73	81.2
FLA-5	G-Df-S-Sf	68.2	0.93	84.2

(1) G = grinding, S = sizing, Df = dolomite flotation, Pf = phosphate flotation, Sf = silica flotation

The above test results confirmed that CLDRI's fine particle flotation technology could be successfully used in processing Florida dolomitic phosphate pebble. Consequently, approval was granted for pilot plant testing of the "G-Df-S-Sf" process on two pebble samples, one from IMC, and one from CF Industries.

In November 1999, CLDRI received four pebble samples shipped by IMC. Chemical analyses of the four dolomitic phosphate samples are shown on Table 3. Samples FLA-6 and FLA-9 had been pre-selected for the pilot plant test program. Samples FLA-7 and FLA-8 were refractory materials added for general interest.

Bench scale testing on these samples was completed in February 2000. The "Grinding-Dolomite flotation-Sizing-Silica flotation" process was tested on the asreceived samples. The demonstration test results are summarized in Table 4, and test conditions are given in Table 5.

Sample ID	Sample Source	% BPL	% MgO	% A.I.
FLA-6	IMC Four Corners, 579 lbs.	48.70	3.11	18.30
FLA-7	IMC Pine Level, 28.8 lbs.	47.96	2.24	13.77
FLA-8	Farmland Industries, 451 lbs.	48.70	3.97	14.33
FLA-9	CF Industries, 403 lbs.	42.36	4.35	20.98

Table 3.	Analysis of	Test Pebble	Samples for	Verification	Test.
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Table 4. Verification Test Results in CLDRI's Lab.

Sample ID		Concentrat		
	Process ⁽¹⁾	% BPL	% MgO	BPL % recoverv ⁽²⁾
FLA-6	G_Df_S_Sf	66.10	0.08	79.62
TLA-0	0-01-0-01	00.10	0.98	17.02
FLA-7	G-Df-S-Sf	60.81	0.99	70.28
FLA-8	G-Df-S-Sf	61.57	1.41	72.48
FLA-9	D-G-Df-S-Sf	66.12	0.93	80.51

- (1) G = grinding, S = sizing, Df = dolomite flotation, Pf = phosphate flotation, Sf = silica flotation, D = desliming
- (2) Overall BPL Recovery

Since samples FLA-6, FLA-7 and FLA-8 contained less than 5% clay they were not deslimed prior to grinding. FLA-9 had more than 10% clay and was the only sample scrubbed and deslimed at 150 mesh before being ground for flotation.

Sample Grou ID %-20	Ground -feed	Reagent Consumption (lb./t flotation feed)						
		H.DO.	<u>ц со</u>	DA 21	No CO	Amina	No. 2	
	70-200 mesn	1131 04	112504	1 A-31	Na ₂ CO ₃	Amme	D.O.	
FLA-6	61.6	8.0	4.0	3.2	0.6	0.6	0.15	
FLA-7	60.0	6.0	4.0	3.4	0.6	0.8	0.15	
FLA-8	55.3	6.0	4.0	3.8	0.6	0.8	0.15	
FLA-9	50.0	6.0	4.0	2.8	0.6	0.6	0.15	

The major carbonate mineral in FLA-7 (from Pine Level) is calcite not dolomite. A concentrate obtained from this sample analyzed less than 1% MgO at 60.81% BPL with an overall BPL recovery of 70.28%. Further research on FLA-7 is needed to identify an ideal collector and to adopt the process for calcite removal.

As is shown on Table 4, an acceptable phosphate concentrate was not obtained for sample FLA-8. Mineralogical characterization studies of the flotation concentrate utilizing polarized light microscopy indicated the dolomite was mostly locked with phosphate in two forms. A combination of flotation and chemical extraction would be required for sample FLA-8 because:

- a) Dolomite and phosphate are disseminated in each other in sizes of 10-20 microns,
- b) Dolomite is coated on phosphate particles in the size range of 20-30 micron.

The final concentrates with BPL more than 65.6%, MgO less than 1%, and overall BPL recovery of 80% were obtained from the demonstration tests on FLA-6 and FLA-9 using the "Grinding-Dolomite flotation-Sizing-Silica flotation" flowsheet. The pilot plant sample corresponding to FLA-6 and FLA-9 are identified as Pebble #1 and Pebble #2 respectively.

PERTINENT LITERATURE AND RELATED WORK

Phosphate rock, as a strategic commodity for the fertilizer industry, plays an important role in agriculture. It is estimated that over 80% of the phosphate rock output in the world is used for fertilizer production. The United States is one of the world's largest producers of phosphate rock. Its phosphate production represents approximately one third of the world's total, while about 70% of the U.S. production comes from Florida.

Unfortunately, the high-grade siliceous phosphate ore from the Bone Valley formation is being depleted rapidly. It is estimated that the Florida phosphate reserves that can be economically processed with available technology may only last about 20 years at the current mining rate. As phosphate mining moves further south and southeast, the phosphate matrix will be leaner in grade and higher in dolomite. Although one company now uses heavy media separation technology to process a portion of the dolomite contaminated phosphate, the dolomitic portions of the ore are bypassed in most cases. To the best of our knowledge, no flotation process has proven to be economically feasible for Florida dolomitic phosphate ores.

Among the deleterious materials (Fe₂O₃, Al₂O₃, CaO, MgO, F) in the feed for phosphoric acid production, MgO is perhaps the most common and problematic. Phosphate rock contaminated with carbonate gangue consumes more sulfuric acid per ton of the P_2O_5 produced in the wet phosphoric acid process than non-contaminated phosphate rock. In addition, MgO increases acid viscosity and reduces the filtering rate of acid. Also, MgO contributes to the formation of sludge when the phosphoric acid is clarified.

The Florida Institute of Phosphate Research conducted a comparative evaluation of four seemingly promising flotation processes for separating dolomite from phosphate (El-Shall 1994). All the processes were evaluated on the same feed, which was a pebble sample analyzing 56.8% BPL and 2.2% MgO. These processes included the University of Florida two-stage conditioning process, the U.S. Bureau of Mines fluosilicic acid process, the IMC cationic process, and the TVA diphosphonic acid process. As is shown in Table 6, three of the processes did not reduce concentrate MgO below 1% and all the processes gave relatively poor overall phosphate recovery ranging from 30 to 60%. Flotation reagent costs were over \$ 2.00 per ton of concentrate in most cases.

Drocoss	Concent	Concentrate %		ery %	Reagent
FIOCESS	BPL	MgO	Flotation	Overall ⁽²⁾	\$/t Conc.
IMC cationic	68.4	0.79	85	59	2.68
USBM	67.7	1.34	59	41	2.38
Univ. of Florida	68.8	1.04	55	36	4.25
TVA	67.1	1.46	97	66	1.93

Tabla 6	Summany of Dolomita Flatation Dragona (1)	.)
I able 0.	Summary of Dolomite Flocation Frocesses.	

(1) Pebble assay: 56.8% BPL, 2.2% MgO. (El-Shall 1994)

(2) Includes feed preparation and desliming in addition to flotation.

One of the major problems with the previously developed dolomite flotation processes is the significant loss of phosphate values due to desliming after grinding. The CLDRI laboratory development work therefore focused on developing a process involving flotation in the presence of slimes.

PROJECT SCOPE

Project Team

The research program examined a flotation process developed by the Chinese Lianyungang Design and Research Institute (CLDRI) to beneficiate low-grade phosphate pebble from Florida. The Florida Institute of Phosphate Research (FIPR) sponsored the project and awarded the prime contract to IMC Phosphates (IMC). The project team comprised personnel from FIPR and IMC, and the two companies subcontracted to perform the research. CLDRI was subcontracted to provide process technology. Jacobs Engineering Group Inc (Jacobs) was subcontracted to set up and operate a pilot plant incorporating the CLDRI Process. An organization chart showing the project team is given in Figure 1.



Figure 1. FIPR #99-02-133S, Project Organization Chart.

Project Tasks

CLDRI and Jacobs Engineering (Jacobs) subcontracted to IMC to perform the following approved tasks.

Sample Collection and Laboratory Flotation Tests

IMC dispatched four high-MgO pebble samples to CLDRI for verification testing. These 500-pound samples were collected from IMC, CF Industries, and Farmland Hydro. One objective of the verification tests was to test the two samples pre-selected for pilot plant testing. CLDRI performed verification bench scale flotation tests in China to establish preliminary reagent dosages and test conditions for the pilot plant operation.

Following the verification tests, Jacobs collected large amounts of the two high-MgO pebble samples, as well as beneficiation plant water, and all reagents for pilot plant flotation, except dolomite collector PA-31 which was provided by CLDRI. The samples collected for pilot plant testing are listed below:

- Pebble #1: 30 tons from the Four Corners mine
- Pebble #2: 20 tons from Hardee Complex II
- Kingsford plant water: 3 tanker trucks (15,000 gallons)
- New Wales phosphoric acid: 60 gallons @ 52% P₂O₅
- Kingsford plant reagents:
 - Sulfuric acid: 30 gallons of 98% solution
 - Soda ash: 100 gallons of plant solution
 - Amine: 5 gallons
 - No. 2 Diesel Oil (No. 2 D.O.): 5 gallons

Pilot Plant Design and Assembly

CLDRI provided Jacobs with a flow diagram showing the unit operations and specified process conditions for the pilot plant. Jacobs configured the unit operations, selected the equipment, designed, and assembled the pilot plant at their Lakeland laboratory. The design was reviewed and mutually agreed upon by IMC, FIPR, CLDRI, and Jacobs.

The pilot plant was originally proposed to be operated by IMC over a six-month period at the Four Corners Mine. Subsequently, the pilot plant operation was subcontracted to Jacobs. During subcontract negotiations some scope changes were proposed and agreed to by all parties. A potentially negative aspect of the agreed upon changes was that the time frame available for CLDRI to optimize the process for each sample was reduced.

Pilot Plant Operation

CLDRI engineers established the parameters to be tested in each pilot plant run. Jacobs scheduled the work and furnished labor and supplies as necessary to achieve the target parameters for each test run.

CLDRI engineers operated the carbonate and silica flotation cells. Jacobs personnel operated all other components of the pilot plant, as well as sampling, sample preparation, and chemical analysis of the samples.

CLDRI engineers produced and tested small quantities of carbonate collector (PA-31 type material) in FIPR's laboratory using available USA chemical feedstock. One of the produced materials (FA-#4) was tested in Run 15. The other pilot plant runs used PA-31 produced in China. PA-31 was developed by CLDRI, and is used for commercial dolomite flotation in China (Zhizhong and Zhengxing 1999). The raw materials used to produce PA-31 are vegetable fatty acids and a surfactant, all of which are available in the United States.

Report Preparation

Jacobs and CLDRI jointly prepared a report describing the comprehensive pilot plant program. The general responsibilities for report writing were assigned as follows:

- CLDRI described the research objective and project background, discussed the results of the process testing, and presented an approach for using the process on Florida pebble.
- Jacobs described the pilot plant operation, presented the results of pilot plant testing, and prepared estimates of the capital and operating costs.

Schedule

Following the award of Jacob's subcontract, the project required 39 weeks to complete. Work commenced in May 2000 and continued through January 2001. The tasks on the critical path and their duration are listed below:

Project Task	Duration
Mobilization	2 weeks
Pilot plant design and parts delivery	12 weeks
Pilot plant assembly and erection	6 weeks
Pilot plant debugging with pebble #1	3 weeks
Pilot plant operation on pebble #1	5 weeks
Pilot plant operation on pebble #2	3 weeks
Decommission and sample dispatch	2 weeks
Final Report Preparation	6 weeks
Total Duration	39 weeks

Four weeks were scheduled for the formal testing of each pebble sample. However, the target performance for Pebble #2 was achieved within three weeks, allowing additional testing of the more refractory Pebble #1. Eleven formal test runs were performed on Pebble #1, and eight formal test runs were performed on Pebble #2.

METHODOLOGY

PILOT PLANT

Process Description

The pilot plant is designed to process low-grade pebble at a controlled feed rate using either tap water or beneficiation plant process water from tanker trucks. Laboratory tap water was used for 16 runs and beneficiation plant process water was used for three runs.

The pilot plant flowsheet includes a pretreatment step and three basic steps of the CLDRI process, which are listed below:

- Pretreatment: Optional desliming to eliminate high surface area clays prior to reagentization and flotation.
- 1. Particle size reduction to liberate phosphate from gangue minerals, to reduce the dolomite to a readily floatable size.
- 2. Conditioning with a carbonate reagent suite, followed by inverse flotation to remove dolomite.
- 3. Conditioning with amine reagent, followed by inverse flotation to remove quartz.

A flowsheet showing the pilot plant configuration is given in Figure 2. An alphanumeric system is used to designate equipment items by prefix E and sample points by prefix S.

A listing of pilot plant equipment used for the CLDRI Dolomite Flotation Process is presented in Appendix A. Listed equipment items are identified by equipment numbers that correspond to the equipment shown on Figure 2.

A backhoe reclaims either Pebble #1 or Pebble #2 from their respective piles and dumps the material into a hopper feeding the belt conveyor. Belt conveyor (E1) transfers low-grade pebble from ground level to an elevated surge bin (E2). Low-grade pebble is withdrawn from the surge bin at a controlled rate by belt feeder (E3) and discharged into a roll crusher (E4) that breaks oversize pebble into particles that are less problematic for small-scale equipment. Bucket elevator (E5) lifts the pebble from ground level to feed the log washer (E6) on the upper floor of the pilot plant.

The log washer removes clay from the pebble by attrition and washing. Deslimed pebble is discharged from the log washer and flows by gravity to the rod mill (E7). Clays and any feed washed from the pebble are sized on a vibrating screen (E18) to scavenge

particles coarser than 270 mesh. The -270 mesh material is discarded as waste. The +270 mesh material is combined with deslimed pebble and fed to the grinding circuit, that includes an open circuit rod mill and a closed circuit ball mill.

Two-stage grinding reduces pebble to the particle size distribution required for flotation. An open circuit rod mill (E7) selectively grinds the coarser pebble and provides the first stage of grinding. The rod mill discharges into pump tank (E8) and mill pump (E9) transfers partially ground slurry from grade to the mill classifier (E11) on the pilot plant upper floor. Particles too coarse to pass through the mill classifier (screen) flow by gravity to the ball mill (E10) for the second stage of grinding. The ball mill is in closed circuit with the screen. Slurry passing through the screen at 30 to 35% solids by weight flows to the carbonate conditioning tanks (E12).

Sulfuric acid for pH control, phosphoric acid for phosphate depression, and PA-31 for carbonate collection are metered into the carbonate conditioning tanks (E12) and mixed with the slurry. The conditioned slurry flows into carbonate flotation cells (E13) where mechanically dispersed air bubbles attach to reagent coated carbonate particles. The bubbles with attached particles (carbonate tailing) form froth, which is skimmed from the cell surface and discarded.

The cell product from carbonate flotation, which is enriched in phosphate, is transferred to the concentrate screen (E15) on the upper floor of the pilot plant by the concentrate pump (E14). The screen classifies the concentrate at nominally 400 mesh. The -400 mesh concentrate bypasses silica flotation to avoid phosphate recovery losses and to reduce reagent consumption. The +400 mesh material flows by gravity to the silica conditioning tanks (E16) for mixing with soda ash, amine, and No. 2 D.O.. The conditioned slurry flows into silica flotation cells (E17) where mechanically dispersed air bubbles attach to reagent coated quartz particles. The bubbles with attached particles (silica tailing) form froth, which is skimmed from the cell surface and discarded. The cell product from silica flotation comprises the +400 mesh concentrate.

Five process streams exit the pilot plant, two of which are concentrates and three of which are tailings. It is probable that the -400 and +400 mesh concentrates would be combined for dewatering and transport; however, for pilot plant operation they were accounted for separately.



Figure 2. Flow Diagram for Jacobs Pilot Plant.

Operation

The pilot plant work schedule is 10 hours per day, Monday through Thursday. Formal test runs were normally performed Mondays and Wednesdays, allowing sample preparation, chemical analyses, and data review to be performed Tuesdays and Thursdays. In this way it was possible to establish the conditions for each test knowing the results of the previous test. The off days were also utilized to prepare and restock flotation reagents.

CLDRI engineers operated the carbonate and silica flotation cells. Jacobs personnel maintained the supplies of low-grade pebble, water, and reagents required for testing, and operated all equipment except the flotation cells. Operator duties are listed below:

- Feed Operator. Load pebble with backhoe. Monitor operations of belt conveyor, belt feeder, and roll crusher. Check feed rate. Maintain house-keeping.
- **Mill Operator.** Monitor operations of bucket elevator, log washer, scavenger screen, rod mill, ball mill, mill pump, and mill screen.
- **Reagent Operator.** Maintain inventory of reagents, check flow rates, and adjust pump settings in cooperation with flotation operators. Measure and record pH levels in carbonate and silica flotation cells. Record readings from water flow meters.
- Flotation Operators. Maintain levels in carbonate and silica flotation cells. Observe appearance of froths, and microscopically examine concentrates and tails samples. Direct Jacobs personnel to modify operating conditions (mesh of grind, slurry percent solids, flotation cell tip speed, reagent dosages, etc.) to investigate the CLDRI process.

Reagents

Reagents utilized in the CLDRI dolomite flotation process are described below in order of the sequence of addition.

Sulfuric and Phosphoric Acids

These reagents were added to the first carbonate-conditioning tank to adjust the slurry pH and to depress the phosphate mineral during the subsequent carbonate flotation step. The P_2O_5 :SO₄ ratio, an important process variable, was examined over the range of 1:0 to 0:1. For the pilot plant operation 98% sulfuric acid and 52% phosphoric acid were mixed at the desired ratio, diluted with tap water to about 20%, and metered with a single pump.

PA-31

This proprietary reagent has been developed by CLDRI. FIPR, IMC, and Jacobs have signed confidentiality agreements concerning PA-31. This reagent is added to the carbonate conditioning tanks and to the carbonate flotation cells as a 7% solution.

Numerous tests showed that PA-31 is an effective collector, which can successfully separate dolomite from phosphate with a high BPL recovery from 100 mesh to micron-sized feed (free of clay). In most of other dolomite flotation processes, phosphate recovery was usually very low due to the removal of either -100 or -150 mesh fraction from ground flotation feed.

The manufacturing process for PA-31 is very simple. There are no wastes and no environmental problems during its production. Material Safety Data Sheets for PA-31 are provided in Appendix B. This dolomite collector developed by CLDRI is currently in commercial use in China. Its raw materials are vegetable fatty acids and a surfactant, which are available at reasonable prices in the US market.

Soda Ash

This reagent (at 15% solution) is added to the + 400 mesh concentrate prior to the first silica conditioning tank. The soda ash solution adjusts the slurry to neutral pH for silica flotation.

Amine

Amine solution (at 2% solution) is added to the first silica-conditioning tank. Amine is a collector for silica (quartz).

No. 2 Diesel Oil

The diesel oil is utilized as an extender for amine, and is added to the first silicaconditioning tank.

Sampling

The sampling program involved manually cutting samples at twelve (12) pilot plant sampling stations. Sample numbers in Figure 2 identify the location of the sampling stations listed below.

Sample Number	<u>Type</u>	Description	Classification
S1	Input	Log Washer Feed	Pebble feed
S2	Output	Log Washer Overflow	Slurry
S2	Internal	Rod Mill Discharge	Slurry
S4	Internal	Mill Classifier Oversize	Slurry
S5	Internal	Mill Classifier Feed	Slurry
S 6	Internal	Flotation Feed	Slurry
S 7	Output	Carbonate Tails	Slurry
S 8	Internal	Carbonate Concentrate	Slurry
S 9	Output	Fine Concentrate	Slurry
S10	Internal	Silica Flotation Feed	Slurry
S11	Output	Silica Flotation Tails	Slurry
S12	Output	Silica Flotation Concentrate	Slurry

Slurry sampling stations were each configured to allow diversion of the entire process stream into a 5-gallon bucket. The pebble feed was collected in a pan directly from the belt feeder discharge. Each sample cut time for all "input" and "output" samples was measured with a stopwatch, and ranged from 10 to 30 seconds depending on the stream rate. The interval between rounds of samples was 30 to 60 minutes. For each sample round the sample cut time and pulp weight were recorded on a log sheet. The samples were wet screened on a 270-mesh screen. For each sample the -270 mesh fraction was flocculated, carefully decanted, and combined into a -270 mesh composite bucket. Similarly, the +270 mesh fraction of each sample were put into an oven to dry.

The sample cut times for "internal" samples were not routinely measured and were intentionally minimized to avoid disruption of the pilot plant operation. The sampling interval was also between 30 to 60 minutes. At the end of a test, each "internal" composite slurry sample was weighted and wet screened using a 270-mesh screen. The +270 mesh and -270 mesh fractions were dewatered and put into the oven to dry.

The samples were usually dried overnight in the oven at 105° C. The dry weights of the +270 and -270 mesh fractions for each sample were determined and representative portions of each were cut out and prepared for chemical analysis. The remaining +270 mesh fraction was screened on a series of sieves, usually 48, 65, 100, 200 and 270 mesh, for size distribution determination.

Data consisting of sampling times, pulp weights, dry solids weights of +/-270 mesh, chemical analysis of the +/-270 mesh, and size distribution data of the +270 mesh were input into a an Excel spreadsheet developed specifically for this project. The sample stream percent solids, flow rates (solids, water, slurry and chemical components), weighted chemical analyses (BPL, MgO and A.I.), distributions of particle size, weight, BPL, MgO and A.I. were calculated.

Chemical Analyses

The Association of Florida Phosphate Chemists (AFPC) approved methods were used to analyze the samples. The referenced procedures from the AFPC manual are listed below:

P2O5 (BPL):AFPC photometric method (page 11-10)Acid Insoluble (A.I.):AFPC gravimetric method (page 9-8)MgO:AFPC atomic adsorption method (page 11-28)

Slurry pH measurements were obtained using hydrogen ion electrodes.

Matbal

Measured flow rates, chemical analysis, and sieve analysis data for process streams were transmitted to IMC for input to their Matbal program. Matbal is used by IMC to statistically convert measured data into a coherent data set, so that output is exactly equal to input for each parameter at each step of the process. Measured data include normally distributed sampling and analytical errors that usually preclude perfect closure of all parameters in a material balance. The Matbal program provides an unbiased method of correcting the data to arrive at 100% closure for all parameters.

A second advantage of the Matbal program is that the magnitude of adjustment required to arrive at 100% closure for all parameters is evident. Large adjustments indicate biased data or that process conditions were not at equilibrium. Small adjustments indicate reliable sample data. Therefore the confidence in test results may be high if the Matbal output shows only small adjustments to the measured data.

Matbal input for each formal test run comprised measured parameters for key streams. The 12 key streams are identified as S1 through S12 in Figure 2, and are described above under **Sampling**. Streams S1, S2, and S3 have inputs for solids rate and chemical analysis only. Streams S4 through S12 have inputs for solids rate, chemical analysis, and particle size analyses. Copies of the transmitted data for each pilot plant run are presented in Appendix C.

PRELIMINARY STUDY

Assumptions

A preliminary study of the CLDRI dolomite flotation process for low-grade phosphate pebble is included in this report. The assumed bases for the engineering study are listed below:

Design Rate: 300-stph dry basis

Low-Grade Pebble Analyses: Moisture content 12% by weight

Particle size	100% passing ³ / ₄ inch
	80% passing 2 ¹ / ₂ Tyler mesh

Process Recoveries:

Circuit	BPL	MgO	A.I.	I&A
Desliming	97.4%	82.1%	97.4%	76.4%
Carbonate Flotation	89.8%	36.2%	93.2%	89.8%
Silica Flotation	95.2%	93.3%	10.5%	95.2%
Overall Process	84.4%	27.5%	22.3%	66.2%

RESULTS

PEBBLE #1

Eleven formal pilot plant runs were performed with this sample. The target parameters for each run were specified by CLDRI. Parameters examined during these runs included reagent dosage, dolomite flotation cell agitator speed, dolomite flotation cell froth paddle speed and clearance, mesh of grind, and flotation with tap water and plant water.

Although the sample clay content was low, the log washer was used and some fine waste was rejected.

A summary of results from the runs is presented on Table 7. The ground flotation feed ranged from 2 to 22% plus 100 mesh and 77 to 57% passing 200 mesh. Dolomite flotation was performed with dosage of PA-31 that ranged from 3.00 to 5.58 lb./ton of pebble feed to the pilot plant. The yield of combined wastes ranged from 32.8 to 50.9% of the pebble weight; however, the phosphate content of the combined wastes was relatively uniform at 30.2 to 34.7% BPL.

The yield of combined concentrates ranged from 49.1 to 67.2% of the pebble weight. At the low yield the concentrate contained 64.5% BPL and 1.06% MgO. At the high yield the concentrate contained 62.1% BPL and 1.25% MgO.

A flow diagram and material balance for each of the 11 formal runs is given on Figures 3 through 14 respectively. The solids rates and chemical analyses on these diagrams are adjusted data, independently developed by IMC's Matbal Program, based on measured sample data.

Although the composite concentrate yield tended to hover around 60%, the proportions of fine and coarse concentrate fluctuated considerably, with the fine:coarse ratio ranging from 0.33 to 0.85. The fine concentrate has a lower MgO content and a higher A.I. content than the coarse concentrate.

Test No.	1	2	3	4	5	6	7	8	16	17	18
PA-31 Source	China										
Water	City	City	City	City	City	Plant	City	Plant	City	City	City
Dosage, lb/t Feed											
P_2O_5	6.70	5.83	5.89	8.17	9.08	8.35	8.70	11.67	9.34	11.16	12.09
H_2SO_4	4.15	4.02	4.05	4.35	4.58	4.18	4.49	5.79	4.68	0.64	0.69
PA-31	4.06	3.89	3.76	3.73	4.04	4.02	3.00	4.58	4.09	3.95	4.22
Soda Ash	0.57	0.52	0.57	0.63	0.79	1.27	0.43	0.86	0.94	0.57	0.57
Amine	1.07	1.03	0.96	0.80	0.97	0.97	0.84	0.94	1.04	0.91	0.91
No. 2	0.25	0.24	0.21	0.23	0.31	0.31	0.22	0.38	0.34	0.18	0.19
Flotation Feed Particle Size											
> 100 Mesh	13%	10%	11%	13%	13%	16%	2%	2%	22%	8%	8%
< 200 Mesh	61%	69%	63%	61%	61%	58%	77%	76%	57%	70%	69%
Pebble Feed											
% BPL	50.71	51.09	51.65	51.52	51.39	51.54	51.81	51.19	51.76	51.26	51.15
% MgO	3.97	3.57	3.41	3.53	3.59	3.68	3.98	3.62	3.61	3.52	3.54
% A.I.	11.04	10.46	10.76	10.81	10.98	10.58	10.16	10.75	10.58	10.37	9.96
Composite Waste											
Yield, % Weight	39.49	39.95	37.68	32.80	39.12	37.76	39.75	50.91	33.35	41.58	39.73
% BPL	31.60	31.66	31.67	30.07	31.09	34.65	33.89	38.48	30.75	33.12	30.27
% MgO	7.32	6.94	6.53	8.21	7.36	7.78	8.18	6.09	7.72	6.90	7.19
% A.I.	21.68	18.83	21.86	16.90	19.61	14.11	16.73	15.81	19.53	17.70	18.11
BPL % Distribution	24.61	24.76	23.11	19.14	23.67	25.38	26.00	38.27	19.81	26.87	23.51
Composite Concentrate											
Yield, % Weight	60.51	60.05	62.32	67.20	60.88	62.24	60.25	49.09	66.65	58.42	60.27
% BPL	63.20	64.01	63.72	61.97	64.42	61.82	63.60	64.39	62.26	64.17	64.91
% MgO	1.78	1.32	1.52	1.25	1.17	1.19	1.21	1.06	1.56	1.12	1.14
% A.I.	4.09	4.89	4.05	7.84	5.43	8.44	5.83	5.50	6.10	5.16	4.59
BPL % Distribution	75.40	75.24	76.88	80.82	76.31	74.64	73.97	61.74	80.17	73.14	76.48

Table 7. Summary of Pebble #1 Pilot Test Results.


Figure 3. Run 1 Mass Balance.



Figure 4. Run 2 Mass Balance.



Figure 5. Run 3 Mass Balance.



Figure 6. Run 4 Mass Balance.



Figure 7. Run 5 Mass Balance.



Figure 8. Run 6 Mass Balance.



Figure 9. Run 7 Mass Balance.



Figure 10. Run 8 Mass Balance.



Figure 11. Run 16 Mass Balance.



Figure 12. Run 17 Mass Balance.



Figure 13. Run 18 Mass Balance.

PEBBLE #2

Pebble #2 was log washed twice because it was heavily contaminated with clay. The first pass through the log washer was made before the formal testing to remove sticky clay that would subsequently cause choking problems in the pilot scale equipment. As Table 8 shows, the first stage of log washing rejected 22% of the pebble weight as a low-grade waste product. No scavenger screen was used to recover low-grade feed from the log washer over flow. The second log washing was performed as a pretreatment during formal testing, as previously shown in Figure 2.

	% Weight	% BPL	% MgO	% A.I.
Log Overflow	22	11.41	5.86	50.97
Washed Pebble ⁽¹⁾	78	49.60	2.10	21.20
Calculated Head	100	41.21	2.93	27.75
Sampled Head	100	40.55	3.48	28.50

Table 8. Pebble #2 First Stage Log Washing.

(1) Pilot plant feed.

Eight formal pilot plant runs were performed with this sample. Parameters examined during these runs included reagent dosage, mesh of grind, and flotation with tap water and plant water. In addition to PA-31, dolomite collector FA-#4, prepared from chemical feedstock available in the USA, was tested on Pebble #2. The target parameters for each run were specified by CLDRI.

A summary of results from the formal runs is presented in Table 9. A flow diagram and material balance for each of the 8 formal runs is given in Figures 15 through 22, respectively. The solids rate and chemical analyses on these diagrams were independently developed by IMC's Matbal Program for processing after the first pass through the log washer.

Test No	9A	9B	10	11	12	13	14	15
PA-31 Source	China	USA						
Water	City	City	City	City	City	City	Plant	City
Dosage, lb/t Feed								
P ₂ O ₅	9.15	7.67	0.00	0.00	5.33	6.67	7.55	6.19
H_2SO_4	4.58	3.89	15.57	19.32	3.55	4.86	4.63	3.87
PA-31	4.35	2.77	2.24	2.16	1.78	2.54	2.42	2.20
Soda Ash	0.99	1.49	1.40	1.45	2.04	0.82	1.42	0.71
Amine	1.18	1.37	1.36	1.93	2.02	1.57	1.98	1.73
No. 2	0.13	0.18	0.47	0.58	0.53	0.38	0.36	0.36
Flotation Feed Particle Size								
> 100 mesh	6%	8%	8%	4%	38%	16%	17%	21%
< 200 mesh	67%	63%	69%	72%	44%	60%	59%	56%
Pebble Feed								
% BPL	49.36	50.54	49.49	49.47	48.11	49.45	49.82	49.42
% MgO	1.82	1.94	2.13	2.00	1.96	1.87	2.27	2.06
% A.I.	22.10	20.43	20.70	21.63	23.51	21.76	20.39	21.18
Composite Waste								
Yield, % weight	47.26	30.59	43.71	44.96	38.85	35.64	34.57	41.58
% BPL	35.51	21.42	33.38	30.39	24.09	25.49	22.09	26.63
% MgO	3.29	4.50	3.71	3.36	3.21	3.51	4.65	3.57
% A.I.	31.56	45.60	33.54	39.14	48.67	46.53	45.73	42.39
BPL % Distribution	34.00	12.97	29.48	27.62	19.45	18.37	15.33	22.40
Composite Concentrate								
Yield, % weight	52.74	69.41	56.29	55.04	61.15	64.36	65.43	58.42
% BPL	61.77	63.37	62.00	65.07	63.37	62.73	64.48	65.66
% MgO	0.51	0.81	0.90	0.89	1.16	0.97	1.01	0.99
% A.I.	13.63	9.34	10.72	7.33	7.52	8.05	6.99	6.09
BPL % Distribution	66.01	87.03	70.52	72.39	80.55	81.65	84.68	77.61

 Table 9. Summary of Pebble #2 Pilot Test Results.



Figure 14. Run 9A Mass Balance.



Figure 15. Run 9B Mass Balance.



Figure 16. Run 10 Mass Balance.



Figure 17. Run 11 Mass Balance.



Figure 18. Run 12 Mass Balance.



Figure 19. Run 13 Mass Balance.



Figure 20. Run 14 Mass Balance.



Figure 21. Run 15 Mass Balance.

DISCUSSION

Data Reliability

For purposes of this report, data reliability is determined by comparing the data before and after Matbal for each formal test run. The inputs to Matbal comprise measured and analyzed data from composite samples. The outputs from Matbal are statistically adjusted data that reduce closure errors to 0%. The magnitude of adjustments or differences between measured and adjusted data are indicative of the reliability of the measured data.

Table 10 shows the average adjustments for runs 1 through 4. The rate adjustments for the first four runs demonstrated the measured feed lb./hr was too high and/or the measured lb./hr of exit streams were too low. A review of sampling procedures identified that exit stream rate measurements were unintentionally biased low. The exit stream sampling procedures were corrected to eliminate the bias. The rate adjustments of the remaining seven runs on Pebble #1, as shown on Table 11 confirm that the rate bias was minimized or eliminated by the corrected procedures.

Adjustments to chemical analyses for the 11 runs on Pebble #1 were small except for the carbonate tails in Runs 1 and 2. The average adjustments for the eight runs on Pebble #2 are shown in Table 12. Adjustments to balance the rates for these runs were also small, adding further confirmation that the pilot plant results are reliable.

Average rate adjustments are compared to average adjusted rates on Table 13, for the pilot plant output streams. The largest adjustment, 4.0% of the corresponding average rate, occurred during the first four runs when the rate measurements were slightly biased. The average adjustments to the BPL, A.I., and MgO analyses, as shown on Tables 10, 11, and 12, are well within the allowable limits of sampling and analytical error. Consequently, the chemical analyses of the pilot plant streams are also considered to be reliable. Because of the close agreement between measured and independently adjusted data, it is evident that the pilot plant operation was in equilibrium during sampling and that the samples taken were representative and accurately analyzed.

Stream	BPL	MgO	A.I.	lbs/hour	% +100m	% 100/200 m	%200/270 m
Log Washer Feed	0.1	0.2	0.1	24.9	na	na	na
Log Washer Overflow	0.0	0.5	0.0	0.8	na	na	na
Rod Mill Discharge	-0.2	0.2	0.1	24.1	na	na	na
Mill Classifier Oversize	-0.2	0.0	0.0	1.0	na	na	na
Mill Classifier Feed	0.5	0.1	0.1	-11.4	na	na	na
Carbonate Flotation Feed	0.4	0.4	-0.2	-12.3	0.8	-0.7	-2.6
Carbonate Flotation Tails	-0.1	0.9	0.0	2.7	0.0	-0.1	0.2
Carbonate Flotation Concentrate	0.2	0.0	0.9	-15.1	-0.9	0.2	-2.0
Fine Concentrate	-0.3	0.0	0.1	-5.1	0.0	0.0	0.0
Silica Flotation Feed	0.2	0.0	-0.1	-10.0	0.0	-1.6	3.2
Silica Flotation Tails	0.0	0.0	0.1	-1.4	0.0	0.2	0.3
Silica Flotation Concentrate	0.1	0.0	-0.1	-8.6	-0.6	1.1	1.2

 Table 10. Data Reliability for Runs 1 through 4 (Pebble #1).

Values shown = (4 Measured values - 4 Adjusted value)/4 = average deviation

Stream	BPL	MgO	A.I.	lbs/hour	% +100m	% 100/200 m	%200/270 m
Log Washer Feed	0.1	-0.2	-0.3	6.9	na	na	na
Log Washer Overflow	0.0	0.1	0.0	0.1	na	na	na
Rod Mill Discharge	-0.3	0.0	0.1	6.9	na	na	na
Mill Classifier Oversize	0.0	0.0	0.1	0.9	na	na	na
Mill Classifier Feed	0.0	0.1	-0.2	-7.7	na	na	na
Carbonate Flotation Feed	0.2	0.2	0.2	-8.6	1.9	1.0	1.7
Carbonate Flotation Tails	0.0	0.2	0.0	-1.9	0.0	0.5	0.1
Carbonate Flotation Concentrate	0.3	0.0	0.0	-6.7	-1.5	-1.3	0.6
Fine Concentrate	0.0	0.0	0.1	-2.2	0.0	0.0	0.0
Silica Flotation Feed	-0.2	0.0	0.5	-4.5	1.2	2.3	1.0
Silica Flotation Tails	0.0	0.0	0.1	0.1	0.0	0.0	0.7
Silica Flotation Concentrate	0.2	0.0	0.1	-4.7	-0.5	-0.7	0.6

 Table 11. Data Reliability for Runs 5 through 8 and Runs 16, 17, & 18 (Pebble #1).

Values shown = (7 Measured values - 7 Adjusted value)/7 = average deviation

Stream	BPL	MgO	A.I.	lbs/hour	% +100m	% 100/200 m	%200/270 m
Log Washer Feed	0.8	0.1	0.9	15.2	na	na	na
Log Washer Overflow	0.0	0.3	0.0	0.5	na	na	na
Rod Mill Discharge	-0.7	0.1	0.4	14.7	na	na	na
Mill Classifier Oversize	0.0	0.0	0.2	2.0	na	na	na
Mill Classifier Feed	0.1	0.1	-0.4	-7.1	na	na	na
Carbonate Flotation Feed	-0.2	0.1	0.2	-9.1	0.6	0.3	-0.6
Carbonate Flotation Tails	-0.2	0.3	0.0	0.3	0.0	0.0	0.1
Carbonate Flotation Concentrate	-0.2	0.0	0.3	-9.4	-1.4	-0.9	1.3
Fine Concentrate	-0.2	0.0	0.1	-1.8	0.0	0.0	0.0
Silica Flotation Feed	-0.6	0.0	0.5	-7.6	2.5	1.7	2.3
Silica Flotation Tails	0.0	0.0	0.1	-1.2	0.2	0.2	0.2
Silica Flotation Concentrate	0.1	0.0	0.0	-6.4	1.5	1.3	0.4

 Table 12. Data Reliability for Runs 9a through 15 (Pebble #2).

Values shown = (8 Measured values - 8 Adjusted value)/8 = average deviation

Table 13.	Average	Rate Ad	justments.
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	Avera	ged Rate Da	ta – First 4	Runs	Averaged Rate Data – Last 15 Runs				
	Rate – lbs/hr		Adjustment		Rate –	Rate – lbs/hr		Adjustment	
Stream	Measured	Adjusted	lbs/hr	%	Measured	Adjusted	lbs/hr	%	
Log Washer Overflow	38.9	38.0	(0.9)	(2.3)	23.3	23.1	(0.2)	(1.0)	
Carbonate Tailing	131.4	128.8	(2.6)	(2.0)	106.2	107.0	0.8	0.8	
Fine Concentrate	122.4	127.5	5.1	4.1	89.9	91.9	2.0	2.2	
Silica Tailing	42.6	43.9	1.3	3.1	48.3	48.9	0.6	1.2	
Coarse Concentrate	216.5	225.1	8.6	4.0	167.6	173.2	5.6	3.3	
Total Outputs	551.9	563.4	11.5	2.1	435.3	444.0	8.7	2.0	
Log Washer Feed	588.5	563.4	(25.1)	(4.3)	455.4	444.1	(11.3)	(2.5)	
Input – Outputs	36.6	(0.0)			20.0	0.0			
% Closure Error	6.6	(0.0)			4.6	0.0			

Notes : Adjustment = lbs/hr Adjusted - lbs/hr Measured % Closure Error = 100 x (Input - Output) / Output

Clay Removal

Clay removal is an optional step in the CLDRI dolomite flotation process. The need for desliming prior to grinding should be determined by testing or the level of clay in the low-grade pebble feed. The clay minerals in Florida pebble have negative effects on flotation performance and reagent consumption. Wavellite reported in phosphate concentrate will be harmful to phosphoric acid processing. If the clay (-150 mesh) content is more than 10%, for example, the CF sample, clay removal prior to grinding is necessary. The IMC sample, with less than 5% clay, can be ground directly without clay removal. The CLDRI fine flotation technology can recover phosphate with a high BPL recovery from a fine feed produced from grinding without desliming.

Average distributions of weight, BPL, MgO, and A.I. in the log washer overflow for Pebble #1 and Pebble #2 are presented on Table 14. For Pebble #1, the average rejection of BPL with the log washer overflow was 4.3%, while 13.4% of the MgO was rejected. For Pebble #2, the combined log washer overflows removed 8.1% of the BPL and 51.4% of the MgO.

	% Distributions								
	Weight	BPL	MgO	A.I.					
Pebble #1 (one pass)	6.06	4.31	13.40	5.01					
Pebble #2 (1 st pass)	22.00	6.09	44.04	40.41					
Pebble #2 (2 nd pass)	2.66	1.97	7.36	1.40					
Pebble #2 (combined)	24.66	8.06	51.40	41.81					

Table 14. Distribution of Pebble Materials in Log Washer Overflow.

For Pebble #2 the 1^{st} pass losses were high because of the clay content and the fact that the scalping screen was not used to reclaim feed material from the overflow. According to CLDRI's criteria, the log washer could have been bypassed for Pebble #1 and for the 2^{nd} pass with Pebble #2. Had this been done, reagent consumption would probably have increased.

Grinding

Grinding is a necessary step of the CLDRI fine flotation technology. The determination of reasonable grinding fineness depends on the following factors: (1) the occurrences and particle sizes of phosphate and carbonate minerals, (2) the amounts of MgO, and (3) the target performance (concentrate quality and recovery). Table 15 shows the effect of grinding on flotation results. Some degree of preferential grinding was

observed from both bench scale and pilot plant data, with dolomite being the easiest to grind followed by francolite. Quartz (silica) was the hardest mineral.

Flotation performance may be adversely influenced by the presence of excess coarse (+100 mesh) and/or fine (-400 mesh) particles. Since the dolomite minerals are not liberated from phosphate in the coarser particles, the removal of MgO is not sufficient. However, too finely ground feed may lead to losses of fine phosphate as carbonate tailing and the increase of fine silica in -400 mesh phosphate fine concentrate.

	Test	Fineness		Cor	Soroon			
Sample	No.	-200	Passing	BPL	MgO	A.I.	Flotation	Cloth
	Mesh (%)	(Mesh)		8 -		Recovery		
Pebble	Run 5	61.3	80	64.5	1.17	5.43	80.8	DF 66
#1	Run 16	57.5	50	62.3	1.56	6.31	83.5	DF 43
Pebble	Run 14	58.6	60	64.9	0.99	7.06	88.0	DF 36
#2	Run 12	43.5	45	62.9	1.22	7.53	82.6	DF 48

Table 15.	Grinding	Effects	on Flotation	Results.
Table 13.	Ormung	Lincus	on riotation	ncourto.

Dolomite Flotation Stage

Dolomite flotation is the key of the CLDRI fine particle flotation process. A mixture of phosphoric and sulfuric acids was used as a phosphate depressant as well as a pH modifier in the dolomite step. A proprietary reagent invented by CLDRI, PA-31, is a selective dolomite collector. The following conditions have significant effects on dolomite flotation:

- 1. Flotation machine operating parameters
- 2. Grinding fineness and particle size distribution
- 3. Dolomite collector selectivity
- 4. Suitable dosages of reagents, especially phosphoric acid.

Results for dolomite flotation from six test runs are presented on Table 16. The tabulated data are specific to the dolomite flotation circuit and exclude pretreatment and silica flotation. For Pebble #1 the best dolomite flotation performance was probably obtained with Run 18. This flotation run, utilizing phosphoric acid only for pH adjustment and phosphate depression, recovered 81.9% of the phosphate and rejected 77.7% of the MgO and 11.2% of the A.I.. Even using plant water in Run 6 the phosphate recovery was 78.2%. For Pebble #2 the best dolomite flotation performance was probably obtained with Run 9B. The use of plant water and a slightly coarser grind in Run 14 impaired phosphate recovery and grade. Run 15 shows results with the dolomite collector made from feed stocks sourced in the USA.

		Pebble #1		Pebble #2		
Test Run No.	5	6	18	9B	14	15
PA-31 Source	China	China	China	China	China	USA
Water	City	Plant	City	City	Plant	City
Reagent Dose, lbs/ton Feed						
P2O5	9.08	8.35	12.09	7.67	7.55	6.19
H2SO4	4.58	4.18	0.69	3.89	4.63	3.87
PA-31	4.04	4.02	4.22	2.77	2.42	2.20
Flotation Feed Particle Size						
% > 100 mesh	13.00	16.40	7.60	8.20	16.80	21.40
% < 200 mesh	61.30	57.90	69.40	62.60	58.60	55.50
Flotation Cells ⁽¹⁾						
Impeller Tip Speed (fpm)	942	942	1,099	N.R.	N.R.	N.R.
Froth Paddles (rpm)	14.5	14.5	17.0	N.R.	N.R.	N.R.
Exit slurry pH	4.9	4.8	5.0	4.7	4.8	4.7
Feed slurry % solids	34.2	34.2	27.1	29.6	32.3	28.3
Dolomite Flotation Feed						
% BPL	52.57	52.40	51.87	51.26	50.91	50.50
% MgO	3.30	3.40	3.33	1.73	1.94	1.81
% Insol	10.92	10.78	10.11	20.69	20.68	21.25
Phosphate Concentrate						
Yield, %	73.64	70.35	71.46	87.03	84.72	79.71
% BPL	58.86	58.25	59.45	54.14	53.45	53.01
% MgO	1.07	1.13	1.04	0.70	0.84	0.80
% Insol	13.42	13.54	12.56	22.31	22.39	23.66
% BPL Recovery	82.45	78.22	81.91	91.93	88.94	83.68
% MgO Rejection	76.12	76.62	77.68	64.78	63.32	64.77
% Insol Rejection	9.50	11.63	11.22	6.15	8.27	11.25
Dolomite Tails						
% BPL	34.96	38.50	32.95	31.97	36.77	40.68
% MgO	9.51	8.80	9.07	8.66	8.06	5.77
% Insol	3.95	4.21	3.98	9.83	11.15	11.77

Table 16. Summary of Pilot Test Dolomite Flotation Results.

(1) Carbonate flotation consisted of two machines in series. Cells 1, 2, 3, & 4, with No. 7 mechanisms in the first machine, were maintained at constant conditions for all tests. Cells 5 & 6 with No. 8 mechanisms in the second machine were operated with the conditions shown. For Pebble #2 the second machine (cells 5 & 6) was not required, and the exit slurry pH was measured at cell 4.

Silica Flotation Stage

The need for silica flotation depends on the acid insoluble content of the lowgrade pebble and the final phosphate concentrate grade specifications. A feed with more than 52% BPL, over 3% MgO and less than 10% A.I. could produce a concentrate from dolomite flotation of more than 59% BPL without a silica flotation step. But, for a feed with about 44% BPL and <2% MgO, CLDRI believes that silica flotation is needed to reject part of the silica to improve final concentrate quality.

The phosphate concentrate from dolomite flotation should be sized to remove the -400 mesh fraction as a fine concentrate. The +400 mesh fraction is subjected to silica flotation. Prior to silica flotation, a pH modifier (soda ash) is added to adjust to about pH 7. An amine is then added as the silica collector and No. 2 D.O. as an extender, with an amine to oil ratio ranging from 4:1 to 5:1.

There are two differences in silica flotation between the pilot test and the current commercial operations in Florida: (1) in the pilot plant the silica flotation feed is +400 mesh versus +150 mesh in existing plants; and (2) the amine flotation feed is not de-oiled prior to flotation in the pilot plant. Because of those differences, amine consumption in the pilot plant was a little bit higher than that currently used in Florida. Amine dosage is also affected by dolomite flotation: as the amount of dolomite removal increases in dolomite flotation, amine usage decreases in the second step.

Averaged results for amine flotation with tap water and plant water are presented on Table 17 for Pebble #1 and Pebble #2. Removal of A.I. with plant water appeared to be less of a problem with Pebble #2, although more amine was required.

The phosphate recovery for amine flotation averaged over 96% for the 11 runs with Pebble #1 and over 94% for the 8 runs with Pebble #2. Similarly, the rejection of A.I. by amine flotation averaged over 76% for Pebble #1 and over 85% for Pebble #2.

Pebble	Pebble	e #1	Pebble #2		
	1, 2, 3, 4,		9A, 9B, 10,		
Test No.	5, 7, 16, 17,	6 & 8	11, 12, 13,	14	
	& 18		& 15		
Water	City	Plant	City	Plant	
Reagent Dose, lbs/ton Feed					
Soda Ash	0.62	1.06	1.27	1.42	
Amine	0.95	0.96	1.59	1.98	
No. 2	0.24	0.35	0.38	0.36	
Silica Flotation Feed					
% BPL	56.40	56.43	50.43	51.68	
% MgO	1.30	1.06	0.63	0.70	
% A.I.	15.56	16.16	27.85	25.75	
Silica Tails					
% BPL	13.21	11.66	10.69	6.45	
% MgO	0.34	0.24	0.15	0.14	
% A.I.	78.89	81.34	83.44	88.00	
Coarse Concentrate					
Yield, %	84.39	86.61	71.69	73.52	
% BPL	64.37	63.42	66.18	67.95	
% MgO	1.48	1.19	0.83	0.90	
% A.I.	3.86	5.96	5.80	3.32	
% BPL Recovery	96.27	97.15	93.90	96.67	
% MgO Rejection	4.03	2.63	7.02	5.48	
% A.I. Rejection	78.69	66.52	84.57	90.52	

 Table 17. Summary of Pilot Test Silica Flotation Results.

Plant Process Water

Pilot tests demonstrated that Florida local plant process water had minimal effects on the performance of the CLDRI fine particle flotation process on both of the test feeds. In contrast, most of the other dolomite flotation processes either suffered significantly of failed completely when plant water was used in place of tap water.

Three runs were performed with plant water: two on Pebble #1 and one on Pebble #2. The results of the plant water runs and comparable tap water runs are compared on Table 18. Considering mesh of grind and reagent consumption as well as water source, the effect of plant water is more pronounced in the silica float than in the dolomite float. Plant process water tends to make the amine reagent less effective.

Sample	Test No.	Water	Composite Concentrate (%)			
			BPL	MgO	A.I.	Flotation Recovery
Pebble #1	Run 5	City	64.4	1.17	5.43	80.83
	Run 6	Plant	61.8	1.19	8.44	77.24
Pebble #1	Run 7	City	63.6	1.21	5.83	73.97
	Run 8	Plant	64.4	1.06	5.50	61.74
Pebble #2	Run 13	City	62.7	0.97	8.05	84.69
	Run 14	Plant	64.5	1.01	6.99	86.91

 Table 18. Plant Process Water Effects on Flotation Results.

Handling of the Fine Concentrate (Dewatering and Transportation)

Concentrate obtained by the CLDRI process has a much finer particle size than flotation concentrates from the existing beneficiation plants. Therefore, the conventional method of dewatering in bins is no longer applicable. CLDRI proposes that the composite concentrate can be thickened to 60% solids, then transported to the chemical plant using pumps. An alternative is to filter and mix the fine concentrate from the dolomite separation process with the concentrate produced from the regular circuits. It may also be feasible, and perhaps more economical and practical, to locate the dolomite separation plant at the chemical processing complex, thus allowing direct pumping of the thickened fine concentrate to the reactor.

Laboratory scale settling tests were performed to investigate the dewatering characteristics of the composite concentrate and the flotation tailings from Pebble #1. Concentrate sedimentation curves for three different pulp densities are shown on Figure 22. In each case, the maximum thickened solids content achieved was about 60% by weight. Sedimentation curves for dolomite tailings and for a mix of dolomite and silica tailings are shown on Figure 23. The dolomite tailings settled to about 40% solids and

the mixture of tailings settled to about 45% solids. No flocculant was used in the concentrate and tailings settling tests.

Laboratory scale vacuum filtration tests were performed to further investigate dewatering of the concentrates from Runs 17 and 18. For Run 17, the concentrate slurry was thickened to 50% solids prior to filtration, and the resulting filter cakes contained 76 to 80% solids with filtration rates of 0.04 to 0.05 tons of solids per square foot per hour. For Run 18, six filtration tests were performed, three with 30% solids pulp and three with 60% solids pulp. Filter cakes obtained from the dilute pulps contained 72 to 73% solids, but filtration rates were only 0.02 to 0.04 tons of solids per square foot per hour. Filter cakes obtained from the thickened pulps contained 75% solids and filtration rates were 0.08 to 0.12 tons of solids per square foot per hour. The material did not filter well.

Operating Costs (Reagents and Grinding Power)

Reagent consumption and grinding energy are major components of the total operating cost for the CLDRI dolomite flotation process. The following points address concerns for these costs:

- The higher overall BPL recovery with CLDRI's fine particle process can justify higher reagent costs to some extent.
- The beneficiation grinding costs reduce grinding costs in the chemical plant.
- Using phosphogypsum pond water as a substitute for sulfuric acid (Gruber 1995 & 1999), and reusing acidic process water in dolomite flotation will reduce the operating costs.
- Eliminating the silica flotation step gives a 30% reduction in reagent costs for Pebble #2.
- The low-grade pebble is currently considered as a waste, for which mining and waste disposal costs are already accounted for in the normal operation. The savings in waste disposal costs may be credited to the dolomite separation plant.

Environmental Viability (Reagent Quantities and Qualities, Water Systems, Etc.)

Processing of PA-31 does not generate any waste, a significant environmental plus. The major amounts of flotation processing water yielded from the CLDRI's fine particle process can be recycled and re-used. The process water recovered from tailings has a pH of 6.0-6.5 and higher chemical oxygen demand (COD), which can easily be treated, if the need for water discharge should arise. There are several successful treatment plants to process such waste acid water operating currently in China. Treatment consists of adding lime to adjust the pH, and aeration to satisfy the COD.

The environmental problems associated with silica flotation are no different from the current practice in Florida.



Figure 22. Bench Scale Sedimentation Curves for Pebble #1 Composite Concentrate.

85



Figure 23. Bench Scale Sedimentation Curves for Pebble #1 Tailings.

59

PA-31 Availability

Cursory lab tests and a pilot test using FA-#4 dolomite collector have demonstrated that some local materials can be used to manufacture collectors to separate dolomite from Florida pebbles instead of PA-31 processed using the original Chinese recipe. However, further studies such as manufacturing conditions and evaluation of mineral processing conditions are necessary.

FA-#4 dolomite collector was manufactured as a substitute for PA-31 by CLDRI engineers in FIPR's lab. One pilot test run was conducted on Pebble #2 using FA-#4, achieving similar phosphate concentrate quality but with relatively lower recovery compared with using PA-31.

The pilot tests demonstrated that CLDRI fine particle flotation technology can be successfully and effectively applied to recover phosphate concentrate from Florida dolomitic pebble. Many of the pilot test runs either duplicated or exceeded bench scale performance. It is therefore gratifying to say that the primary objective of the pilot testing program has been met.

A pilot test concentrate analyzing less than 1% MgO with overall BPL recovery of more than 85% (with flotation BPL recovery of approximately 90%) was achieved from a pebble feed containing MgO less than 2%. For a feed containing more than 3.5% MgO, the concentrate analyzed less than 1.2% MgO with an overall BPL recovery of more than 76% (flotation BPL recovery of about 80%). The BPL grades of phosphate concentrate ranged from 63.4 to 64.9%.
PRELIMINARY STUDY

CONCEPT

CLDRI prepared a block flow diagram, as shown on Figure 24, to illustrate their concept of how the CLDRI dolomite flotation process would be applied to low-grade phosphate pebble from Florida. A corresponding material balance for the assumed quality of low-grade pebble is presented on Figure 25.

FLOWSHEET

Using CLDRI's process concept and material balance, Jacobs developed the battery limits flowsheet and solids: liquid balance for a design feed rate of 300 tph, as shown on Figure 26. The battery limits of the dolomite flotation plant are defined as follows:

- Ore Supply: A 600-ton pebble surge bin receives low-grade pebble from a conveyor system provided by others.
- Water Supply: 900-gpm low-pressure water provided by others.
- Concentrate Dispatch: Two agitated tanks having about 8 hours capacity each are provided for thickened concentrate slurry. The concentrate pumping system and slurry pipeline is by others.
- Tailings disposal: A general mill tailing pump, a return water pump, and associated pipelines are included. The tailing pond construction is excluded from the capital cost, but is included as an annual accrual of \$850,000 to the operating cost estimate.

An equipment list for the battery limits of the dolomite flotation plant is given in Appendix D. The process elements within the battery limits are listed below:

- Pebble pretreatment (desliming)
- Closed circuit grinding
- Dolomite flotation and reagent supply
- Silica flotation and reagent supply, preceded by two stage classification
- Thickening of coarse and fine concentrates with water recycle
- Disposal of clay, dolomite, and silica wastes with water recycle

Low-grade pebble is received in the 600-ton capacity pebble surge bin. Pebble is recovered at a controlled rate and distributed to parallel log washers for attrition and washing. The clean pebble is discharged to the ball mill feed chute. Wash water overflowing the logs is collected in the overflow pump box and pumped to the feed scavenger cyclones. Material coarser than 150 mesh is discharged from the cyclone apex to the ball mill feed chute, and the cyclone overflow collected in the general mill tailing pump box. In the event of low clay content in the pebble, it is envisioned that the screw

jacks would be lowered and the wash water would be shut off so that the log washers would act as conveyors only.

Clean pebble and recovered feed are ground in a 4,500 Hp ball mill. Slurry discharging the mill is collected in the mill pump box and pumped to the mill cyclones for classification at 80 mesh. The cyclone underflows are recycled to the ball mill and the overflows at about 30% solids, report to the first dolomite-conditioning tank.

Sulfuric acid and phosphoric acid are added to the first dolomite-conditioning tank to adjust slurry pH and depress phosphate. Dolomite collector is added to the second dolomite-conditioning tank and also to the dolomite flotation cells. The conditioned pulp flows to a single circuit of five dolomite flotation cells. Tailings overflowing the cells are transferred to the general mill tailing pump box by the dolomite froth pump. The cell product (concentrate) flows into the first rougher concentrate pump box and is pumped to the first rougher concentrate cyclones for classification at 270 The cyclone overflows report to the acid slurry thickener where they are mesh. dewatered to about 60% solids. Acidic water recovered from the thickener is recycled to The cyclone underflows are pumped to the second rougher the mill pump box. concentrate cyclones for reclassification at 270 mesh. The cyclone overflows are recycled to the first rougher concentrate pump box, and the cyclone underflows report to the first silica conditioning tank. Without the silica flotation circuit, the first rougher concentrate pump would discharge directly to the acid slurry thickener.

Soda ash is added to the first silica-conditioning tank to adjust the slurry pH to neutral. Amine and an extender are added to the second silica-conditioning tank. The conditioned pulp flows to a single circuit of two silica flotation cells. Tailings overflow the cells to the general mill tailing pump box, and the cell product is pumped to the coarse concentrate thickener. Water recovered from the thickener is recycled to the second rougher concentrate pump box. The coarse concentrate thickener and the acid slurry thickener underflows, at about 60% solids, are pumped to the agitated slurry storage tanks.

The general mill tailing, which contains clay, dolomite, and silica, is pumped to the tailings pond. Solids are separated from water by natural sedimentation in the pond, and recovered water is recycled to various points in the flowsheet by the tailings return water pump.



Figure 24. CLDRI Block Flow Diagram.



Figure 25. Material Balance for the Assumed Quality of Low-Grade Pebble.



Figure 26. The 300 tph Battery Limits Beneficiation Plant PFD and Solids/Liquid Balance.

COST ESTIMATES

Operating Costs

The components of Jacobs operating cost estimate for the battery limits plant are presented on Table 19. The estimated variable (production rate dependent) costs sum to \$12.52/ton of concentrate. Fixed costs total an estimated \$3.10/ton of concentrate. Total operating costs, which are the sum of variable and fixed costs, are estimated at \$15.62/ton of concentrate.

Variable Costs	\$/Year	\$/Ton Feed	\$/Ton Concentrate
Raw Materials	5,925,000	2.50	3.75
Electric Power	1,699,185	0.72	1.08
Reagents	5,040,985	2.13	3.19
Consumables	2,261,481	0.95	1.43
Severance Tax	2,564,719	1.08	1.62
Dam Building	850,000	0.36	0.54
Contract Maintenance	791,580	0.33	0.50
Services	649,096	0.27	0.41
Subtotal	19,782,046	8.35	12.52
Fixed Costs			
Labor	1,379,040	0.58	0.86
Overhead	1,200,000	0.51	0.76
Depreciation	1,220,000	0.51	0.77
Supplies	68,952	0.03	0.04
Taxes	900,000	0.38	0.56
Insurance	150,000	0.06	0.09
Subtotal	4,917,992	2.08	3.10
Total Operating Cost	24,700,038	10.42	15.62

Table 19. Estimated Operating Costs for 300 tph Battery Limit Plant.

The raw materials cost item on Table 19 is an allowance by Jacobs to cover the cost of retrieving and transporting waste pebble to the battery limits plant surge bin. Electric power is based on the connected motor Hp and a unit price of \$0.04 per kWh. The grinding work index (10 kWh per ton) that was used in the year 1 report was also assumed for this report. Jacobs recommends that this critical parameter be specifically confirmed for detailed studies.

Reagents, which are a significant component of the variable costs, are estimated in the following tabulation. The consumption of sulfuric acid, phosphoric acid, dolomite collector, soda ash, amine and diesel oil were determined by CLDRI based on test data and the characteristics of the low-grade pebble. CLDRI also derived the unit cost of the dolomite collector, considering raw material cost, manufacturer's cost and profit, license fee, and freight. Jacobs assigned unit costs for all other reagents and determined the consumption of flocculent for thickening and water clarification.

Reagent	Pound per Year	\$ per Pound	\$ per Year
Sulfuric acid	11,494,784	0.015	172,422
Phosphoric acid	17,242,177	0.09	1,551,796
Dolomite collector	6,269,882	0.30	1,880,965
Soda ash	3,657,431	0.07	256,020
Amine	4,179,922	0.25	1,044,980
Diesel oil	940,482	0.06	56,429
Flocculent	104,498	0.75	78,374
Totals	43,889,177		5,040,985

Consumables are wear parts and maintenance parts required for sustaining the operation. Severance tax is levied by the State for each ton of phosphate rock product. Dam building provides for tailing dam expansion and maintenance. Contract maintenance provides for outside maintenance services. Services include chemical analyses for process and quality control, assistance with environmental, regulatory, and technical issues, as well as clerical functions for personnel and accounting.

Labor, which is the most significant component of fixed costs, is based on the following manning table.

Description	Personnel per Shift	Shifts per Day ⁽¹⁾	Total Personnel ⁽¹⁾
Laborers	1	3	4
Dam tender	1	3	4
Reagent man	1	1	1
Operators	3	3	12
Maintenance	3	1	3
Engineer	1	1	1
Supervisors	1	3	4
Superintendent	1	1	1
			30

(1) Four shifts are required to maintain three shifts per day, seven days per week.

Other elements of the estimated fixed costs are overhead, depreciation, supplies, taxes, and insurance. Overhead provides for allocated costs for purchasing, accounting, personnel, and safety, as well as vehicle operating costs.

Constructed Cost Estimate

Jacobs' estimate of the battery limits plant constructed cost is 31.6 million dollars. This estimate includes all the equipment listed in Appendix D, plus construction materials, construction labor, subcontracts, field indirect costs, engineering and procurement services, and an allowance of 10% for unforeseen costs. Excluded from the estimate are land costs, soil testing and major site preparation, working capital, start up expenses, and escalation.

Potential Savings

The battery limits plant includes silica flotation, which reduces the concentrate A.I. from 19 to 6%. Capital and operating cost savings would be obtained if the phosphoric acid plant could tolerate the higher level of inert material. Excluding the silica flotation circuit would save about 14% of the constructed cost (4.5 million dollars) and 10% of the operating cost (\$1.50 per ton of concentrate).

CONCLUSIONS

DATA RELIABILITY

The validity of conclusions based on test data is dependent on the reliability of the data obtained. For this project the measured data were analyzed by IMC's Matbal program to obtain material balances with 0% closure errors. Comparisons of measured and independently adjusted data showed that the required adjustments were typically small and within allowable error for sampling and analysis. The test data obtained by this project are therefore considered to be reliable and provide a sound basis for evaluation.

PROCESS PERFORMANCE – PILOT SCALE

BPL Recovery

Phosphate losses were incurred at three points, namely desliming, dolomite flotation, and silica flotation. If the log washer overflows are scavenged, desliming losses of BPL will be controlled to less than 5% on average. Phosphate losses to dolomite tailings were about 7% for Pebble #2 and 18% for Pebble #1. Silica flotation did not reject much phosphate, with losses of BPL at 2 to 3%.

The possible overall phosphate recovery, taking into account desliming, dolomite flotation, and silica flotation is about 85% for Pebble #2 and 75% for Pebble #1.

MgO Rejection

Dolomite is rejected from the pebble before grinding by the log washer, and after grinding by dolomite flotation. Desliming removed about 13% of the MgO from Pebble #1 and 53% of the MgO from Pebble #2. Dolomite flotation rejected about 68% of the MgO from Pebble #1 and 32% of the MgO from Pebble #2. The MgO rejection values are based on original pebble. For Pebble #2, both stages of log washing are considered. Silica flotation rejected less than 1% of the MgO from either Pebble #1 or #2.

The possible overall dolomite rejection is about 82% for Pebble #1 and 86% for Pebble #2.

Rejection of Acid Insolubles

The acid insoluble material removed from Pebble #1 by desliming, dolomite flotation, and silica flotation ranged respectively from 3 to 6%, 7 to 13%, and 35 to 69%. Similarly for Pebble #2, the removal was about 42%, 3 to 12%, and 27 to 44%, respectively, for desliming, dolomite flotation, and silica flotation. The rather large range

of acid insoluble rejection for silica flotation was influenced both by flotation performance and the fineness of grind.

Concentrate Grade

The majority of Florida phosphate rock is acidulated to produce phosphoric acid, which in turn is converted to granular fertilizers. The minor element ratio (MER) of the rock is one parameter used to gauge the rock's utility in the production of phosphoric acid and DAP. The MER of rock is determined from the sum of iron, aluminum, and magnesium oxides divided by the P_2O_5 . Similarly, the CaO: P_2O_5 ratio is a useful parameter to estimate sulfuric acid consumption during acidulation. Analytical data of a typical phosphate rock are compared below to Pebble #1 concentrate and Pebble #2 concentrate.

		Pebble #1	Pebble #2
	Typical Rock	Concentrate	Concentrate
% BPL	65.90	64.94	63.37
% CaO	44.00	46.31	44.94
% MgO	0.46	1.14	0.81
% I & A	2.45	2.31	2.53
CaO:P ₂ O ₅	0.668	0.713	0.709
MER	0.044	0.053	0.053

The concentrates produced are at the high limit of $CaO:P_2O_5$ for rock accepted by phosphoric acid plants, and exceed the MER limit for DAP production. For both concentrates the high MER is caused by elevated levels of MgO and I&A. Elevated I&A is particularly a problem for Pebble #2. The CLDRI Process, similar to other dolomite flotation schemes, does not specifically reject iron oxides and alumina. Herein lies a problem, because low-grade pebble often contains elevated levels of I&A.

REAGENT CONSUMPTION

H₂SO₄ and P₂O₅

Sulfuric and phosphoric acids are somewhat interchangeable in the CLDRI process; however, the best dolomite flotation performances were obtained when the P_2O_5 :H₂SO₄ weight ratio was two or higher. The indicated consumption of P_2O_5 is 14 pounds or more, per ton of concentrate. Both chemicals are readily available at local phosphoric acid plants.

PA-31

This proprietary fatty acid soap is not now readily available in the USA. Similar or equal quality soaps may be produced from vegetable fatty acids available in the USA. The consumption of PA-31 is somewhat dependent on the quantity of dolomite to be removed. The indicated consumption was 7 pounds per ton of concentrate for Pebble #1 and 5 pounds per ton of concentrate for Pebble #2. Additional work may be warranted to develop a "Florida" version of PA-31, so that cost and availability can be better determined.

Amine, Soda Ash, and No. 2 Diesel Oil

These reagents are readily available in Florida. Their use and costs for the conventional Crago Process are well known. For the CLDRI Process the consumption of these reagents exceeds that of the Crago Process.

PRELIMINARY STUDY

Estimates of constructed cost and operating cost for a 300-tph plant utilizing the CLDRI Process indicate economic feasibility, relative to a grass roots mine with the same capacity. The battery limits plant would cost about 32 million dollars and produce about 1.58 million tons of concentrate annually. The estimated production cost of \$15.62 per ton of concentrate is less than the industry average operating cost of about \$20 to \$22 per ton.

ADDITIONAL WORK

Pilot test results have demonstrated that the CLDRI fine particle flotation process can be successfully applied to separating dolomite from Florida dolomitic pebble. These tests have also pointed to further improvements. The following programs are recommended by CLDRI.

Reagent

Detailed test work on local reagents and raw materials is needed to search for new dolomite and/or carbonate collectors to float both finely and coarsely liberated dolomite and/or carbonate. FA-#4 was manufactured and tested, but the pilot test run was not extremely satisfactory. Both the budget and time restraints prevented optimization of the formulation of the dolomite collector using local ingredients.

Beneficiation

A combination of the IMC cationic process with the CLDRI fine particle flotation process for processing Florida pebble may prove to be a better approach. The coarse-liberated dolomite particles are difficult to reject by the CLDRI fine particle flotation process. To reduce more dolomite to a floatable particle size, grinding finer than liberation size may be necessary, which increases grinding costs. A sized-flotation approach is therefore recommended. In this approach, the coarse flotation feed, say the +100 mesh fraction, would be processed using the IMC cationic process, while the fine fraction would be processed with the CLDRI fine particle flotation technique.

Based on pilot test, phosphoric acid consumption in some cases accounted for 40% of the total reagent costs for the CLDRI process. Therefore, reduction in phosphoric acid dosage can significantly cut down the operation costs. Low-grade phosphoric and sulfuric acids are recommended for further evaluation. Pond water is another candidate for more investigation.

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Appendix A

EQUIPMENT LIST – JACOBS PILOT PLANT FOR CLDRI PROCESS

Area	Equipment	Num	ber			
Number	Number	Operating	Spare	Equipmer	nt/Description	
1	E 1	1	0	Pebble Conveyor		
				Size:	18"w x 50'l x 14'lift	
				Type:	Belt conveyor	
				Material:	Carbon steel, rubber belt	
				Connected Motor HP:	5	
1	E2	1	0	Surge Hopper		
				Size:	60 cubic feet	
				Type:	fabricated	
				Material:	Carbon steel	
				Connected Motor HP:	na	
1	E3	1	0	Belt Feeder		
				Size:	8"w x 72"l	
				Type:	QPEC	
				Material:	By vendor	
				Connected Motor HP:	0.25	
1	E4	1	0	Roll Crusher		
				Size:	10" x 16"	
				Type:	QPEC	
				Material:	By vendor	
				Connected Motor HP:	3	
1	E5	1	0	Bucket Elevator		
				Size:	6" x 4" x 215"	
				Type:	QPEC	
				Material:	By vendor	
				Connected Motor HP:	2	
1	E6	1	0	Log Washer		
				Size:	24"w x 10"l	
				Type:	EIW 1/4 scale	
				Material:	By vendor	
				Connected Motor HP:	10	
1	E7	1	0	Rod Mill		
				Size:	16"dia x 48"l	
				Type:	QPEC o'flow discharge	
				Material:	By vendor	
				Connected Motor HP:	5	

EQUIPMENT LIST – JACOBS PILOT PLANT FOR CLDRI PROCESS

EQUIPMENT LIST – JACOBS PILOT PLANT FOR CLDRI PROCESS (CONT.)

Area	Equipment	Num	ber			
Number	Number	Operating	Spare	Equipmer	nt/Description	
1		1	-			
	E8 & E9	1	0	Mill Pump Box & Pun	np	
				Size:	1 ula Sond Dump	
				Type. Motorial:	Sand Fullip Du vender	
				Connected Motor HD		
				Connected Motor IIF.	Z	
1	E10	1	0	Ball Mill		
				Size:	16"dia x 48"l	
				Type:	DECO grate discharge	
				Material:	By vendor	
				Connected Motor HP:	5	
1	E11	1	0	Mill Screen		
				Size:	3'w x '1	
				Type:	Derrick sandwich	
				Material:	By vendor	
				Connected Motor HP:	1	
1	E12	2	0	Carbonate Conditione	er Tanks	
				Size:	2@8"dia x 6"	
				Type:	fabricated	
				Material:	Carbon steel	
				Connected Motor HP:	1	
1	E13	6	0	Carbonate Flotation (Cells	
				Size:	4@No.7 & 2@No. 8	
				Туре:	Denver Sub A	
				Material:	By vendor	
				Connected Motor HP:	4	
1	E14	1	0	Concentrate Pump		
				Size:	1"dia	
				Type:	Sand Pump	
				Material:	By vendor	
				Connected Motor HP:	1.5	
1	E15	1	0	Concentrate Screen		
				Size:	3'w x 2'1	
				Type:	Derrick sandwich	
				Material:	By vendor	
				Connected Motor HP:	1	

EQUIPMENT LIST – JACOBS PILOT PLANT FOR CLDRI PROCESS (CONT.)

Area	Equipment	Nun	ıber		
Number	Number	Operating	Spare	Equipmer	nt/Description
1	E16	2	0	Silica Conditioner Tar	ıks
				Size:	2@10"dia x 8"
				Type:	fabricated
				Material:	Carbon steel
				Connected Motor HP:	1.25
1	E17	2	0	Silica Flotation Cells	
				Size:	2@No. 18
				Type:	Wemco
				Material:	By vendor
				Connected Motor HP:	1
1	F 10	1	0	a a	
1	E18	1	0	Scavenger Screen	
				Size:	30"dia
				Type:	Sweco
				Material: Connected Motor HP:	stainless steel 0.5

Appendix B

MATERIAL SAFETY DATA SHEETS FOR PA-31

MATERIAL SAFETY DATA SHEET

Manufacturer

LIANYUNGANG DESIGN & RESEARCH INSTITUTE No. 51, West Caoyang Road, Lianyungang, Jiangsu, China Post code : 222004

> Emergency Phone & Fax Number Tel.: (86)-518-5520169 Fax: (86)-518-5410645

PRODUCT INFORMATION

Trade Name Chemical Family Composition D.O.T. Shipping PA-31 Fatty Acid Soap Mixture of C-16 to C-22 fatty acid Soap Not Regulated

PHYSICAL DATA

Acidic Value Iodine Value Boiling Point (°F) Solubility in Water Appearance Odor Specific Gravity(at 100°F) >184 mg KOH/g 55-93g/100g 230 Soluble in water Light yellowish Paste Typical Fatty acid Odor 0.93±0.05

FIRE, EXPLOSION

Extinguishing Media
Water fog, foam, dry chemical. Water is unsuitable on burning material. It may be used to cool exposed containers
Special Fire Fighting Procedures
Self contained breathing apparatus should be worn when fighting a chemical fire. Water spray may cause frothing if applied to burning material. Water spray may be used to cool exposed containers or to flush material away from flames

HEALTH HAZARD INFORMATION

EYE

Effect	Contact with material may irritate eyes						
First Aid	In case of contact flush eyes with water for minutes. Get medical attention if symptoms severe or persistent.						
Protection	Safety glasses and/or goggles.						
	SKIN						
Effect	Prolonged or repeated contact may cause skin irritation.						
First Aid	Washing with soap and water.						
Protection	Rubber gloves and clean body covering clothing. Wash clothing before reuse and do not wear contaminated boots or shoes.						
_	INHALATION						
Effect	None expected under normal conditions. Sufficient concentrations of fumes or mist may irritate mucous membranes and lungs.						
First Aid	Move to fresh air. If necessary, aid in breathing and get immediate medical attention.						
Protection	None required for normal conditions. Use an organic vapor/mist respirator in instances of heavy mist or vapors						
-	INGESTION						
Effect	If swallowed material may irritate mouth and gastrointestinal tract. Toxicity has not been determined.						

REACTIVITY DATA Stable Stability Hazardous Polymerization Will NOT occur Incompatibilities Avoid contact with oxidizers of organic material, strong mineral acids, halogenated organic compounds. Hazardous Decomposition Major amounts of oxides of carbon, as well as other unidentified organic compounds. Products SPILL, LEAK, AND DISPOSAL PROCEDURES Spills and Leaks Contain spill and remove by mechanical means. Use absorbent material or pads on remaining material or on small spills. Advise authorities if material has entered or enter waterways or sewer drains. Waste Disposal Dispose of according to local, state, and federal regulations. SPECIAL PROTECTION INFORMATION **Respiratory Protection** No respiratory protection is normally required. Use a NIOSH/MSHA approved organic vapor/mist respirator in instances of heavy mist or vapors. **Protective Gloves** Use rubber or other chemical resistant gloves. Eye Protection Use safety glasses and goggles. Other Protective Equipment Safety shower, eye bath, and as needed to prevent skin contact.

STORAGE AND SPECIAL PRECAUTIONS

Storage Precautions	Protect from freezing and high ambient temperatures.
Other Precautions	Material spilled on floors may be slippery. Wash
	contaminated clothing before wearing. Do not wear
	contaminated shoes or boots.

DISCLAIMER OF EXPRESSED OR IMPLIED WARRANTS

This material safety data sheet and the information it contains is offered to you in good faith as accurate. We have reviewed any information contained in this data sheet which we received from sources outside our company. We believe that information to be correct but can not guarantee its accuracy or completeness. Health and safety precautions in this data sheet may not be adequate for all individuals and/or situations. It is the user's obligation to evaluate and use this product safely and to comply with all applicable laws and regulations. No statement made in this data sheet shall be construed as a permission or recommendation for the use of any product in a manner that might infringe existing patents. No warranty is made, either expressed or implied.

Appendix C

MATBAL INPUT SHEETS

MATBAL INPUT SHEETS

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

	Approved	Date
CLDRI		
Jacobs		

Test Number: Real Run – 1 Date of Test: 11-Oct-00 Pebble Sample: IMC

	Stream		Analyses (% of Solids by Weight)						Rates (kg/h)		
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.48	3.74	10.11	n/a	n/a	n/a	257.74	29.43	287.17	89.75%
2	Logwasher O'Flow	16.62	10.21	7.39	n/a	n/a	n/a	17.08	694.71	711.78	2.40%
3	Rod Mill Discharge	23.25	3.92	11.51	n/a	n/a	n/a	240.67	121.73	362.40	66.41%
4	Mill Classifier O'Size	24.14	3.34	11.83	71.80%	12.68%	1.84%	86.67	44.36	131.03	66.15%
5	Mill Classifier Feed	24.21	3.64	11.60	37.73%	21.89%	3.93%	306.13	362.22	632.35	48.41%
6	Flotation Feed	23.84	3.90	10.55	12.91%	26.49%	5.05%	219.46	347.28	566.74	38.72%
7	Carbonate Tails	16.76	10.96	4.08	0.02%	0.53%	4.31%	57.19	280.65	337.83	16.93%
8	Carbonate Concentrate	26.09	1.59	15.91	19.19%	27.26%	14.47%	162.27	293.73	456.01	35.59%
9	Fine Concentrate	28.12	1.30	7.71	0.37%	1.03%	0.35%	46.34	631.01	677.35	6.84%
10	Silica Flotation Feed	24.97	1.73	16.01	28.74%	45.34%	10.46%	115.94	517.11	633.05	18.31%
11	Silica Flotation Tails	7.38	0.39	75.07	23.81%	33.32%	14.77%	23.15	148.51	171.66	13.49%
12	Silica Flotation Concentrate	29.19	2.10	2.34	29.54%	37.10%	18.73%	92.78	418.33	511.11	18.15%

Test Objective: Real Run – 1

Comments:

Pilot plant operation was relatively smooth.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet ApprovedDateCLDRI______Jacobs______

Test Number: Real Run – 2 Date of Test: 16-Oct-00 Pebble Sample: IMC

	Stream	Analyses (% of Solids by Weight)				by Weight)		Rates (kg/h)			Cw
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.69	3.22	11.90	n/a	n/a	n/a	261.45	28.91	290.36	90.04%
2	Logwasher O'Flow	16.78	8.47	7.91	n/a	n/a	n/a	17.01	723.89	740.91	2.30%
3	Rod Mill Discharge	24.14	3.38	10.50	n/a	n/a	n/a	244.43	135.01	379.45	64.42%
4	Mill Classifier O'Size	24.99	2.54	13.04	71.92%	15.15%	3.96%	277.14	116.01	393.15	70.49%
5	Mill Classifier Feed	24.59	3.02	12.13	43.62%	18.02%	8.38%	508.93	516.45	1,025.38	49.63%
6	Flotation Feed	24.39	3.66	10.58	10.00%	20.92%	13.28%	231.79	411.16	642.95	36.05%
7	Carbonate Tails	16.12	9.78	4.33	0.57%	5.09%	9.67%	67.43	322.26	389.69	17.30%
8	Carbonate Concentrate	26.85	1.25	13.74	14.25%	27.19%	17.04%	164.36	291.01	455.38	36.09%
9	Fine Concentrate	27.79	1.06	6.78	0.92%	2.08%	0.49%	66.19	779.65	845.84	7.83%
10	Silica Flotation Feed	26.13	1.29	15.62	16.75%	45.83%	10.35%	98.17	188.50	286.67	34.25%
11	Silica Flotation Tails	6.55	0.31	77.02	20.83%	32.84%	17.45%	19.26	260.77	280.03	6.88%
12	Silica Flotation Concentrate	29.86	1.58	3.13	18.27%	48.83%	10.15%	78.91	200.71	279.62	28.22%

Test Objective: Real Run – 2

Comments:

Pilot plant operation was relatively smooth.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

	Approved	Date
CLDRI		
Jacobs		

Test Number: Real Run – 3 Date of Test: 18-Oct-00 Pebble Sample: IMC

	Stream			Analyses	(% of Solids	by Weight)	F	Cw			
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.74	3.22	10.59	n/a	n/a	n/a	274.25	30.39	304.64	90.02%
2	Logwasher O'Flow	16.81	8.39	8.66	n/a	n/a	n/a	18.26	679.64	697.90	2.62%
3	Rod Mill Discharge	23.92	3.28	11.46	n/a	n/a	n/a	255.99	156.37	412.35	62.08%
4	Mill Classifier O'Size	24.58	2.50	13.69	75.03%	14.13%	1.24%	332.13	135.04	467.18	71.09%
5	Mill Classifier Feed	24.40	2.95	12.43	44.64%	20.21%	3.09%	574.22	625.04	1,199.26	47.88%
6	Flotation Feed	23.92	3.55	10.84	11.23%	25.44%	4.42%	242.08	424.16	666.24	36.34%
7	Carbonate Tails	16.98	9.37	3.84	0.10%	1.83%	2.79%	60.37	253.26	313.63	19.25%
8	Carbonate Concentrate	26.39	1.39	13.43	12.92%	31.55%	13.92%	181.71	352.58	534.29	34.01%
9	Fine Concentrate	28.57	1.27	7.80	0.87%	1.48%	0.59%	49.58	600.19	649.78	7.63%
10	Silica Flotation Feed	25.52	1.42	15.82	18.15%	44.48%	18.46%	132.13	298.25	430.37	30.70%
11	Silica Flotation Tails	6.44	0.36	77.38	17.08%	38.21%	15.64%	21.24	116.47	137.71	15.43%
12	Silica Flotation Concentrate	29.79	1.69	2.30	18.63%	46.00%	19.14%	110.88	239.31	350.19	31.66%

Test Objective: To improve MgO rejection adjusting frother paddle so the gap between paddle and weirs is constant. Reagent dosages were the same as Real Run - 2.

Comments: The air valves on the first four small cells were almost closed. The froth was foaming. Pilot plant operation was relatively smooth.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

	Approved	Date
CLDRI		
Jacobs		

Test Number: Real Run – 4 Date of Test: 23-Oct-00 Pebble Sample: IMC

	Stream			Analyses	(% of Solids	Rates (kg/h)			Cw		
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.16	3.46	10.85	n/a	n/a	n/a	274.23	31.27	305.50	89.76%
2	Logwasher O'Flow	17.06	7.95	8.53	n/a	n/a	n/a	18.18	680.29	698.46	2.60%
3	Rod Mill Discharge	24.00	3.25	10.90	n/a	n/a	n/a	256.05	165.08	421.13	60.80%
4	Mill Classifier O'Size	24.88	2.49	12.28	75.91%	12.90%	1.28%	297.62	139.63	437.26	68.07%
5	Mill Classifier Feed	24.68	2.92	11.75	44.85%	20.20%	3.02%	535.30	591.14	1,126.44	47.52%
6	Flotation Feed	24.25	3.42	11.18	12.65%	26.57%	4.26%	237.68	453.71	691.39	34.38%
7	Carbonate Tails	14.77	10.66	3.68	0.25%	3.39%	3.66%	53.72	337.55	391.27	13.73%
8	Carbonate Concentrate	26.77	1.15	13.48	12.71%	30.97%	13.11%	183.96	442.25	626.21	29.38%
9	Fine Concentrate	28.79	1.03	7.77	0.61%	1.16%	0.47%	59.99	744.23	804.21	7.46%
10	Silica Flotation Feed	25.71	1.25	16.12	19.11%	45.48%	18.79%	123.97	214.43	338.40	36.63%
11	Silica Flotation Tails	5.71	0.33	78.44	10.38%	38.62%	20.42%	13.52	68.31	81.83	16.52%
12	Silica Flotation Concentrate	28.28	1.41	7.61	19.85%	47.34%	18.68%	110.45	229.93	340.38	32.45%

Test Objective: To depress dolomite using P2O5/H2SO4 ratio 2:1

Comments: Carbonate flotation froth was brittle; no launder water was used. All air valves were widely open.

Pilot plant operation was relatively smooth.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

	Approved	Date
CLDRI		
Jacobs		

Test Number: Real Run – 5 Date of Test: 25-Oct-00 Pebble Sample: IMC

	Stream			Analyses	(% of Solids	by Weight)		Rates (kg/h)			Cw
No.	Name	P_2O_5	MgO	Insol	> 100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.73	3.29	10.15	n/a	n/a	n/a	271.64	29.12	300.75	90.32%
2	Logwasher O'Flow	17.06	7.21	11.7	n/a	n/a	n/a	20.89	786.15	807.04	2.59%
3	Rod Mill Discharge	24.08	3.21	10.71	n/a	n/a	n/a	250.75	176.35	427.10	58.71%
4	Mill Classifier O'Size	24.67	2.46	12.69	77.33%	12.30%	1.87%	233.39	102.50	335.89	69.48%
5	Mill Classifier Feed	24.41	2.99	11.57	45.07%	19.16%	5.18%	477.25	574.27	1,051.51	45.39%
6	Flotation Feed	24.19	3.49	10.90	12.95%	25.79%	9.27%	243.86	470.10	713.96	34.16%
7	Carbonate Tails	15.96	9.87	3.99	0.40%	5.18%	3.81%	65.46	435.65	501.11	13.06%
8	Carbonate Concentrate	27.05	1.05	13.60	13.20%	31.85%	13.37%	178.40	367.96	546.36	32.65%
9	Fine Concentrate	28.87	1.00	7.23	0.48%	1.14%	0.53%	49.79	566.45	616.24	8.08%
10	Silica Flotation Feed	25.95	1.09	15.88	19.89%	43.59%	17.72%	128.60	308.05	436.65	29.45%
11	Silica Flotation Tails	5.06	0.27	82.77	15.15%	40.33%	15.88%	19.87	78.69	98.56	20.16%
12	Silica Flotation Concentrate	29.56	1.30	4.76	22.56%	45.54%	16.64%	108.73	251.70	360.43	30.17%

Test Objective: To reject more MgO and increase P2O5 grade in the concentrate adding more PA-31 in the 3rd

Comments: Carbonate flotation froth was brittle; no launder water was used. All air valves were widely open. Silicate flot. did O.K. due to amine dosage was increased back to Real Run – 3' level and kerosene was up. The froth paddle on the large cells of carbonate flotation was increased to 59 times/min from 34 times/min. Pilot plant operation was relatively smooth.

Approved

CLDRI

Jacobs

Date

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

Test Number: Real Run – 6 Date of Test: 30-Oct-00 Pebble Sample: IMC

	Stream			Analyses	(% of Solids	F	Cw				
No.	Name	P_2O_5	MgO	Insol	> 100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.41	3.50	9.97	n/a	n/a	n/a	266.52	29.57	296.09	90.01%
2	Logwasher O'Flow	16.11	9.24	6.73	n/a	n/a	n/a	13.45	475.15	488.60	2.75%
3	Rod Mill Discharge	23.85	3.37	10.78	n/a	n/a	n/a	253.08	203.38	456.46	55.44%
4	Mill Classifier O'Size	24.66	2.42	13.16	76.30%	13.41%	1.98%	219.05	92.38	311.42	70.34%
5	Mill Classifier Feed	24.42	3.01	11.83	44.61%	20.78%	4.71%	474.40	531.96	1,006.36	47.14%
6	Flotation Feed	24.25	3.67	10.67	16.36%	25.79%	7.22%	255.36	492.50	747.85	34.15%
7	Carbonate Tails	17.67	9.16	4.25	0.23%	3.21%	2.22%	76.77	437.32	514.09	14.93%
8	Carbonate Concentrate	26.86	1.09	13.48	14.85%	34.40%	7.73%	178.59	273.01	451.60	39.55%
9	Fine Concentrate	29.44	1.06	7.05	1.06%	2.48%	0.70%	41.35	431.78	473.13	8.74%
10	Silica Flotation Feed	25.76	1.20	15.94	20.17%	43.23%	10.95%	137.23	292.12	429.35	31.96%
11	Silica Flotation Tails	4.90	0.29	82.13	8.77%	36.16%	12.35%	12.72	142.57	155.29	8.19%
12	Silica Flotation Concentrate	28.07	1.24	9.09	21.06%	43.79%	10.82%	124.51	255.23	379.74	32.79%

Test Objective: To test plant water using the same conditions as Real Run - 5.

Comments: Carbonate flotation behaved the same as that with tap water. However, the silicate flotation was poor. This could be because less water was used and the plant water contains more suspended solids. At 11:00 AM it was found that the 3rd flotation cell sheave was loose; the shaft was only 550 rpm, no air was induced. However, it was fixed. The test samples were taken after 12:15. Pilot plant operation was relatively smooth.

Approved Date

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Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

> Test Number: Real Run – 7 Date of Test: 30-Oct-00 Pebble Sample: IMC

	Stream	Analyses (% of Solids by Weight)						R	Cw		
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	24.24	3.58	9.46	n/a	n/a	n/a	192.48	17.29	209.77	91.76%
2	Logwasher O'Flow	16.23	9.61	6.72	n/a	n/a	n/a	11.05	654.07	665.12	1.66%
3	Rod Mill Discharge	23.52	3.82	9.93	n/a	n/a	n/a	181.43	154.19	335.62	54.06%
4	Mill Classifier O'Size	24.78	2.82	11.59	61.42%	23.46%	2.52%	317.65	163.28	480.93	66.05%
5	Mill Classifier Feed	24.37	3.43	11.44	32.55%	24.29%	3.22%	499.46	691.18	1,190.64	41.95%
6	Flotation Feed	24.02	3.91	10.36	2.15%	21.09%	6.36%	181.81	435.88	617.69	29.43%
7	Carbonate Tails	17.77	9.34	4.84	0.07%	2.36%	1.89%	53.48	325.59	379.07	14.11%
8	Carbonate Concentrate	27.16	1.16	12.32	1.79%	26.08%	9.23%	128.33	288.78	417.11	30.77%
9	Fine Concentrate	28.15	1.06	9.42	0.29%	4.70%	1.66%	52.17	536.74	588.91	8.86%
10	Silica Flotation Feed	25.89	1.27	16.06	2.85%	42.54%	15.11%	76.17	307.92	384.09	19.83%
11	Silica Flotation Tails	4.17	0.61	84.80	3.05%	44.14%	12.85%	11.94	73.99	85.94	13.90%
12	Silica Flotation Concentrate	30.13	1.28	3.27	3.13%	49.04%	12.55%	64.22	269.71	333.94	19.23%

Test Objective: Reducing flotation feed particle size to <5% +100 mesh and increasing #5&6 cells tip speed to 6.

Comments: Flotation of carbonates behaved the same as before. The DF-66 screen cloth was used. The circulating load was controlled at about 100% by reducing the feed rate to about 195 kg/hr.

Pilot plant operation was relatively smooth.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

Test Number: Real Run – 8 Date of Test: 2-Nov-00 Pebble Sample: IMC

	Stream		Analyses (% of Solids by Weight)						Rates (kg/h)			
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %	
1	Logwasher Feed	23.46	3.22	11.43	n/a	n/a	n/a	190.94	18.31	209.25	91.25%	
2	Logwasher O'Flow	16.00	8.86	6.97	n/a	n/a	n/a	10.91	550.03	560.94	1.95%	
3	Rod Mill Discharge	23.67	3.30	10.85	n/a	n/a	n/a	180.03	139.47	319.49	56.35%	
4	Mill Classifier O'Size	24.26	2.42	14.37	59.69%	25.00%	2.52%	244.86	125.55	370.41	66.11%	
5	Mill Classifier Feed	24.14	2.88	12.80	31.88%	25.28%	3.97%	423.18	517.46	940.64	44.99%	
6	Flotation Feed	23.93	3.56	11.02	2.42%	21.14%	5.56%	178.31	393.62	571.94	31.18%	
7	Carbonate Tails	19.88	7.02	6.13	0.05%	2.49%	4.32%	74.90	356.17	431.06	17.38%	
8	Carbonate Concentrate	26.75	0.92	14.28	1.75%	30.31%	17.60%	103.42	169.48	272.89	37.90%	
9	Fine Concentrate	28.44	0.93	9.41	0.36%	3.52%	2.02%	35.24	406.25	441.49	7.98%	
10	Silica Flotation Feed	25.72	0.98	16.85	2.74%	40.30%	25.56%	68.17	254.91	323.08	21.10%	
11	Silica Flotation Tails	5.77	0.18	80.47	3.23%	41.04%	23.76%	11.94	79.41	91.35	13.07%	
12	Silica Flotation Concentrate	30.26	1.15	2.99	2.98%	43.34%	28.38%	56.23	260.17	316.40	17.77%	

Test Objective: Test plant water @ flotation feed particle size <5% +100 mesh and increasing #5&6.

Comments: Flotation of carbonates behaved the same as before. The DF-66 screen cloth was used. The circulating load was controlled at about 100% by reducing the feed rate to about 195 kg/hr.

Pilot plant operation was relatively smooth.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

Test Number: Real Run – 9A Date of Test: 8-Nov-00 Pebble Sample: CF

	Stream			Analyses	(% of Solids	by Weight)	Rates (kg/h))	Cw
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.25	1.58	21.64	n/a	n/a	n/a	193.91	16.71	210.62	92.07%
2	Logwasher O'Flow	13.29	8.44	13.78	n/a	n/a	n/a	6.92	471.07	477.99	1.45%
3	Rod Mill Discharge	22.42	1.81	22.59	n/a	n/a	n/a	186.99	156.97	343.96	54.36%
4	Mill Classifier O'Size	21.78	1.04	29.07	60.85%	27.54%	2.48%	285.84	270.01	555.85	51.42%
5	Mill Classifier Feed	22.16	1.34	26.57	38.46%	29.07%	3.73%	455.65	496.47	952.12	47.86%
6	Flotation Feed	22.65	1.91	22.66	5.81%	27.26%	6.99%	169.81	377.44	547.25	31.03%
7	Carbonate Tails	20.43	4.68	14.39	0.22%	3.67%	5.95%	59.35	400.01	459.35	12.92%
8	Carbonate Concentrate	23.81	0.43	26.71	6.65%	37.25%	18.62%	110.47	161.87	272.34	40.56%
9	Fine Concentrate	26.70	0.59	17.40	0.54%	2.11%	0.32%	18.91	378.58	397.49	4.76%
10	Silica Flotation Feed	23.11	0.41	28.98	8.11%	43.19%	22.86%	91.56	237.46	329.02	27.83%
11	Silica Flotation Tails	5.67	0.13	81.28	8.07%	43.02%	18.44%	20.82	121.88	142.70	14.59%
12	Silica Flotation Concentrate	28.77	0.51	12.48	8.43%	45.02%	23.41%	70.74	191.96	262.70	26.93%

Test Objective: First run on Sample 2-CF according to CLDRI reagent dosages.

Comments: The DF-56 screen cloth was used. The mill pump sump water was split to screen oversize launder to reduce overgrinding as it can be seen that the weight % solids is only 51.42%. After quick analysis of flotation feed, carbonate tails and concentrate, it was found that the feed MgO was 1.8 and concentrate. MgO was ~0.5% and P2O5 in tails >20%. The dosages of PA-31 were reduced and flotation time was reduced by bypassing the two large cells. The test under the original conditions was referred to as Run – 9A, and that under the new conditions was referred to as Run 9 – B.

	Approved	Date
CLDRI		
Jacobs		

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

	Approved	Date
CLDRI		
Jacobs		

Test Number: Real Run – 9B Date of Test: 8-Nov-00 Pebble Sample: CF

Stream				Analyses	(% of Solids	Rates (kg/h)			Cw		
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	24.01	1.82	18.27	n/a	n/a	n/a	197.89	9.47	207.36	95.43%
2	Logwasher O'Flow	13.79	8.19	13.15	n/a	n/a	n/a	7.13	492.86	500.00	1.43%
3	Rod Mill Discharge	22.54	1.88	22.38	n/a	n/a	n/a	190.76	154.49	345.25	55.25%
4	Mill Classifier O'Size	22.42	1.08	27.24	66.75%	23.06%	1.98%	289.95	362.23	652.18	44.46%
5	Mill Classifier Feed	22.69	1.47	24.89	37.01%	26.23%	4.11%	482.86	650.81	1,133.67	42.59%
6	Flotation Feed	23.14	1.94	19.80	8.20%	29.16%	6.68%	192.91	459.00	651.91	29.59%
7	Carbonate Tails	14.59	8.48	9.89	0.21%	3.58%	4.71%	24.67	162.83	187.51	13.16%
8	Carbonate Concentrate	24.89	0.74	21.63	6.98%	31.09%	15.83%	168.23	498.77	667.00	25.22%
9	Fine Concentrate	26.64	0.99	15.01	0.13%	0.34%	0.15%	49.23	604.57	653.80	7.53%
10	Silica Flotation Feed	23.14	0.59	26.63	10.24%	45.26%	22.16%	119.00	376.42	495.42	24.02%
11	Silica Flotation Tails	4.72	0.13	84.64	7.87%	35.83%	19.94%	31.52	225.97	257.48	12.24%
12	Silica Flotation Concentrate	30.31	0.65	6.46	10.58%	44.49%	23.21%	87.49	252.69	340.17	25.72%

Test Objective: First run on Sample 2-CF according to CLDRI reagent dosages.

Comments: According to trial run of Run-9A and quick analysis of flotation feed and carbonate flotation concentrate, the feed MgO is only about 1.9%. The two large cells were bypassed and the PA-31 and acid mix dosages were reduced. The two large cells were used as a surge tank for amine flotation. Therefore, the amine flotation circuit was very stable during the run.

Pilot plant operation was relatively smooth.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

	Approved	Date
CLDRI		
Jacobs		

Test Number: Real Run – 10 Date of Test: 13-Nov-00 Pebble Sample: CF

Stream				Analyses	(% of Solids	Rates (kg/h)			Cw		
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	22.78	2.00	20.50	n/a	n/a	n/a	204.17	13.58	217.75	93.76%
2	Logwasher O'Flow	13.05	8.62	14.15	n/a	n/a	n/a	7.63	587.22	594.85	1.28%
3	Rod Mill Discharge	22.60	1.82	22.85	n/a	n/a	n/a	196.54	173.52	370.06	53.11%
4	Mill Classifier O'Size	22.15	1.06	27.98	54.85%	30.80%	4.19%	549.21	387.43	936.64	58.64%
5	Mill Classifier Feed	22.40	1.31	26.13	40.89%	30.11%	5.35%	717.62	700.81	1,418.43	50.59%
6	Flotation Feed	23.41	1.92	20.65	7.99%	23.01%	10.53%	168.41	368.10	536.51	31.39%
7	Carbonate Tails	20.58	5.13	12.67	0.07%	2.19%	2.90%	51.18	306.83	358.01	14.30%
8	Carbonate Concentrate	24.09	0.74	24.39	5.32%	32.13%	11.35%	117.23	181.43	298.66	39.25%
9	Fine Concentrate	26.00	1.09	15.37	0.49%	1.55%	0.49%	32.82	497.60	530.42	6.19%
10	Silica Flotation Feed	23.19	0.66	27.69	6.99%	41.40%	15.51%	84.41	166.75	251.16	33.61%
11	Silica Flotation Tails	3.95	0.16	86.56	5.89%	37.84%	13.66%	20.69	84.15	104.84	19.74%
12	Silica Flotation Concentrate	29.44	0.86	8.27	11.45%	49.19%	11.23%	63.72	164.53	228.25	27.92%

Test Objective: Flotation of carbonates using only sulfuric acid as depressant.

Comments: Flotation of carbonates used only first 4 small cells, and last cell's paddles didn't work. The DF-56 screen cloth was used. Feed rate was about 200 kg/hr.

Grinding circuit was not stable due to the pump working discontinuously. Big pieces of rubber in the line.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

> Test Number: Real Run – 11 Date of Test: 13-Nov-00 Pebble Sample: CF

	Stream			Analyses	(% of Solids	Rates (kg/h)			Cw		
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.04	1.86	20.75	n/a	n/a	n/a	202.38	18.00	220.38	91.83%
2	Logwasher O'Flow	13.43	8.41	14.42	n/a	n/a	n/a	9.07	688.56	697.63	1.30%
3	Rod Mill Discharge	22.68	1.76	22.73	n/a	n/a	n/a	193.31	97.79	291.09	66.41%
4	Mill Classifier O'Size	21.73	0.97	29.88	48.82%	34.04%	4.76%	453.91	310.68	764.59	59.37%
5	Mill Classifier Feed	22.45	1.13	27.48	36.75%	33.31%	6.21%	617.38	454.51	1,071.90	57.60%
6	Flotation Feed	22.93	1.79	21.52	3.77%	24.70%	12.29%	163.47	321.63	485.10	33.70%
7	Carbonate Tails	20.48	4.93	13.78	0.03%	1.93%	3.09%	42.67	242.14	284.81	14.98%
8	Carbonate Concentrate	23.92	0.71	24.61	4.01%	28.16%	12.40%	120.80	211.17	331.97	36.39%
9	Fine Concentrate	26.03	1.09	16.09	0.28%	1.48%	0.58%	38.08	539.32	577.40	6.60%
10	Silica Flotation Feed	22.84	0.59	28.59	5.56%	39.88%	17.19%	82.72	185.22	267.94	30.87%
11	Silica Flotation Tails	3.64	0.12	86.97	6.84%	44.76%	12.41%	27.74	56.26	84.00	33.02%
12	Silica Flotation Concentrate	32.11	0.76	1.65	5.95%	54.68%	9.79%	54.98	174.95	229.93	23.91%

Test Objective: Flotation of carbonates using only sulfuric acid as depressant at higher dosage than Real Run 10.

Comments: Flotation of carbonates behaved the same as before. The DF-56 screen cloth was used. Feed rate was about 200 kg/hr.

Grinding circuit was not stable. Big pieces of rubber in the line.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

Test Number: Real Run – 12 Date of Test: 15-Nov-00 Pebble Sample: CF

Stream				Analyses	(% of Solids	Rates (kg/h)			Cw		
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	22.32	1.79	23.00	n/a	n/a	n/a	184.05	17.67	201.71	91.24%
2	Logwasher O'Flow	13.18	8.53	14.78	n/a	n/a	n/a	8.79	950.29	959.08	0.92%
3	Rod Mill Discharge	22.22	1.72	24.47	n/a	n/a	n/a	175.26	98.59	273.85	64.00%
4	Mill Classifier O'Size	24.03	1.33	20.77	90.94%	3.58%	0.49%	48.48	110.62	159.10	30.47%
5	Mill Classifier Feed	22.68	1.62	23.28	47.48%	15.12%	2.87%	215.78	342.36	558.14	38.66%
6	Flotation Feed	22.41	1.72	23.98	38.18%	18.32%	3.22%	167.30	315.68	482.98	34.64%
7	Carbonate Tails	17.44	7.71	9.29	0.37%	2.70%	1.86%	22.10	159.32	181.43	12.18%
8	Carbonate Concentrate	23.15	0.83	25.99	44.35%	19.87%	3.40%	145.19	217.90	363.09	39.99%
9	Fine Concentrate	27.23	1.25	12.34	0.00%	0.02%	0.14%	32.43	574.85	607.27	5.34%
10	Silica Flotation Feed	21.87	0.75	30.18	53.19%	25.38%	6.65%	112.77	170.18	282.94	39.85%
11	Silica Flotation Tails	7.23	0.21	75.93	51.83%	24.49%	5.64%	37.34	160.41	197.75	18.88%
12	Silica Flotation Concentrate	29.48	1.20	5.47	56.94%	26.80%	4.59%	75.43	163.86	239.28	31.52%

Test Objective: Coarse grinding using DF36 screen cloth to make 42 mesh (0.354 mm) cut.

Comments: Flotation of carbonates behaved the same as before.

The DF-56 screen cloth was used. The circulating load was controlled at about 100% by reducing the feed rate to about 195 kg/hr.

Approved Date

CLDRI

Jacobs

Pilot plant operation was relatively smooth.
Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

> Test Number: Real Run – 13 Date of Test: 20-Nov-00 Pebble Sample: CF

	Stream	Analyses (% of Solids by Weight)							Rates (kg/h)		
No.	Name	P_2O_5	MgO	Insol	> 100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	22.29	1.72	23.37	n/a	n/a	n/a	194.96	16.50	211.46	92.20%
2	Logwasher O'Flow	13.31	8.41	14.96	n/a	n/a	n/a	8.81	1,069.12	1,077.93	0.82%
3	Rod Mill Discharge	23.05	1.85	21.11	n/a	n/a	n/a	186.15	115.56	301.71	61.70%
4	Mill Classifier O'Size	22.81	1.25	24.54	73.49%	12.68%	2.68%	387.54	224.88	612.42	63.28%
5	Mill Classifier Feed	22.82	1.59	22.72	35.96%	19.97%	7.33%	563.00	714.74	1,277.75	44.06%
6	Flotation Feed	22.94	1.71	22.48	15.81%	24.62%	9.26%	175.46	303.76	479.22	36.61%
7	Carbonate Tails	18.02	6.74	12.12	0.28%	3.77%	3.26%	27.54	127.57	155.10	17.75%
8	Carbonate Concentrate	23.95	0.80	24.46	16.60%	26.09%	7.46%	147.92	260.36	408.28	36.23%
9	Fine Concentrate	26.22	1.18	14.68	0.21%	0.33%	0.09%	46.03	543.08	589.11	7.81%
10	Silica Flotation Feed	22.74	0.68	28.19	23.32%	36.48%	11.81%	101.89	250.50	352.40	28.91%
11	Silica Flotation Tails	4.53	0.18	85.14	26.75%	39.06%	6.62%	30.14	108.63	138.77	21.72%
12	Silica Flotation Concentrate	30.63	0.85	3.81	24.05%	39.46%	11.63%	71.75	160.52	232.27	30.89%

Test Objective: Finer grinding using DF-48 (60 mesh) screen.

Comments: Flotation of carbonates behaved the same as before.

Pilot plant operation was relatively smooth.

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Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

> Test Number: Real Run – 14 Date of Test: 22-Nov-00 Pebble Sample: CF

	Stream	Analyses (% of Solids by Weight)							Rates (kg/h)		
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.07	2.45	17.07	n/a	n/a	n/a	197.46	13.76	211.21	93.49%
2	Logwasher O'Flow	12.69	8.60	14.60	n/a	n/a	n/a	9.08	1,065.25	1,074.34	0.85%
3	Rod Mill Discharge	23.09	1.87	20.95	n/a	n/a	n/a	188.37	94.52	282.90	66.59%
4	Mill Classifier O'Size	23.42	1.27	23.09	79.74%	9.58%	1.10%	78.15	60.91	139.06	56.20%
5	Mill Classifier Feed	23.73	1.89	19.33	35.34%	20.34%	3.84%	274.44	397.22	671.66	40.86%
6	Flotation Feed	23.28	1.95	22.56	16.80%	24.64%	5.11%	196.29	411.53	607.82	32.29%
7	Carbonate Tails	16.77	7.34	11.35	0.22%	3.86%	3.45%	27.07	138.05	165.12	16.40%
8	Carbonate Concentrate	24.04	0.83	24.18	19.21%	28.31%	12.39%	169.21	285.06	454.27	37.25%
9	Fine Concentrate	26.46	1.19	14.17	0.00%	0.07%	0.12%	46.46	481.68	528.14	8.80%
10	Silica Flotation Feed	23.24	0.71	26.83	25.81%	40.11%	17.47%	122.75	266.59	389.34	31.53%
11	Silica Flotation Tails	2.95	0.14	88.80	29.52%	40.25%	11.72%	33.77	99.34	133.11	25.47%
12	Silica Flotation Concentrate	31.43	0.89	3.34	24.91%	39.86%	17.26%	88.98	241.84	330.82	26.90%

Test Objective: Test plant water using the same conditions as Real Run - 13.

Comments: Flotation of carbonates behaved the same as before.

Pilot plant operation was relatively smooth.

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Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

Test Number: Real Run – 15 Date of Test: 28-Nov-00 Pebble Sample: CF

	Stream	Analyses (% of Solids by Weight)							Rates (kg/h)		
No.	Name	P_2O_5	MgO	Insol	> 100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.15	1.82	20.13	n/a	n/a	n/a	198.58	13.39	211.97	93.68%
2	Logwasher O'Flow	13.64	7.23	20.01	n/a	n/a	n/a	10.88	1,245.14	1,256.02	0.87%
3	Rod Mill Discharge	23.11	2.00	20.20	n/a	n/a	n/a	187.71	118.62	306.33	61.28%
4	Mill Classifier O'Size	23.30	1.45	22.55	82.86%	7.22%	1.58%	176.98	166.97	343.95	51.45%
5	Mill Classifier Feed	23.20	1.84	21.22	40.28%	18.42%	6.78%	361.97	569.48	931.45	38.86%
6	Flotation Feed	22.85	2.02	22.02	21.39%	23.09%	9.11%	185.00	468.73	653.73	28.30%
7	Carbonate Tails	18.48	6.28	11.89	0.34%	3.47%	2.83%	39.38	248.39	287.77	13.68%
8	Carbonate Concentrate	23.80	0.78	24.45	22.76%	27.27%	7.80%	145.62	231.97	377.59	38.56%
9	Fine Concentrate	27.01	1.00	13.94	0.00%	0.05%	0.05%	37.55	484.22	521.76	7.20%
10	Silica Flotation Feed	22.96	0.72	27.35	30.11%	36.15%	10.90%	108.07	247.12	355.18	30.43%
11	Silica Flotation Tails	4.51	0.15	83.93	36.45%	32.78%	8.09%	33.26	86.97	120.23	27.66%
12	Silica Flotation Concentrate	31.42	1.01	2.26	32.31%	40.72%	11.62%	74.81	242.75	317.56	23.56%

Test Objective: Test alternate collector PA-31 using the same conditions as Real Run - 13.

Comments: Flotation of carbonates behaved differently than before. Less acids were used for the same range pH values.

Foaming frother.

Pilot plant operation was relatively smooth.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

Test Number: Real Run – 16 Date of Test: 30-Nov-00 Pebble Sample: IMC

	Stream	Analyses (% of Solids by Weight)							Rates (kg/h)		
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.49	3.68	9.76	n/a	n/a	n/a	211.88	12.95	224.82	94.24%
2	Logwasher O'Flow	17.51	7.35	8.70	n/a	n/a	n/a	9.30	448.85	458.16	2.03%
3	Rod Mill Discharge	23.82	3.31	10.93	n/a	n/a	n/a	202.57	89.82	292.40	69.28%
4	Mill Classifier O'Size	24.95	2.48	11.70	87.90%	3.23%	0.95%	90.35	69.19	159.54	56.63%
5	Mill Classifier Feed	24.30	3.44	8.88	38.78%	14.62%	5.57%	289.00	492.78	781.78	36.97%
6	Flotation Feed	24.22	3.42	10.71	21.73%	20.79%	7.58%	198.65	465.16	663.81	29.93%
7	Carbonate Tails	15.69	9.65	4.33	0.43%	3.74%	2.52%	43.83	299.64	343.47	12.76%
8	Carbonate Concentrate	26.63	1.40	12.89	24.31%	26.14%	7.01%	154.82	347.96	502.78	30.79%
9	Fine Concentrate	28.99	1.22	6.93	0.00%	0.04%	0.07%	41.86	607.57	649.43	6.45%
10	Silica Flotation Feed	25.79	1.45	15.76	34.77%	35.76%	9.31%	112.96	239.78	352.74	32.02%
11	Silica Flotation Tails	6.67	0.32	77.42	26.28%	32.93%	10.01%	15.20	53.96	69.16	21.97%
12	Silica Flotation Concentrate	28.29	1.71	6.05	37.22%	36.86%	9.06%	97.76	210.87	308.63	31.68%

Test Objective: Coarse grinding using DF-43 screen (48 mesh).

Comments: Flotation of carbonates behaved the same as before.

The drive pulley on the big cells was changed so that the tip speeds were about 5.64 M/sec. All suction inlets were plugged. There was no boiling in the cells. Air was induced. Froth phase was stable.

Pilot plant operation was relatively smooth.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

> Test Number: Real Run – 17 Date of Test: 4-Dec-00 **Pebble Sample: IMC**

	Stream	Analyses (% of Solids by Weight)							Rates (kg/h)		
No.	Name	P_2O_5	MgO	Insol	> 100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.38	3.39	10.79	n/a	n/a	n/a	212.84	10.15	222.98	95.45%
2	Logwasher O'Flow	17.03	6.47	14.40	n/a	n/a	n/a	12.66	598.93	611.59	2.07%
3	Rod Mill Discharge	24.07	3.34	9.77	n/a	n/a	n/a	200.18	95.29	295.47	67.75%
4	Mill Classifier O'Size	24.90	2.41	11.60	80.70%	12.67%	1.02%	251.53	157.18	408.71	61.54%
5	Mill Classifier Feed	24.47	2.78	11.59	46.39%	16.42%	2.84%	424.65	453.22	877.88	48.37%
6	Flotation Feed	23.88	3.40	10.76	7.58%	22.79%	4.65%	173.12	458.13	631.26	27.43%
7	Carbonate Tails	16.95	8.91	4.47	0.16%	2.63%	3.00%	53.99	298.63	352.62	15.31%
8	Carbonate Concentrate	27.25	1.00	12.59	6.17%	26.09%	14.21%	119.13	246.50	365.63	32.58%
9	Fine Concentrate	28.49	0.98	7.80	0.07%	0.14%	0.21%	42.83	577.36	620.19	6.91%
10	Silica Flotation Feed	26.11	1.06	15.51	11.25%	42.58%	21.77%	76.30	164.41	240.70	31.70%
11	Silica Flotation Tails	4.45	0.15	84.94	9.49%	38.23%	21.45%	10.96	91.95	102.91	10.65%
12	Silica Flotation Concentrate	30.04	1.23	3.33	12.27%	42.74%	23.66%	65.33	232.22	297.55	21.96%

Test Objective: Test only phos acid as depressant @ 80 mesh (DF-66) cut.

Comments: Flotation of carbonates behaved the same as before.

The tip speeds on big cells were kept at 5.64 M/sec. No boiling.

Approved Date CLDRI Jacobs

Pilot plant operation was relatively smooth.

Measured Analyses & Rates for Material Balance Calculations CLDRI Dolomite Flotation Process – Jacobs Pilot Plant Reference Flowsheet: See Flowsheet

	Approved	Date
CLDRI		
Jacobs		

Test Number: Real Run – 18 Date of Test: 6-Dec-00 Pebble Sample: IMC

	Stream	Analyses (% of Solids by Weight)							Rates (kg/h)		
No.	Name	P_2O_5	MgO	Insol	>100 M	100/200 M	200/270 M	Solids	Water	Slurry	Solids %
1	Logwasher Feed	23.53	3.30	9.71	n/a	n/a	n/a	192.40	11.94	204.34	94.16%
2	Logwasher O'Flow	16.57	7.89	6.79	n/a	n/a	n/a	8.75	598.20	606.95	1.44%
3	Rod Mill Discharge	23.63	3.42	9.98	n/a	n/a	n/a	183.65	91.40	275.05	66.77%
4	Mill Classifier O'Size	24.43	2.61	11.77	82.58%	11.86%	1.23%	114.65	75.73	190.38	60.22%
5	Mill Classifier Feed	23.91	3.17	10.95	38.07%	18.70%	5.11%	285.94	373.35	659.28	43.37%
6	Flotation Feed	23.83	3.58	10.61	7.63%	22.93%	9.21%	171.29	460.24	631.53	27.12%
7	Carbonate Tails	15.05	9.72	3.97	0.16%	3.15%	3.59%	49.76	265.84	315.61	15.77%
8	Carbonate Concentrate	27.13	1.03	12.75	6.49%	26.03%	15.63%	121.53	268.31	389.83	31.17%
9	Fine Concentrate	28.74	0.97	8.26	0.00%	0.01%	0.07%	43.89	533.26	577.15	7.60%
10	Silica Flotation Feed	26.14	1.14	15.82	10.50%	39.74%	24.54%	77.64	253.06	330.70	23.48%
11	Silica Flotation Tails	7.95	0.29	73.23	10.99%	36.62%	22.27%	13.59	88.01	101.60	13.38%
12	Silica Flotation Concentrate	30.41	1.26	2.00	12.26%	43.36%	22.39%	64.05	355.52	419.57	15.26%

Test Objective: Test only phos acid as depressant @ 80 mesh (DF-66) cut with no acids to big cells and no PA-31.

Comments: Flotation of carbonates behaved the same as before. The tip speeds on big cells were kept at 5.64 M/sec. No boiling.

Pilot plant operation was relatively smooth.

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Appendix D

EQUIPMENT LIST – 300 TPH BATTERY LIMITS DOLOMITE FLOTATION PLANT

EQUIPMENT LIST – 300 TPH BATTERY LIMITS DOLOMITE FLOTATION PLANT

Area	Eq.	Number			Equipment				Hp
No.	No.	Work	Spare	Title	Size	Туре	Material	Unit	Total
1	E1	1	0	Pebble Surge Bin	600 ton	fabricated	Carbon steel	na	na
1	E2	2	0	Pebble Feeders	50 to 200 tph	electro mechanical	By vendor	5	10
1	E3	2	0	Log Washers	38" x 30' L	EIW - Logwasher	By vendor	200	400
1	E4	1	0	O'flow Pump Box	5.5 ft dia.x6.0 ft height	fabricated	Carbon steel	na	na
1	E5	1	0	Feed Scavenger Pump	1,140 gpm, 60 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	30	30
1	E6	1	0	Feed Scavenger Cyclone	DS20LB	Krebs	By vendor	na	na
1	E7	1	0	Ball Mill	16.5 ft. dia x 27 ft. L	Svedalla	By vendor	4500	4500
1	E8	1	0	Mill Pump Box	10 ft dia.x8 ft height	fabricated	Carbon steel	na	na
1	E9	1	0	Mill Pump	5,870 gpm, 60 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	250	250
1	E10	2	1	Mill Cyclones	3 DS33/Cluster	Krebs 3 Units/Cluster	By vendor	na	na
1	E11	2	0	Dolomite Conditioner Tanks	5.5 ft dia.x6.0 ft height	fabricated	Carbon steel	na	na
1	E12	2	0	Tank Agitators	Impeller Dia. 40"	Lightnin	By vendor	25	50
1	E13	5	0	Dolomite Flotation Cells	5 RCS 30 (1060 ft ³)	Svedalla	By vendor	60	300
1	E14	1	0	Cell Air Blower	2240 ICFM	multi stage	By vendor	75	75
1	E15	1	0	Dolomite Froth Pump	1,010 gpm, 40 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	30	30
1	E16	1	0	1st Rghr Concentrate Pump Box	10 ft dia.x8 ft height	fabricated	Carbon steel	na	na
1	E17	1	0	1st Rghr Concentrate Pump	3,650 gpm, 67 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	125	125
1	E18	1	0	Acid Slurry Thickener (AST)	100 ft dia x 30 ft H	High Rate Thickener	By vendor	10	10
1	E19	1	1	AST Underflow Pump	825 gpm, 60 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	60	60
1	E20	2	0	Concentrate Storage Tanks	46 ft dia.x46 ft height	fabricated	Carbon steel	na	na
1	E21	2	0	Storage Tank Agitators	Impeller Dia. 17'3"		By vendor	200	400
1	E22	1	0	AST Overflow Pump Box	5.5 ft dia.x6.0 ft height	fabricated	Carbon steel	na	na
1	E23	1	0	AST Overflow Pump	2,075 gpm, 73 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	100	100
1	E24	1	0	General Mill Tailing Pump Box	5.5 ft dia.x6.0 ft height	fabricated	Carbon steel	na	na
1	E25	1	0	General Mill Tailing Pump	2,640 gpm, 115 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	125	125
1	E26	1	0	Tailings Return Water Pump	2,010 gpm, 150 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	125	125
				Dolomite Flotation Reagent Farm					
1		6	0	field fabricated tanks					0
1		2	0	tank agitators			By vendor	25	50
1		7	0	reagent pumps			By vendor	7.5	52.5
1		1	0	PA-31 Rail Car Unloading Pump	600 gpm, 150 ft TDH		Ductile Iron	40	40
1		0	2	Clean up Pumps	440 gpm, 120 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	60	0
1		1	0	Bridge Crane	25 ton, 67 ft span, 40 ft lift	Overhead Crane	By vendor	30	30
1		1	0	Instrument&Plant Air Compressor	770 gpm @125 psi w/200 gl	Rotary screw	By vendor	25	25
1		1	0	Flotation Feed Sampler	LF-7	Inter Systems	By vendor	0	0
1		1	0	Carbonate tailing Sampler	LF-7	Inter Systems	By vendor	0	0
1		1	0	1st Rghr Concentrate Sampler	LF-7	Inter Systems	By vendor	0	0
Battery I	imits To	tals for l	Dolomit	te Flotation Only					6.788

EQUIPMENT LIST – 300 TPH BATTERY LIMITS DOLOMITE FLOTATION PLANT (CONT.)

Area	Eq.	Nui	nber		Equipment		-		Hp
No.	No.	Work	Spare	Title	Size	Туре	Material	Unit	Total
2	E27	3	0	1st Rghr Concentrate Cyclones	3 DS15LB/Cluster	Krebs 3 Units/Cluster	By vendor	na	na
2	E28	1	0	2nd Rghr Concentrate Pump Box	5.5 ft dia.x6.0 ft height	fabricated	Carbon steel	na	na
2	E29	1	0	2nd Rghr Concentrate Pump	2,730 gpm, 67 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	125	125
2	E30	3	0	2nd Rghr Concentrate Cyclones	3 DS15LB/Cluster	Krebs 3 Units/Cluster	By vendor	na	na
2	E31	2	0	Silica Conditioner Tanks	10 ft dia.x8 ft height	fabricated	Carbon steel	na	na
2	E32	2	0	Silica Conditioner Agitators	Impeller Dia. 40"	Lightnin	By vendor	25	50
2	E33	2	0	Silica Flotation Cells	2 RCS 15 (530 ft ³)	fabricated	Carbon steel	40	80
2	E34	1	0	Coarse Concentrate Pump Box	5.5 ft dia.x6.0 ft height	fabricated	Carbon steel	na	na
2	E35	1	0	Coarse Concentrate Pump	1,490 gpm, 40 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	30	30
2	E36	1	0	Coarse Concentrate Thickener	100 ft dia x 30 ft H	High Rate Thickener	By vendor	10	10
2	E37	1	0	CCT Overflow Pump Box	5.5 ft dia.x6.0 ft height	fabricated	Carbon steel	na	na
2	E38	1	0	CCT Overflow Pump	925 gpm, 73 ft TDH	H. Centrifugal Slurry Pump	Gasite 28G	30	30
				Silica Flotation Reagent Farm					
		6		field fabricated tanks				0	0
		2		tank agitators			By vendor	25	50
		6		reagent pumps			By vendor	7.5	45
2		1	0	Fine Concetrate Sampler	LF-7	Inter Systems	By vendor		0
2		1	0	Coarse Concentrate Sampler	LF-7	Inter Systems	By vendor		0
2		1	0	Silica Tailing Sampler	LF-7	Inter Systems	By vendor		0
Battery I	imits To	tals for	Silica F	lotation Only					420
Battery I	imits To	tals for	Dolomi	te and Silica Flotation					7,208