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ANIONIC FLOTATION OF FLORIDA PHOSPHATE



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FLORIDA INSTITUTE OF PHOSPHATE RESEARCH



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of
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PERSPECTIVE

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Florida phosphate ore occurs in deposits of unconsolidated marine sediments. Prior to the development of a reliable flotation process, the ore was beneficiated by washing and classification to obtain pebble and debris fractions. The pebble product was marketable phosphate rock and debris was wasted.

The advent of froth flotation significantly increased the phosphate reserves that could be economically exploited in the Florida land pebble district. Many flotation schemes were examined, but the Crago process, which was patented in 1942, became the standard method of extracting phosphate rock from the deslimed washer debris. The Crago process achieved its inventors' objective to "provide an improved method of concentration which will be economical and practical and which will not only facilitate the production of concentrates of high grade, but will also result in the recovery of a high percentage of the phosphate values of the ore". However, the Crago process evolved in an industry where the economics of easily recoverable inexpensive pebble dominated beneficiation.

As mining advances from high pebble deposits to high concentrate deposits, the cost to produce a ton of combined pebble and concentrate increases. Therefore, Florida Institute of Phosphate Research has assigned high priority to research efforts to improve phosphate recovery at lower cost. In this regard, the Institute granted Zellars-Williams Company funds to evaluate the applicability of an anionic rougher-cleaner process on present and future ores from Florida as compared to the Crago process.

In order to achieve these goals, three samples of phosphate ore were obtained, two of which were selected for use in the evaluation of anionic rougher-cleaner flotation. One sample (present ore) was selected to represent presently mined ore. The second sample, obtained from lower zone material containing carbonate minerals, was chosen to represent future ore.

Six commercially available anionic collectors were selected for evaluation. Three of the anionic collectors were fatty acid type reagents obtained from Florida reagent vendors. The fourth anionic collector, a petroleum sulfonate, was obtained from a domestic vendor. The fifth and sixth anionic collectors, an organophosphoric acid and an N-substituted sarcosine, were obtained from European vendors.

Thirty-six formal bench scale flotation tests were conducted to evaluate the six anionic collectors on 28 x 150 mesh flotation feed obtained from present ore. The three most promising reagents were examined in a second set of 36 tests to identify process variables that significantly influenced flotation performance. After the critical variables had been identified, a third series of tests was conducted to optimize the anionic rougher-cleaner process performance. A parallel series of tests were conducted to optimize the Crago process performance.

Additional testing was carried out to evaluate anionic rougher-cleaner flotation of 14 x 150 mesh, 14 x 35 mesh, and 35 x 150 mesh size fractions of flotation feed obtained from present ore. The program for these feed fractions included sets of tests to identify critical process variables and to optimize the performances of the anionic rougher-cleaner and Crago processes.

Relative to the Crago process, the anionic rougher-cleaner process gave lower grade concentrate, higher phosphate recovery, and lower reagent cost for 28 x 150 mesh feed. Chemical analyses indicate the anionic concentrate is a suitable feedstock for a commercial acidulation plant.

A field investigation of six phosphate plants followed by conceptual engineering indicated that these plants could be easily modified to utilize the anionic rougher-cleaner process. The estimated cost to modify a flotation plant having 1100 tph feed capacity is \$88K to \$123K. A grass-roots plant of the same capacity would cost about \$1.3 million less if the anionic rougher-cleaner process was utilized in place of the Crago process.

Test results from bench scale flotation of the 14 x 150 mesh, 14 x 35 mesh and 35 x 150 mesh, size fractions of present feed confirmed that the anionic rougher-cleaner process gives lower concentrate grade and lower reagent cost than the Crago process. Reagent cost savings for these feeds ranged from 36 to 46 percent of the Crago reagent costs.

The test program for 28 x 150 mesh flotation feed obtained from future ore was essentially identical to that described previously for present 28 x 150 mesh feed. Three sets of tests were conducted to evaluate the six anionic collectors, identify the critical process variables, and to optimize process performance.

Relative to the Crago process, the anionic rougher-cleaner process gave lower grade concentrate and lower reagent costs. Neither process was effective in rejecting carbonate minerals from the concentrate. The carbonate contaminants would not be acceptable feedstock for commercial acidulation plants.

The investigators concluded that the evaluation confirmed the potential benefits of the anionic rougher-cleaner process.

If the obtained results are scaled up, an existing plant could convert to the anionic rougher-cleaner process and pay back the conversion costs from savings in operating costs realized during the first year.

They also recommended that additional work is required to prove the anionic rougher-cleaner process in continuous operation. Also, the potential benefits of the process must be weighed against any negative effects of lower grade reagentized concentrate on the acidulation process. The total impact of the anionic rougher-cleaner process on acid plant performance is not predictable from the concentrate chemical composition alone. Impacts such as defoamer consumption and filtration rate can only be established from pilot scale or full scale plant operation. FIPR's staff agree with these recommendations. However, such additional work will be more meaningful to individual companies if they perform it on their own ores to reach conclusions pertaining to their practical and economic situations.

Table of Contents

	<u>Page</u>
1.0 SUMMARY	
1.1 Background	1-1
1.2 Test Materials	1-2
1.3 Present Ore Evaluation	1-3
1.4 Future Ore Evaluation	1-4
1.5 Conclusions	1-5
1.6 Recommendations	1-6
2.0 PROCEDURES AND TEST MATERIALS	
2.1 Procedures	2-1
2.2 Ore Samples	2-1
2.2.1 Present Ore 'A'	2-2
2.2.2 Present Ore 'B'	2-2
2.2.3 Future Ore	2-4
2.2.4 Ore Consistency	2-4
2.3 Reagents	2-4
2.3.1 Collectors	2-4
2.3.2 Extenders	2-7
2.3.3 Modifiers	2-7
2.3.4 Frothers	2-8
2.3.5 Reagent Cost	2-8
2.3.6 Reagent Analysis	2-8
2.4 Water	2-9
3.0 INITIAL PROCESS EVALUATION	
3.1 Objective	3-1
3.2 Procedures	3-1
3.3 Reagent R1	3-1
3.3.1 Present Feed	3-2
3.3.2 Future Feed	3-2
3.4 Reagent R2	3-2
3.4.1 Present Feed	3-4
3.4.2 Future Feed	3-4

Table of Contents (cont'd.)

	<u>Page</u>	
3.5	Reagent R3	3-4
	3.5.1 Present Feed	3-7
	3.5.2 Future Feed	3-7
3.6	Reagent R4	3-7
	3.6.1 Present Feed	3-7
	3.6.2 Future Feed	3-9
3.7	Reagent R5	3-9
	3.7.1 Present Feed	3-9
	3.7.2 Future Feed	3-11
3.8	Reagent R6	3-11
	3.8.1 Present Feed	3-11
	3.8.2 Future Feed	3-11
3.9	Discussion of Results	3-13
	3.9.1 Recovery vs Cost	3-13
	3.9.2 Recovery vs Grade	3-17
	3.9.3 Selected Reagents	3-17
4.0	PROCESS DEFINITION	
4.1	Objective	4-1
4.2	Procedures	4-1
4.3	Reagent R1	4-3
	4.3.1 Present Feed	4-3
4.4	Reagent R2	4-6
	4.4.1 Present Feed	4-6
	4.4.2 Future Feed	4-6
4.5	Reagent R3	4-11
	4.5.1 Present Feed	4-11
	4.5.2 Future Feed	4-14
4.6	Reagent R4	4-14
	4.6.1 Future Feed	4-17
4.7	Discussion of Results	4-17

Table of Contents (cont'd.)

	<u>Page</u>
5.0 PROCESS OPTIMIZATION	
5.1 Objective	5-1
5.2 Test Program	5-1
5.2.1 Summary	5-1
5.2.2 Procedures	5-3
5.2.3 Results	5-3
5.3 Reagent R1	5-4
5.3.1 Present Feed	5-4
5.4 Reagent R2	5-7
5.4.1 Present Feed	5-7
5.4.2 Future Feed	5-12
5.5 Reagent R4	5-12
5.5.1 Future Feed	5-12
5.6 Reagent R2 - Crago Tests	5-22
5.6.1 Present Feed	5-22
5.6.2 Future Feed	5-25
5.7 Miscellaneous Tests	5-30
5.7.1 Water Recycle	5-30
5.7.2 Gangue Rejection Using Sodium Metasilicate	5-31
5.8 Locked Cycle Flotation Test	5-32
5.9 Flotation of Feed Fractions (Task 5A)	5-38
5.9.1 Unsized Feed	5-38
5.9.2 Coarse Feed	5-41
5.9.3 Fine Feed	5-48
5.10 Discussion of Results	5-48
6.0 ROUGHER-CLEANER PROCESS VS. CRAGO PROCESS	
6.1 Background	6-1
6.2 Concentrate Grade	6-1
6.3 Phosphate Recovery	6-4
6.4 Reagent Consumption	6-8
6.5 Other Factors	6-8

Table of Contents (cont'd.)

	<u>Page</u>
7.0 PLANT DESIGN AND OPERATING CONSIDERATIONS	
7.1 Introduction	7-1
7.1.1 Objective	7-1
7.1.2 Methodology	7-1
7.2 Summary	7-2
7.3 Plant Retrofit	7-3
7.3.1 Hypothetical Plant Configuration	7-3
7.3.2 Process Considerations	7-4
7.3.3 Modifications to Existing Plants	7-6
7.4 Grass-roots Plants	7-8
7.5 Operating Cost Impacts	7-12
7.6 Preliminary Risk/Benefit Analysis	7-14
8.0 RECOMMENDATIONS	
8.1 Potential of Process	8-1
8.2 Proposed Program	8-1
8.2.1 Beneficiation Testing	8-1
8.2.2 Acidulation Testing	8-2
8.2.3 Techno-Economic Analysis	8-2
8.3 Discussion	8-3

Index of Figures

<u>Figure</u>		<u>Page</u>
3.1	BPL Recovery vs Collector Cost - Rougher Flotation	3-16
3.2	BPL Recovery vs Collector Cost - Rougher Flotation	3-18
3.3	Recovery vs Grade - Rougher Flotation	3-19
5.1	Present Feed Locked Cycle Series 1	5-33
5.2	Present Feed Locked Cycle Series 2	5-37
7.1	Crago Reverse Flotation - Modified to Anionic Rougher-Cleaner	7-7
7.2	Crago Reverse Flotation	7-10
7.3	Rougher-Cleaner Flotation	7-11

Index of Tables

<u>Table</u>		<u>Page</u>
2-1	Composition of Ore Samples	2-3
2-2	Flotation Feed Chemical Analyses	2-5
2-3	Test Program - Chemical Reagents	2-9
2-4	Analysis of Laboratory Tap and Recycle Water	2-10
3-1	Reagent R1 - Process Evaluation Data	3-3
3-2	Reagent R2 - Process Evaluation Data	3-5
3-3	Reagent R3 - Process Evaluation Data	3-6
3-4	Reagent R4 - Process Evaluation Data	3-8
3-5	Reagent R5 - Process Evaluation Data	3-10
3-6	Reagent R6 - Process Evaluation Data	3-12
3-7	Best Performance Data - Present Feed	3-14
3-8	Best Performance Data - Future Feed	3-15
4-1	Test Variables for Feed/Reagent Combinations	4-2
4-2	Test Design - Collector R1 - Present Feed	4-4
4-3	Test Results Summary - Collector R1 - Present Feed	4-5
4-4	Test Design - Collector R2 - Present Feed	4-7
4-5	Test Results Summary - Collector R2 - Present Feed	4-8
4-6	Test Design - Collector R2 - Future Feed	4-9
4-7	Test Results Summary - Collector R2 - Future Feed	4-10
4-8	Test Design - Collector R3 - Present Feed	4-12
4-9	Test Results Summary - Collector R3 - Present Feed	4-13
4-10	Test Design - Collector R3 - Future Feed	4-15
4-11	Test Results Summary - Collector R3 - Future Feed	4-16
4-12	Test Design - Collector R4 - Future Feed	4-18
4-13	Test Results Summary - Collector R4 - Future Feed	4-19
4-14	Task 4 Bench Flotation Performance	4-20
5-1	Test Design - Collector R1 - Present Feed	5-5
5-2	Test Results Summary - Collector R1 - Present Feed	5-6

Index of Tables (cont'd.)

<u>Table</u>		<u>Page</u>
5-3	Test Design - Collector R1 - Present Feed (Optimization)	5-8
5-4	Test Design - Collector R2 - Present Feed	5-9
5-5	Test Results Summary - Collector R2 - Present Feed	5-10
5-6	Test Design - Collector R2 - Present Feed (Optimization)	5-11
5-7	Test Design - Collector R2 - Future Feed	5-13
5-8	Test Results Summary - Collector R2 - Future Feed	5-14
5-9	Test Design - Collector R2 - Future Feed (Optimization)	5-15
5-10	Test Design - Collector R4 - Future Feed	5-16
5-11	Test Results Summary - Collector R4 - Future Feed	5-18
5-12	Test Design - Collector R4 - Future Feed (Optimization)	5-19
5-13	Test Design - Collector R4 - Future Feed (Re-Run)	5-20
5-14	Test Design - Collector R4 - Future Feed (Optimization Re-Run)	5-21
5-15	Test Design - Collector R2 - Present Feed	5-23
5-16	Test Results Summary - Collector R2 - Present Feed	5-24
5-17	Test Design - Collector R2 - Present Feed (Crago Optimization)	5-26
5-18	Test Design - Collector R2 - Future Feed (Crago)	5-27
5-19	Test Results Summary - Collector R2 - Future Feed (Crago)	5-28
5-20	Test Design - Collector R2 - Future Feed (Crago Optimization)	5-29
5-21	Summary Data - Locked Cycle Test No. 1	5-34
5-22	Summary Data - Locked Cycle Test No. 2	5-35
5-23	Summary Data - Locked Cycle Test No. 2 (Crago)	5-36
5-24	Test Design - Collector R2 - 14 x 150 Feed (Anionic)	5-39
5-25	Test Results Summary - Anionic and Crago Tests - 14 x 150 Feed	5-40
5-26	Test Design - Collector R2 - 14 x 150 Feed (Anionic Optimization)	5-42
5-27	Test Design - Collector R2 - 14 x 150 Feed (Crago Optimization)	5-43
5-28	Test Design - Collector R2 - 14 x 35 Feed (Anionic)	5-44
5-29	Test Results Summary - Anionic and Crago Tests - 14 x 35 Feed	5-45
5-30	Test Design - Collector R2 - 14 x 35 Feed (Anionic Optimization)	5-46
5-31	Test Design - Collector R2 - 14 x 35 Feed (Crago Optimization)	5-47

Index of Tables (cont'd.)

<u>Table</u>		<u>Page</u>
5-32	Test Design - Collector R2 - 35 x 150 Feed (Anionic)	5-49
5-33	Test Results Summary - Anionic and Crago Tests - 35 x 150 Feed	5-50
5-34	Test Design - Collector R2 - 35 x 150 Feed (Anionic Optimzation)	5-51
5-35	Test Design - Collector R2 - 35 x 150 Feed (Crago Optimization)	5-52
5-36	Bench Scale Process Comparison - Present Feed	5-55
6-1	Concentrate Quality Comparison	6-3
6-2	Bench Scale Process Comparison (without recycle)	6-5
6-3	Bench Scale Process Comparison (with recycle)	6-7
6-4	Bench Scale Reagent Cost Comparison	6-8
7-1	Estimated Installed Capital Cost of Modifications	7-9
7-2	Operating Cost Comparison (annual)	7-13
7-3	Operating Cost Comparison (per ton)	7-14

SECTION 1

SUMMARY**1.1 Background**

Florida phosphate ore occurs in deposits of unconsolidated marine sediments. Prior to the development of a reliable flotation process, the ore was beneficiated by washing and classification to obtain pebble and debris fractions. The pebble product was marketable phosphate rock and the debris was wasted.

The advent of froth flotation significantly increased the phosphate reserves that could be economically exploited in the Florida land pebble district. Many flotation schemes were examined, but the Crago process, which was patented in 1942, became the standard method of extracting phosphate rock from the deslimed washer debris. The Crago process achieved its inventors objective to “provide an improved method of concentration which will be economical and practical and which will not only facilitate the production of concentrates of high grade, but will also result in the recovery of a high percentage of the phosphate values of the ore.” However, the Crago process evolved in an industry where the economics of easily recoverable inexpensive pebble dominated beneficiation.

As mining advances from high pebble deposits to high concentrate deposits, the cost to produce a ton of combined pebble and concentrate increases. Two phosphate producers in the southeastern U.S., who produce flotation concentrate only, have reverted to anionic rougher flotation to provide less expensive feedstock for their own phosphoric acid plants. Compared to the Crago process, anionic rougher flotation provides a lower grade concentrate at higher phosphate recovery and lower reagent cost.

Anionic rougher flotation does not give satisfactory results for lower grade flotation feeds; however anionic flotation may be adapted to a rougher-cleaner process with broader capabilities. As in the Crago process, the feed

is reagentized at controlled conditions with an anionic suite of flotation reagents. The first or "rougher" flotation step produces rougher phosphate concentrate, and a final tailing. The rougher concentrate is refloated in the second or "cleaner" flotation step to produce a cleaner concentrate. Tailings from cleaner flotation may be discarded or recycled as required to maintain grade and recovery.

The Board of Directors of the Florida Institute of Phosphate Research approved Zellars-Williams proposal to evaluate the preliminary feasibility of anionic rougher-cleaner flotation of present and future phosphate ore. FIPR contract 86-02-063R, for the bench scale testing and process evaluation, was approved May 1, 1987. The contract was amended September 30, 1987 to incorporate additional testing of sized feed and to allow a no-cost schedule extension and budget reorganization.

1.2 **Test Materials**

With the cooperation of local mining companies, three samples of phosphate ore were obtained, two of which were selected for use in the evaluation of anionic rougher-cleaner flotation. One sample (present ore 'B') was selected to represent presently mined ore. The second sample, obtained from lower zone material containing carbonate minerals, was chosen to represent future ore.

Six commercially available anionic collectors were selected for evaluation. Three of the anionic collectors were fatty acid type reagents obtained from Florida reagent vendors. The fourth anionic collector, a petroleum sulfonate, was obtained from a domestic vendor. The fifth and sixth anionic collectors, an organophosphoric acid and an N-substituted sarcosine, were obtained from European vendors.

1.3 Present Ore Evaluation

Thirty-six formal bench scale flotation tests were conducted to evaluate the six anionic collectors on 28 x 150 mesh flotation feed obtained from present ore. The three most promising reagents were examined in a second set of 36 tests to identify process variables that significantly influenced flotation performance. After the critical variables had been identified, a third series of tests was conducted to optimize the anionic rougher-cleaner process performance. A parallel series of tests were conducted to optimize the Crago process performance.

In accordance with the amended scope of work, additional testing was carried out to evaluate anionic rougher-cleaner flotation of 14 x 150 mesh, 14 x 35 mesh, and 35 x 150 mesh size fractions of flotation feed obtained from present ore. The program for these feed fractions included sets of tests to identify critical process variables and to optimize the performances of the anionic rougher-cleaner and Crago processes.

Relative to the Crago process, the anionic rougher-cleaner process gave lower grade concentrate, higher phosphate recovery, and lower reagent cost for 28 x 150 mesh feed. Chemical analyses indicate the anionic concentrate is a suitable feedstock for a commercial acidulation plant. The performance data from 28 x 150 mesh feed and cash operating cost data obtained with the ZW phosphate mining cost model are tabulated below:

<u>Concentrate Recovery</u>	<u>Anionic Process</u>	<u>Crago Process</u>	<u>Difference</u>
Flotation circuits	95.9%	93.9%	+2%
Deoiling & rinsing	100.0%	98.0%	+2%
Overall plant	95.9%	91.9%	+4%
<u>Concentrate Quality</u>			
% BPL	66.9	70.6	-3.7
% Acid insoluble	8.0	3.1	+4.9
<u>Cash Operating Costs</u>			
per ton concentrate	\$19.94	\$22.46	-\$2.52
per ton concentrate + pebble	\$ 9.33	\$ 9.95	-\$0.62

A field investigation of six phosphate plants followed by conceptual engineering indicated that these plants could be easily modified to utilize the anionic rougher-cleaner process. The estimated cost to modify a flotation plant having 1100 tph feed capacity is \$88K to \$123K. A grass-roots plant of the same capacity would cost about \$1.3 million less if the anionic rougher-cleaner process was utilized in place of the Crago process.

Test results from bench scale flotation of the 14 x 150 mesh, 14 x 35 mesh and 35 x 150 mesh, size fractions of present feed confirmed that the anionic rougher cleaner process gives lower concentrate grade and lower reagent cost than the Crago process. Reagent cost savings for these feeds ranged from 36 to 46 percent of the Crago reagent costs.

1.4 **Future Ore Evaluation**

The test program for 28 x 150 mesh flotation feed obtained from future ore was essentially identical to that described previously for present 28 x 150 mesh feed. Three sets of tests were conducted to evaluate the six anionic collectors, identify the critical process variables, and to optimize process performance.

Relative to the Crago process, the anionic rougher-cleaner process gave lower grade concentrate and lower reagent costs. Neither process was effective in rejecting carbonate minerals from the concentrate. The carbonate contaminated concentrates would not be acceptable feedstock for commercial acidulation plants.

1.5 Conclusions

The Phase I evaluation confirmed the potential benefits of the anionic rougher-cleaner process. The anticipated benefits of the process are compared below to the benefits determined in Phase I.

	<u>“Anticipated” Benefits</u>	<u>“Phase I” Potential</u>
BPL recovery improvement	4 to 8%	2 to 4%
Reagent cost reduction	20 to 40%	36 to 48%
Cash cost reduction/ton concentrate	\$1.0 to 1.5	\$2.5
Reduction in well water make-up	yes	yes
Reduction in energy	yes	yes
Existing plant retrofit	easy	easy
Grass-roots plant capital cost	reduced	reduced

If the Phase I test results scale up, an existing plant could convert to the anionic rougher-cleaner process and pay back the conversion costs from savings in operating costs realized during the first year.

Additional work is required to prove the anionic rougher-cleaner process in continuous operation. Also, the potential benefits of the process must be weighed against any negative effects of lower grade reagentized concentrate on the acidulation process. The total impact of the anionic rougher-cleaner process on acid plant performance is not predictable from the concentrate chemical composition alone. Impacts such as defoamer consumption and filtration rate can only be established from pilot scale or full scale plant operation.

Additional study is warranted by the potential benefits of the anionic rougher-cleaner process.

1.6 **Recommendations**

Zellers-Williams recommends a Phase II program to directly compare the anionic rougher-cleaner process to the Crago process by pilot scale flotation and subsequent pilot scale acidulation of their respective flotation concentrates. The recommended Phase II program includes:

- o pilot scale flotation of different phosphate plant feeds with the Crago process and the anionic rougher-cleaner process
- o pilot scale acidulation of anionic concentrate and of Crago concentrate from one of the test feeds
- o techno-economic analysis of the pilot plant test results and updating the Phase I cost estimates

The Phase II program will provide a comprehensive and more realistic basis to compare the two processes.

SECTION 2

PROCEDURES AND TEST MATERIALS

2.1 Procedures

In order to obtain reproducible results and minimize error, the testwork presented in this report was conducted utilizing analytical procedures approved by the Association of Florida Phosphate Chemists and the ZW test procedures listed below by procedure number.

- 001.0: Ore Blending and Sampling
- 002.2: Ore Washing
- 003.1: Anionic Rougher Flotation - Bench Testing
- 004.0: Bench Test Mass Balance Calculation
- 005.0: Feed Scrubbing
- 006.0: Anionic Cleaner Flotation - Bench Testing
- 007.1: Reagent Preparation and Use
- 008.0: Cationic Cleaner Flotation - Bench Testing
- 009.0: Anionic Rougher - Cleaner Locked Cycle Testing

A step-by-step description of each of the above procedures is presented in Appendix 'A'.

2.2 Ore Samples

The objective of the study was to examine anionic rougher-cleaner flotation of two ore samples representing present and future ore types.

Ore was specified as the feedstock for the test work. Time related surface changes of the feed particles were avoided by washing ore samples to provide fresh feed each week.

Three ore samples were collected from phosphate mines as identified below:

- o Present Ore A - Payne Creek
- o Present Ore B - Fort Meade
- o Future Ore - Four Corners

At each mine the personnel were exceedingly cooperative and helpful. A similar sampling procedure was followed at each site. A production dragline extracted the ore sample and placed it at the surface adjacent to the mining pit. The sample was loaded onto a clean dump truck by a front-end loader, then hauled to the ZW laboratory, where it was unloaded, blended and sampled in accordance with laboratory procedure 001.0.

2.2.1 Present Ore 'A'

Approximately 7,200 pounds of this material were collected. After blending, a representative sample of this ore was washed according to laboratory procedure 002.2. The flotation feed obtained from this ore had an unexpectedly low BPL content of 9.4 percent. Testing the low grade feed with the Crago flotation process utilizing laboratory procedures 003.1 and 006.0 indicated a reasonable flotation performance. The results from washing and flotation are presented on Table 2-1.

The BPL content of the 28 x 150 mesh feed obtained from present ore "A" was too low to be considered typical and it was decided to obtain a second sample of present ore.

2.2.2 Present Ore 'B'

Some 3,600 pounds of this material were collected and blended. The flotation feed obtained from a representative sample of this ore contained 14.1 percent BPL. The results from washing and flotation of present ore 'B' are given on Table 2-1.

Present ore "B" was selected as the ore sample representing present ore. Subsequently in this report the term "present ore" refers to present ore "B".

Table 2-1

Composition of Ore Samples

	<u>Present Ore</u>		<u>Future</u>
	<u>A</u>	<u>B</u>	<u>Ore</u>
+14 mesh % weight	15.6	25.6	26.9
14/28 mesh - % weight	6.5	12.9	4.0
28/150 mesh - % weight	59.3	36.3	41.6
-150 mesh - % weight	18.6	25.2	27.5
<u>+14 mesh (pebble)</u>			
% BPL	67.0	65.8	61.0
% AI*	9.2	7.5	9.2
% MgO	0.3	0.4	1.8
<u>28/150 mesh (concentrate)**</u>			
% BPL	72.9	72.0	65.5
% AI*	2.7	2.4	2.2
% MgO	0.3	0.4	1.5
<u>28/150 mesh (flotation feed)</u>			
% BPL	9.4	14.1	14.0
% AI*	87.1	79.6	72.3

* Acid insoluble material

**Crago Flotation Process

2.2.3 Future Ore

Future ore was obtained from lower zone material at an existing mine. Conventional beneficiation of some lower zone ores yields phosphate rock with an MgO content in excess of market specifications. Because this material is not presently mined, it is considered as a resource or future ore.

Approximately 5,600 pounds of future ore were obtained and blended. The data from washing and flotation of this material are presented on Table 2-1. The flotation feed contained 14.0 percent BPL and the pebble and concentrate products contained 1.8 and 1.5 percent MgO, respectively.

2.2.4 Ore Consistency

Ore samples were washed to provide fresh flotation feed on a weekly basis. Head analyses of the flotation feeds obtained from present and future ore are listed on Table 2-2. The uniformity of the analysis confirms that procedure 001.0 is a reliable method of blending and sampling Florida phosphate ore and that procedure 002.2 provides uniform samples of flotation feed.

2.3 **Reagents**

The chemical reagents used in this flotation test program are categorized as collectors, extenders, modifiers, and frothers.

2.3.1 Collectors

A multitude of experimental anionic collectors for phosphate rock flotation have been reported by investigators. However, for this program, six anionic collectors were selected for testing on the basis that they were commercially available and proven successful in plant and/or pilot plant flotation of various phosphate ores. A cationic collector, used in Florida phosphate beneficiation

Table 2-2
Flotation Feed Chemical Analyses

<u>Future Feed Sample</u>	<u>% BPL</u>	<u>% AI</u>	<u>% MgO</u>
87-07-09-03	14.04	72.57	1.36
87-08-18-26	14.08	73.80	1.15
87-08-19-01	13.86	73.67	1.09
87-08-24-02	14.08	73.83	1.10
87-09-21-01	14.41	73.49	1.26
87-11-04-29	13.92	73.91	1.26
87-11-09-02	14.28	72.55	1.26
87-11-16-02	14.07	72.88	1.30
87-11-30-02	14.47	72.94	1.23
87-12-07-01	13.66	73.80	1.24
87-12-14-01	<u>14.18</u>	<u>72.51</u>	<u>1.17</u>
mean	14.10	73.27	1.22
Std. Dev.	.24	.58	.08
<u>Present Feed Sample</u>			
87-07-09-07	14.06	79.58	
87-08-18-25	13.52	80.64	
87-08-19-02	13.67	80.35	
87-08-24-01	13.61	80.22	
87-09-15-01	14.06	79.15	
87-11-02-01	14.02	80.61	
87-11-09-01	14.37	78.74	
87-11-16-01	14.35	79.39	
87-11-30-01	14.20	79.84	
87-12-04-01	<u>14.31</u>	<u>79.80</u>	
mean	14.02	79.83	
Std. Dev.	.31	.63	

plants, was selected for use in testing the cleaner step of the Crago flotation process. A listing of collectors used in the test program is as follows:

- o Custafloat 27AR: blend of fatty acid soaps and sulfates
Westvaco
P.O. Box 237
Mulberry, FL 33860
- o Fatty Acid 5821-8M: tall oil fatty acid blend
Westvaco
P.O. Box 237
Mulberry, FL 33860
- o 21-7301: tall oil fatty acid blend
Nottingham Company
P. O. Box 250049 Station N
Atlanta, GA 30325-0049
- o Aero 801S Promotor: petroleum sulfonate
American Cyanamid Company
Water Treatment & Mining Chemicals
One Cyanamid Plaza
Wayne, NJ 07470
- o Melioran P301: organophosphoric acid
Gerlund Chemi Petrole
21, Square St. Charles
75583 Paris, France
CEDEX 12
- o MCS 87 K1: N substituted saracosine
Berol Kemi AB
Box 851
S-44401 Stenungsund
Sweden
- o Custamine 710: condensate amine
Westvaco
P.O. Box 237
Mulberry, FL 33860

2.3.2 Extenders

Non-polar hydrocarbons, used to extend the coverage of collectors, are referred to as extenders. Frothing is also influenced by these hydrocarbons. The three extenders included in the test program are:

- o No 2 Fuel Oil (diesel fuel)
- o No. 5 Fuel Oil
- o Kerosene

The two fuel oils were obtained from:
Fleetwing Corporation
742 S. Combee Road
Lakeland, FL 33803

Kerosene was obtained from:
Phillips 66 Service Station
Lakeland, Florida

2.3.3 Modifiers

Chemical reagents that modulate the action of collector reagents by changing the surface characteristics of minerals are called modifiers. A listing of the modifiers used in the test program follows:

- o NaOH: sodium hydroxide
Fisher Scientific
7464 Chancellor Dr
Orlando, FL
- o H₂SO₄: sulfuric acid
Fisher Scientific
7464 Chancellor Dr.
Orlando, FL
- o Metasilicate: sodium pentahydrate metasilicate
Preferred Products Ltd.
Winter Haven, FL

Note: The formula Na₂SiO₃ and the words "sodium silicate" are to be used to represent metasilicate in the remainder of the report.

2.3.4 Frothers

This group of reagents reduces the surface tension of water and promotes the formation of a stable froth. Several of the anionic collectors contain effective frothing agents. The one reagent used solely for frothing was:

Aerofroth 65: polypropylene glycol
American Cyanamid Company
Water Treatment & Mining Chemicals
One Cyanamid Plaza
Wayne, NJ 07470

2.3.5 Reagent Cost

Cost data for all reagents used in the test program are summarized on Table 2-3. The code by which the six anionic collectors were identified during the testwork is also shown on Table 2-3.

2.3.6 Reagent Analysis

The fatty acid-type collectors coded R1, R2, and R3 were analyzed and the results are reported below:

	<u>R1</u>	<u>R2</u>	<u>R3</u>
Saponification Value	n.a.	172	141
Acid Value	n.a.	165	134
Iodine Number	38	109	169
Water	64.8*	1.8	1.4
Unsaponifiabiles	5.4	9.9	19.6

*Saponified

2.4 Water

Lakeland tap water was used in all tests, except those noted for recycle water use. Laboratory tap and recycle water were analyzed and the results are reported in Table 2-4. Recycle water was taken from locked cycle test cell water (refer to Section 5.7.1). The analyses show elevated concentrations of P_2O_5 and F in the recycle water compared to laboratory tap water.

Table 2-3
Test Program - Chemical Reagents

<u>Code</u>	<u>Reagent</u>	<u>Type</u>	<u>Price \$/pound</u>
R1	Custafloat 27AR	anionic collector	0.120*
R2	Fatty Acid 5821-8M	" "	0.085*
R3	21-7301	" "	0.070*
R4	Aero 801S	" "	0.350*
R5	Melioran P301	" "	1.270*
R6	MCS 87-K1	" "	1.500*
	Custamine 710	cationic collector	0.275**
	No. 2 Fuel Oil	extender	0.074**
	No. 5 Fuel Oil	extender	0.056**
	Kerosene	extender	0.091**
	NaOH	modifier	0.105**
	H ₂ SO ₄	modifier	0.030**
	Metasilicate	modifier	0.177**
	Aerofroth 65	frother	0.915**

* unit prices in August 1987, subject to change.

**typical prices of commercial flotation reagents, assumed for study.

Table 2-4

Analysis of Laboratory Tap and Recycle Water

	Analysis (mg/liter)	
	<u>Tap Water</u>	<u>Recycle Water(1)</u>
P ₂ O ₅	0.3	2.6
F	2.6	9.1
SO ₄	5.1	4.2
Mg	8.9	8.9
Ca	27.0	25.0
CaCO ₃ hardness	104	99

(1) Recycle water taken from locked cycle test cell water (tests 5F84-5F93).

SECTION 3

INITIAL PROCESS EVALUATION

3.1 Objective

The purpose of the initial process evaluation was to select three anionic collectors showing the greatest potential for good flotation performance at low cost on present and future feed.

3.2 Procedures

Six anionic collectors were tested on flotation feed from samples of present and future ore to determine flotation response as a function of collector dosage. For this series of tests the parameters of conditioning and flotation were maintained within the range recommended by the reagent vendors for initial testing.

The specific ZW procedures used in the testwork are listed below and are presented in Appendix 'A'.

- 002.2: Ore Washing
- 003.1: Anionic Rougher Flotation - Bench Testing
- 004.0: Bench Test Mass Balance Calculations
- 007.1: Reagent Preparation and Use

Laboratory report sheets for each flotation test listed in Section 3 are presented in Volume II of this report.

3.3 Reagent R1

Anionic collector R1 is prepared from blended fatty acid soaps and sulfates, The initial testing conditions for reagentizing the flotation feeds with reagent R1 were as follows:

pulp density:	70% solids
pulp pH:	ambient
retention time:	3 minutes
ratio of collector to extender:	1:1 (No. 5 fuel oil)

The rougher flotation test results from present feed and future feed are summarized on Table 3-1.

3.3.1 Present Feed

Six rougher flotation tests were conducted to examine the flotation response at dosages of R1 ranging from 0.4 to 0.8 pounds of collector per ton of feed. The data indicate that collector R1, at dosages of 0.65 to 0.80 pounds per ton, recovered over 90 percent of the phosphate in the rougher concentrate. Concentrate grade analyzed greater than 64 percent BPL and less than 12 percent acid insoluble material.

3.3.2 Future Feed

Six rougher flotation tests were performed with future feed and dosages of R1 ranging from 0.3 to 0.8 pounds per ton of feed. More than 90 percent of the phosphate was recovered in the rougher concentrate at dosages of greater than 0.4 pounds R1 per ton of feed. The BPL content of the concentrates were less than 60 percent due to dilution from acid insoluble material and dolomitic material.

3.4 **Reagent R2**

Anionic collector R2 is a blend of tall oil fatty acids. The initial testing conditions for reagentizing flotation feed with collector R2 were as follows:

pulp density:	70% solids
pulp pH:	about 8.5
retention time:	3 minutes
ratio of collector to extender:	1:1 (No. 5 fuel oil)

Table 3-1

Reagent R1 - Process Evaluation Data

Test	Concentrate			% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾	
	% BPL ⁽¹⁾	% AI ⁽²⁾	% MgO	Wt. ⁽³⁾	BPL	AI	R1	FO ⁽⁶⁾
<u>Present Feed</u>								
3F2	57.3	15.0		0.1	0.6	0	0.40	0.40
3F3	64.6	11.9		21.3	94.6	3.2	0.80	0.80
3F4	65.9	9.6		20.0	90.8	2.4	0.70	0.70
3F5	66.0	9.6		17.9	81.8	2.2	0.60	0.60
3F6	64.9	6.8		11.9	57.0	1.0	0.50	0.50
3F7	64.8	11.0		20.8	94.4	2.9	0.65	0.65
<u>Future Feed</u>								
3F8	50.5	10.5	4.1	28.1	96.5	4.0	0.81	0.81
3F9	52.0	8.7	4.0	27.2	97.5	3.2	0.61	0.61
3F10	55.9	5.0	3.6	24.4	94.3	1.7	0.40	0.40
3F12	56.6	3.7	4.0	12.5	49.0	0.6	0.30	0.30
3F13	57.1	4.6	3.8	20.4	80.4	1.3	0.35	0.35
3F14	52.6	8.8	3.9	26.2	95.1	3.1	0.50	0.50

(1) Bone Phosphate of Lime $\text{Ca}_3(\text{PO}_4)_2$

(2) Acid Insoluble material

(3) $100 \text{ (Concentrate weight) / (Feed weight)}$

(4) $100 \text{ (Concentrate Units) / (Feed Units)}$

(5) Pounds reagent per short ton of feed

(6) No. 5 fuel oil

The rougher flotation test results for present and future feed are shown on Table 3-2.

3.4.1 Present Feed

The rougher flotation response of present feed was tested at six dosages of R2 ranging from 0.4 to 0.8 pounds per ton of feed. At dosages above 0.5 pounds per ton, BPL recovery above 90 percent and concentrate grades of 63 to 67 percent BPL were obtained.

3.4.2 Future Feed

Six tests were conducted using future feed to examine the rougher flotation response at dosages of 0.2 to 0.6 pounds of R2 per ton of feed. BPL recoveries of greater than 90 percent were obtained at R2 dosages above 0.3 pounds per ton; however, BPL analyses of the corresponding concentrates remained below 60 percent because of the presence of insoluble and carbonate materials.

3.5 **Reagent R3**

Anionic collector R3 is a tall oil fatty acid blend. The initial testing conditions for reagentizing the flotation feed with reagent R3 were as follows:

pulp density:	70% solids
pulp pH:	about 8.5
retention time:	3 minutes
ratio of collector to extender:	1:1 (No. 5 fuel oil)

Test results for rougher flotation of present and future feed are given on Table 3-3.

Table 3-2
Reagent R2 - Process Evaluation Data

<u>Test</u>	<u>Concentrate</u>			<u>% Recovery⁽⁴⁾</u>			<u>Reagent, lb/T⁽⁵⁾</u>		
	<u>% BPL⁽¹⁾</u>	<u>% AI⁽²⁾</u>	<u>% MgO</u>	<u>Wt.⁽³⁾</u>	<u>BPL</u>	<u>AI</u>	<u>R2</u>	<u>FO⁽⁶⁾</u>	<u>NaOH</u>
<u>Present Feed</u>									
3F16	63.1	13.0		21.8	96.6	3.6	0.80	0.80	0.15
3F17	65.8	9.6		20.9	96.8	2.5	0.59	0.59	0.15
3F18	67.1	8.4		19.2	90.6	2.0	0.56	0.56	0.15
3F19	67.4	8.1		19.1	91.2	1.9	0.50	0.50	0.14
3F20	71.1	4.1		8.4	44.2	0.4	0.40	0.40	0.13
3F21	68.6	7.3		17.4	84.5	1.6	0.45	0.45	0.13
<u>Future Feed</u>									
3F22	51.4	11.5	3.8	27.8	96.6	4.4	0.60	0.60	0.14
3F23	53.1	6.9	4.0	24.8	92.2	2.3	0.40	0.40	0.12
3F24	54.7	5.7	3.7	23.7	92.0	1.9	0.30	0.30	0.12
3F25	61.2	2.5	2.8	7.0	30.9	0.2	0.20	0.20	0.11
3F26	51.0	9.5	4.1	26.8	95.5	3.5	0.51	0.51	0.12
3F27	53.3	6.4	4.2	24.8	92.2	2.2	0.46	0.46	0.12

- (1) Bone Phosphate of Lime $\text{Ca}_3(\text{PO}_4)_2$
- (2) Acid Insoluble Material
- (3) 100 (Concentrate weight)/(Feed weight)
- (4) 100 (Concentrate units)/(Feed units)
- (5) Pounds reagent per short ton of feed
- (6) No. 5 fuel oil

Table 3-3

Reagent R3 - Process Evaluation Data

Test	Concentrate			% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾		
	% BPL ⁽¹⁾	% AI ⁽²⁾	% MgO	Wt. ⁽³⁾	BPL	AI	R3	FO ⁽⁶⁾	NaOH
<u>Present Feed</u>									
3F28	63.9	11.2		21.2	95.1	3.0	0.61	0.61	0.13
3F29	69.9	4.4		12.5	63.4	0.7	0.40	0.39	0.12
3F30	65.8	8.8		20.1	92.7	2.2	0.50	0.50	0.12
3F31	65.5	7.4		18.3	87.3	1.7	0.45	0.46	0.12
3F32	67.9	5.2		8.9	45.8	0.6	0.35	0.35	0.12
3F33	61.1	13.3		22.0	97.0	3.8	0.72	0.72	0.12
<u>Future Feed</u>									
3F34	44.3	22.0	3.0	32.7	97.9	10.1	0.62	0.62	0.12
3F35	45.6	20.0	3.0	31.1	96.9	8.7	0.51	0.51	0.12
3F36	46.1	20.3	2.9	30.4	96.8	8.6	0.40	0.40	0.12
3F37	53.0	10.3	2.9	24.9	90.0	3.5	0.31	0.31	0.11
3F38	58.9	4.0	2.7	10.6	43.5	0.6	0.21	0.20	0.11
3F39	40.1	28.6	2.8	35.1	97.9	13.9	0.71	0.71	0.13

(1) Bone Phosphate of Lime $\text{Ca}_3(\text{PO}_4)_2$

(2) Acid Insoluble material

(3) $100 (\text{Concentrate weight})/(\text{Feed weight})$

(4) $100 (\text{Concentrate Units})/(\text{Feed Units})$

(5) Pounds reagent per short ton of feed

(6) No. 5 fuel oil

3.5.1 Present Feed

Dosages of R3 from 0.3 to 0.7 pounds per ton of feed were tested on present feed to determine the rougher flotation response. The data show collector R3, at dosages above 0.5 pounds per ton, produced concentrates containing more than 61 percent BPL while maintaining over 90 percent BPL recovery.

3.5.2 Future Feed

Six rougher flotation tests were performed with future feed and dosages of R3 of 0.2 to 0.7 pounds per ton feed. Above 0.3 pounds R3 per ton, BPL recoveries of 90 percent were obtained; however concentrate grades were below 53 percent BPL due to carbonate gangue and excessive acid insoluble material.

3.6 **Reagent R4**

Anionic reagent R4 is a petroleum sulfonate. The initial testing conditions for reagentizing the flotation feed with reagent R4 were as follows:

pulp density:	70% solids
pulp pH:	9
retention time:	3 minutes
ratio of collector to extender:	4.5:1 (No. 5 fuel oil)

The rougher flotation test results for present and future feed are summarized on Table 3-4.

3.6.1 Present Feed

The rougher flotation response of present feed at six dosages of R4 was examined. BPL recoveries less than 90 percent were obtained using present feed, even at R4 dosages of two to four pounds per ton of feed. Concentrate

Table 3-4

Reagent R4 - Process Evaluation Data

Test	Concentrate			% Recovery ⁽⁴⁾				Reagent, lb/T ⁽⁵⁾	
	% BPL ⁽¹⁾	% AI ⁽²⁾	% MgO	Wt. ⁽³⁾	BPL	AI	MgO	R4	FO ⁽⁶⁾
<u>Present Feed</u>									
3F82	63.5	12.4		6.9	31.9	1.1		1.53	0.34
3F83	64.1	11.9		10.7	49.1	1.6		2.04	0.45
3F84	63.8	12.1		16.7	76.7	2.5		3.05	0.67
3F85	63.6	12.6		17.3	78.8	2.8		4.08	0.90
3F86	66.9	7.7		5.3	26.9	0.5		1.02	0.23
3F87	64.1	11.9		14.3	65.8	2.1		2.55	0.56
<u>Future Feed</u>									
3F88	58.2	10.0	2.1	16.7	68.1	2.3	31.8	1.01	0.22
3F89	45.6	24.6	2.4	30.2	96.6	10.2	63.2	2.06	0.45
3F90	52.6	15.1	2.3	25.3	90.3	5.2	54.5	1.54	0.34
3F91	62.6	5.1	1.9	7.2	31.6	0.5	13.5	0.51	0.11
3F92	59.7	7.3	2.1	16.4	67.5	1.6	33.2	0.83	0.18
3F93	33.8	45.5	1.6	35.8	84.7	22.2	53.5	1.24	0.27

(1) Bone Phosphate of Lime $\text{Ca}_3(\text{PO}_4)_2$

(2) Acid Insoluble material

(3) $100 (\text{Concentrate weight}) / (\text{Feed weight})$

(4) $100 (\text{Concentrate units}) / (\text{Feed units})$

(5) Pounds reagent per short ton of feed

(6) No. 5 fuel oil

grades were fairly consistent at about 64 to 67 percent BPL for all dosages of R4. For the test conditions, R4 did not exhibit sufficient collecting power for the phosphate.

3.6.2 Future Feed

Dosages of R4 from 0.5 to 2.0 pounds per ton of future feed were tested. The collecting power of R4 was improved with future feed, and BPL recoveries of 90 percent were obtained at dosages of 1.5 to 2.0 pounds per ton, with corresponding concentrate grades of 45 to 53 percent BPL. Reagent R4 showed some promise for dolomite rejection as MgO recoveries lagged BPL recoveries by about 30 percent.

3.7 **Reagent R5**

Anionic collector R5 is an organophosphoric acid. This type of reagent has been proven effective in bench and pilot flotation of some phosphorites from North Africa. The initial testing conditions for reagentizing the flotation feed with reagent R5 were as follows:

pulp density:	30% solids
pulp pH:	ambient
retention time:	15 seconds
water temperature:	140°F

A summary of rougher flotation test results from present and future feed are given on Table 3-5.

3.7.1 Present Feed

Reagent R5 exhibited neither selectivity or pulling power for present feed. Concentrate grades remained low at 16 to 21 percent BPL, and recovery remained below 75 percent, even at a dosage of 3.33 pounds R5 per ton of feed.

Table 3-5
Reagent R5 - Process Evaluation Data

Test	Concentrate			Wt. ⁽³⁾	% Recovery ⁽⁴⁾		Reagent, lb/T ⁽⁵⁾ R5
	% BPL ⁽¹⁾	% AI ⁽²⁾	% MgO		BPL	AI	
<u>Present Feed</u>							
3F 52	20.1	71.7		26.8	40.2	23.8	0.41
3F 53	21.2	70.2		35.9	56.8	31.2	0.83
3F 54	20.1	71.7		44.5	67.9	39.5	1.66
3F 55	18.2	74.3		58.3	73.2	54.7	3.33
3F 56	16.6	76.7		51.9	65.0	49.2	2.46
3F 57	20.2	71.1		9.4	13.7	8.4	0.21
<u>Future Feed</u>							
3F 58	27.1	55.2	1.4	29.3	56.6	21.9	0.41
3F 59	26.4	57.2	1.2	41.6	80.4	31.7	0.81
3F 60	24.6	59.6	1.2	49.9	88.5	40.0	1.67
3F 61	20.0	66.7	1.0	58.7	86.7	52.2	2.49
3F 62	25.3	53.2	2.0	11.9	21.4	8.5	0.21
3F 63	17.9	70.1	0.9	63.5	82.7	59.6	3.31

- (1) Bone Phosphate of Lime $\text{Ca}_3(\text{PO}_4)_2$
- (2) Acid Insoluble material
- (3) $100 \text{ (Concentrate weight) / (Feed weight)}$
- (4) $100 \text{ (Concentrate units) / (Feed units)}$
- (5) Pounds reagent per short ton of feed

3.7.2 Future Feed

Reagent R5 was more effective with future feed than present feed; however concentrate grades remained unacceptably low at 18 to 27 percent BPL. Concentrate BPL recoveries of 80 to 88 percent were obtained at R5 dosages of 0.8 to 3.3 pounds per ton of feed.

3.8 **Reagent R6**

Collector R6 is an N-substituted sarcosine. This amphoteric reagent is normally anionic in the alkaline pH range. The initial testing conditions for reagentizing the flotation feeds with reagent R6 were as follows:

pulp density:	30% solids
pulp pH:	10
retention time:	15 seconds*
ratio of collector to extender:	2:1 (No. 2 fuel oil)

*Informal testing at conditioning times of 180 and 15 seconds indicated that the lower time was best.

Data from the rougher flotation tests of present and future feed are summarized on Table 3-6.

3.8.1 Present Feed

At the recommended test conditions R6 acted as a cationic reagent for present feed and floated acid insoluble material from the phosphate. Dosages of 0.6 to 2.0 pounds per ton of feed of R6 did not yield satisfactory performance, as concentrate grades ranged from 14 to 25 percent BPL.

3.8.2 Future Feed

The results obtained from future feed were similar to those obtained from present feed with reagent R6. The performance was not satisfactory, as concentrate grades ranged from 15 to 20 percent BPL.

Table 3-6

Reagent R6 - Process Evaluation Data

Test	Concentrate			% Recovery ⁽⁴⁾				Reagent, lb/T ⁽⁵⁾		
	% BPL ⁽¹⁾	% AI ⁽²⁾	% MgO	Wt. ⁽³⁾	BPL	AI	MgO	R6	FO ⁽⁶⁾	NaOH
<u>Present Feed</u>										
3F68	15.3	77.7		61.8	69.1	59.8		1.03	0.52	0.99
3F69	22.8	64.2		4.1	6.8	3.3		2.04	1.02	0.94
3F70	25.3	60.7		6.7	12.2	5.0		1.53	0.77	0.94
3F71	15.1	78.0		74.9	82.9	73.0		0.82	0.41	1.02
3F72	14.7	78.8		83.6	88.1	82.5		0.71	0.35	1.01
3F73	14.1	79.7		90.3	92.1	89.8		0.60	0.30	1.00
<u>Future Feed</u>										
3F74	18.7	54.4	3.55	22.6	29.1	16.9	68.0	1.03	0.51	0.97
3F75	19.4	62.5	1.85	47.7	63.7	40.5	82.0	0.82	0.41	0.97
3F76	18.2	66.3	1.54	71.9	91.5	64.7	93.4	0.62	0.31	0.98
3F77	14.7	73.5	1.06	95.5	99.5	94.2	99.1	0.42	0.21	0.96
3F78	20.4	62.3	1.69	59.8	84.9	50.6	90.0	0.73	0.36	0.96
3F79	15.9	70.4	1.39	82.1	93.1	78.2	95.7	0.51	0.26	0.94

(1) Bone Phosphate of Lime $\text{Ca}_3(\text{PO}_4)_2$

(2) Acid Insoluble material

(3) $100 \text{ (Concentrate weight) / (Feed weight)}$

(4) $100 \text{ (Concentrate Units) / (Feed Units)}$

(5) Pounds reagent per short ton of feed

(6) No. 2 fuel oil

3.9 Discussion of Results

Task 3 comprised 72 formal flotation tests of six anionic collectors and two feed samples. The test results were evaluated to select the three most promising reagents for each feed. Major criteria for evaluating the reagents were phosphate (BPL) recovery, reagent cost, and concentrate grade.

The best performance data from each of the reagents with present feed are summarized on Table 3-7. Reagents R1, R2 and R3 gave superior performance and costs for present feed. For example, these reagents had unit costs not exceeding \$0.6 per concentrate ton and yielded BPL recoveries and concentrate BPL analyses of greater than 94 percent and 63 percent, respectively.

The best performance data from future feed with the same six anionic collectors is shown on Table 3-8. Reagents R1, R2, and R3 also gave superior performance and costs for future feed. Reagent R4 gave equivalent BPL recovery to reagents R1, R2, and R3 but at a substantially greater unit cost. Also, R4 showed some promise for dolomite (MgO) rejection. Reagent R4 recovered only 63 percent of the MgO while reagents R1, R2, and R3 recovered about 87 to 95 percent of the MgO.

3.9.1 Recovery vs Cost

Anionic collectors R1, R2, and R3 are fatty acid-type reagents currently available in Florida. A plot of flotation BPL recoveries versus collector costs is given on Figure 3.1 for the Florida-type reagents. Figure 3.1 shows that the present feed sample required a higher reagent cost than the future feed sample to achieve the same level of recovery. The data also show that in order of increasing cost per ton of feed, the reagents rank R3, R2, and R1 for both present and future feeds. Reagent R1 is saponified and its cost includes pH modifier while R2 and R3 costs exclude pH modifier. Adjusting R1 costs to exclude pH modifier would result in a credit (cost reduction) but would not change the ranking by cost.

Table 3-7

Task 3 - Best Performance Data - Present Feed

<u>Anionic Collector</u>	<u>R2</u>	<u>R3</u>	<u>R1</u>	<u>R4</u>	<u>R6</u>	<u>R5</u>
Test Number	3F17	3F28	3F7	3F85	3F71	3F55
Concentrate % Recovery:						
BPL	96.82	95.08	94.39	78.79	82.92	73.23
A.I.	2.52	3.04	2.85	2.75	72.96	54.73
Concentrate Grade:						
% BPL	65.76	63.89	64.78	63.59	15.11	18.16
% A.I.	9.59	11.17	10.99	12.58	78.02	74.34
Collector Dosage (lbs/T feed)	0.59	0.61	0.65	4.08	0.82	3.33
Reagent Cost (\$/T concentrate)	0.49	0.46	0.60	8.74	1.83	7.25

Table 3-8

Task 3 - Best Performance Data - Future Feed

<u>Anionic Collector</u>	<u>R2</u>	<u>R3</u>	<u>R1</u>	<u>R4</u>	<u>R6</u>	<u>R5</u>
Test Number	3F26	3F36	3F14	3F89	3F78	3F60
Concentrate % Recovery:						
BPL	95.46	96.78	95.11	96.57	84.89	88.54
A.I.	3.51	8.58	3.13	10.15	50.58	40.00
MgO	(95)*	(87)*	(89)*	63.24	89.99	55.09
Concentrate Grade:						
% BPL	50.98	46.11	52.64	45.61	20.38	24.63
% A.I.	9.50	20.27	8.76	24.64	62.25	59.61
MgO	4.10	2.90	3.90	2.35	1.69	1.23
Collector Dosage (lbs/T feed)	0.51	0.40	0.50	2.06	0.73	1.67
Reagent Cost (\$/T concentrate)	0.32	0.22	0.37	2.59	2.04	4.25

*Concentrate MgO units/feed MgO units

% BPL RECOVERY vs COLLECTOR COST

Rougher Flotation

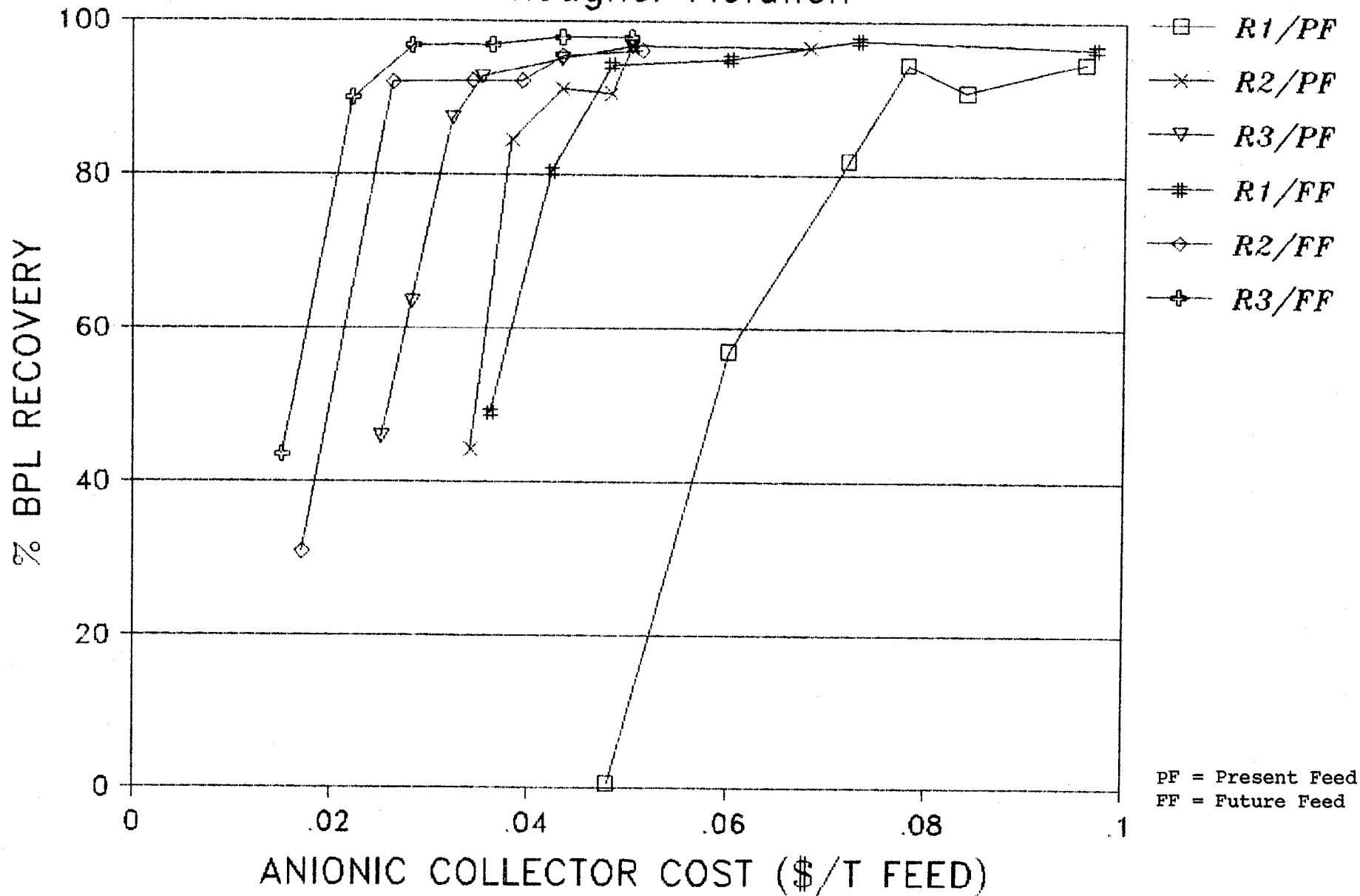


Figure 3.1

Anionic collectors R4, R5, and R6 have been used for flotation of phosphate from other locations. A plot of flotation BPL recoveries versus the costs of the collectors is presented on Figure 3.2. In most instances, the cost of these reagents exceeds \$1.00 per feed ton, while the Florida-type reagents cost less than \$0.10 per feed ton.

Based on cost and BPL recovery, it is clear the reagents R1, R2 and R3 are superior for both feeds.

3.9.2 Recovery versus Grade

Compared to the Crago flotation process anionic rougher-cleaner flotation is expected to produce lower grade concentrate. To minimize the reduction in concentrate grade, collectors that exhibit collecting power and selectivity are essential for the anionic rougher-cleaner process. A plot of phosphate BPL recoveries versus concentrate percent BPL for present and future feed with Reagents R1, R2, R3 and R4 are shown on Figure 3.3. The more selective reagents give high grade and high recovery. In order of decreasing selectivity, the collectors are ranked R2, R1, R3, and R4 for present feed and R1, R2, R4, and R3 for future feed. None of the reagents gave acceptable concentrates for future feed.

2.9.3 Selected Reagents

Anionic collectors R1, R2, and R3 were selected for the subsequent anionic rougher-cleaner flotation testing of present feed. For future feed, Reagents R2, R3, and R4 were selected. The choice of R4 instead of R1 was made so that the MgO rejection potential of R4 could be further studied.

% BPL RECOVERY vs COLLECTOR COST

Rougher Flotation

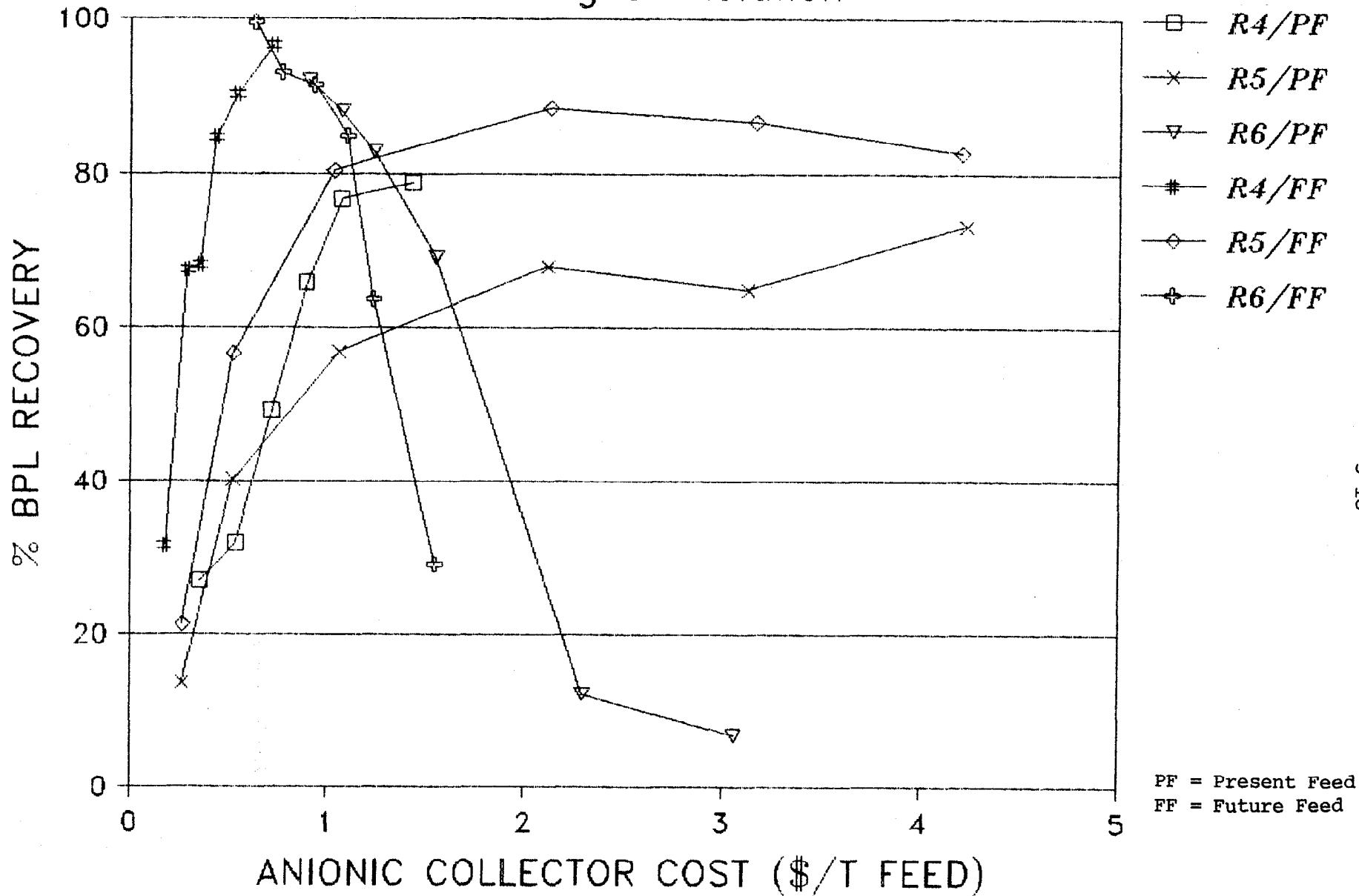
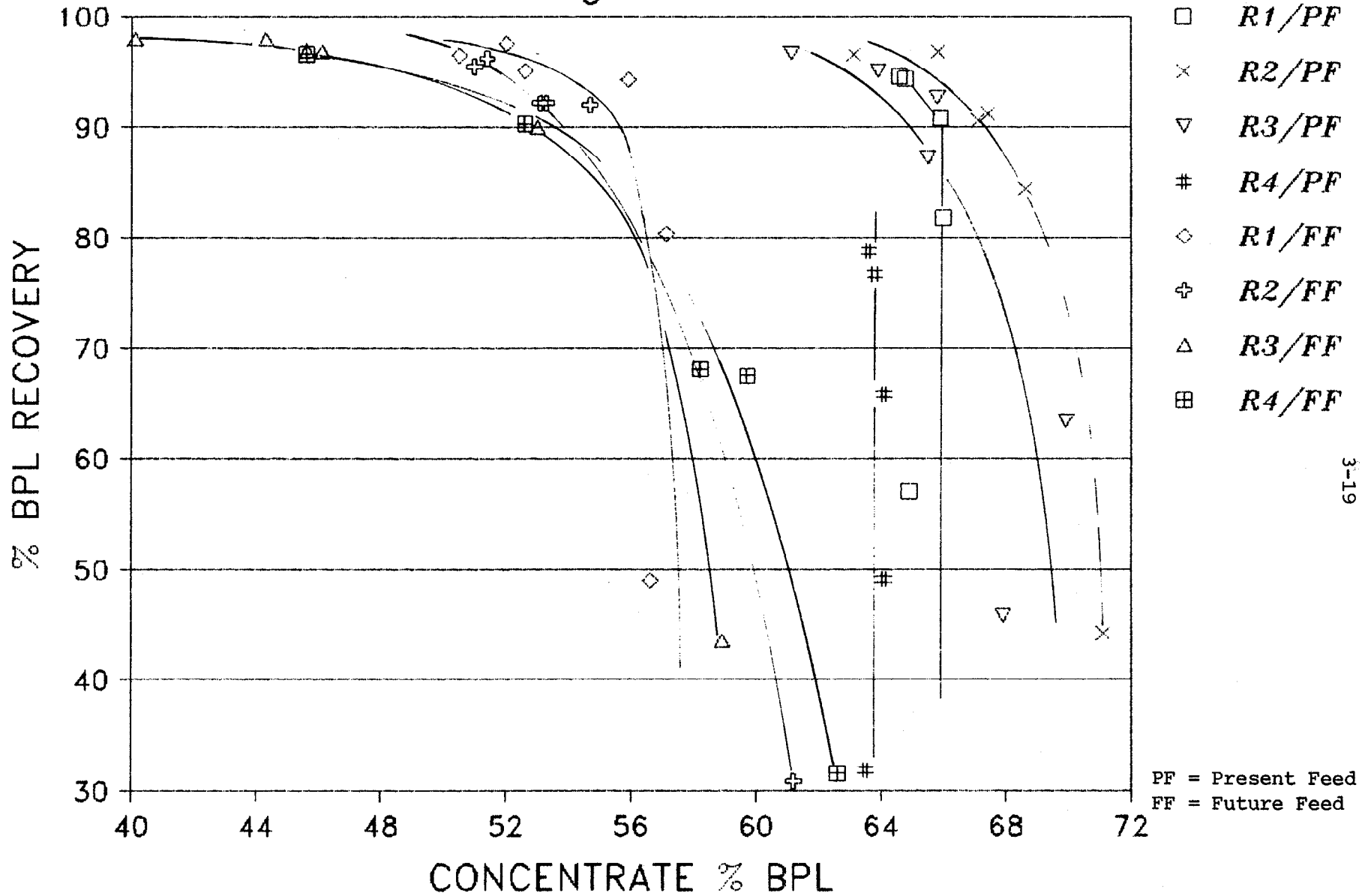


Figure 3.2

RECOVERY vs GRADE

Rougher Flotation



3-19

Figure 3.3

SECTION 4

PROCESS DEFINITION

4.1 Objective

The objective of Task 4 was to develop the rougher-cleaner process by examining the effects of different process variables in tests of present and future feed using the three anionic collectors selected in Task 3 and to select two anionic collectors for process optimization studies in Task 5. The significance of each test variable was established by statistical evaluation of its influence on BPL recovery and concentrate percent acid insoluble. Concentrate percent MgO was included in the evaluation of future feed.

4.2 Procedures

The three reagents tested with present feed in Task 4 were R1, R2 and R3. With future feed, the three reagents tested were R2, R3 and R4. Twelve formal flotation tests were performed on each feed/reagent combination for a total of 72 formal tests.

A Plackett-Burman experimental design comprising 12 tests to examine eight process variables and three dummy variables was used in Task 4. The variables examined for each feed/reagent combination are listed in Table 4-1. Table 4-1 shows that the process variables were inserted into the design as factors 1, 2, 3, 4, 6, 8, 9 and 11 whereas the dummy variables were inserted into the test design as factors 5, 7, and 10.

Specific procedures followed in the execution of Task 4 are listed on page 4-3 and are presented in Appendix 'A' of this report.

Table 4-1

Test Variables for Feed/Reagent Combinations

Variable	Present Feed Reagents			Future Feed Reagents		
	R1	R2	R3	R2	R3	R4
1	Tc	Tc	Tc	Tc	Tc	Tc
2	Sc	Sc	Sc	Sc	Sc	Sc
3	C	C	C	C	C	C
4	Sf	Sf	Sf	Sf	Sf	Sf
5	d	d	d	d	d	d
6	Dc	Dc	Dc	Dc	Dc	Dc
7	d	d	d	d	d	d
8	R	R	R	R	R	O
9	A	P	P	P	P	P
10	d	d	d	d	d	d
11	Dd	Dd	Dd	Dd	Dd	A

Tc = conditioning time

Sc = conditioning % solids

C = cleaning stages (1 vs 2)

Sf = flotation % solids*

d = dummy variables

Dc = collector dosage

R = ratio of oil to collector

Dd = depressant dosage

A = attrition scrub (with or without)

P = conditioner pH

O = fuel oil type (No. 2 or No. 5)

* Flotation % solids or % water is varied by changing cell size. A 3 liter cell gives a nominal 32% solids or 68% water, while a 5 liter cell gives a nominal 20% solids or 80% water.

<u>Procedure</u>	<u>Title</u>
002.2	Ore Washing
003.1	Anionic Rougher Flotation-Bench Testing
004.0	Bench Test Mass Balance Calculation
005.0	Feed Scrubbing
006.0	Anionic Cleaner Flotation-Bench Testing
007.1	Reagent Preparation and Use

4.3 Reagent R1

Anionic collector R1 is a blend of fatty acid soaps and sulfates. Refer to Table 2-3 for specific data for collector R1.

4.3.1 Present Feed

The Plackett-Burman experimental test design for collector R1 on present feed is presented in Table 4-2 and the statistical design analysis is presented in Appendix 'B'. Results from the 12 tests are summarized in Table 4-3.

The data indicate that feed scrubbing, increasing the collector dosage, decreasing the cell pulp density and increasing the conditioner % solids are significant in improving the BPL recovery. The addition of sodium silicate is shown to decrease the BPL recovery. The significant variables and their effects on BPL recovery and concentrate percent AI are shown below:

<u>Variable</u>	<u>Change</u>	<u>Effect</u>
Feed scrubbing	from without to with	increased BPL recovery by 14.7%. increased AI by 1.3%
Collector dosage	from 0.51 to 0.71 lb/T	increased BPL recovery by 9.4%
Flotation cell water content	from 68% to 80% (3 liter cell to 5 liter cell)	increased BPL recovery by 9.3%
Conditioner % solids	from 65 to 70	increased BPL recovery by 5.8%
Na ₂ SiO ₃ addition	from 0 to 1 lb/T	reduced BPL recovery by 7.9%
Cleaning stages	from 1 to 2 stages	reduced AI by 1.3%

Table 4-2

**Test Design
Collector R1 - Present Feed
(Task 4)**

<u>Test</u>		<u>Conditioning</u>		<u>Cleaning</u>	<u>Size Cell</u>	<u>Collector</u>	<u>Collector to</u>	<u>Feed</u>	<u>Na₂SiO₃⁽³⁾</u>
<u>No.</u>	<u>Sequence</u>	<u>Time(min)</u>	<u>% Solids</u>	<u>Stages</u>	<u>(liters)</u>	<u>(lb/T)</u>	<u>FO⁽¹⁾ Ratio</u>	<u>Scrub⁽²⁾</u>	<u>(lb/T)</u>
4F6	6	3.5	70	1	5	0.71	1:1.0	None	None
4F11	11	2.5	70	2	3	0.71	1:1.0	None	1.0
4F7	7	3.5	65	2	5	0.71	1:1.5	None	None
4F3	3	2.5	70	1	5	0.59	1:1.5	Yes	None
4F8	8	2.5	65	2	3	0.71	1:1.5	Yes	None
4F9	9	2.5	65	1	5	0.71	1:1.0	Yes	1.0
4F12	12	3.5	65	1	3	0.59	1:1.5	None	1.0
4F10	10	3.5	70	1	3	0.71	1:1.5	Yes	1.0
4F2	2	3.5	70	2	3	0.59	1:1.0	Yes	None
4F5	5	2.5	70	2	5	0.59	1:1.5	None	1.0
4F4	4	3.5	65	2	5	0.59	1:1.0	Yes	1.0
4F1	1	2.5	65	1	3	0.59	1:1.0	None	None
4F37 ⁽⁴⁾	-	2.5	70	2	5	0.71	1:10	None	None
4F38 ⁽⁴⁾	-	2.5	70	1	5	0.71	1:1.0	None	None

(1) No. 5 fuel oil

(2) Feed scrub at 65% solids for 2 minutes, followed by jet rinse - per procedure number 005.0

(3) When used, Na₂SiO₃ is to be added to conditioner.

(4) Supplemental Tests

No adjustment of conditioner pH for reagent R1.

Table 4-3

Test Results Summary
Collector R1 - Present Feed
(Task 4)

Test	Concentrate		% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾		
	%BPL ⁽¹⁾	%AI ⁽²⁾	Wt. ⁽³⁾	BPL	AI	R1	FO ⁽⁶⁾	Na ₂ SiO ₃
4F1	69.13	7.17	14.40	69.47	1.29	0.61	0.61	.00
4F2	69.41	6.39	19.42	94.15	1.55	0.59	0.59	.00
4F3	68.19	7.56	20.17	96.42	1.90	0.60	0.90	.00
4F4	69.70	5.71	19.05	93.40	1.35	0.60	0.60	1.01
4F5	70.71	4.21	15.51	79.58	0.81	0.57	0.86	0.97
4F6	67.36	8.26	20.33	96.03	2.09	0.71	0.71	.00
4F7	69.31	5.27	19.34	93.95	1.27	0.71	1.07	.00
4F8	68.38	6.96	20.12	96.09	1.75	0.72	1.08	.00
4F9	68.19	7.12	18.93	91.13	1.68	0.71	0.71	1.00
4F10	66.61	8.81	20.39	95.25	2.24	0.72	1.08	1.01
4F11	69.69	5.09	15.73	78.23	1.00	0.72	0.72	1.01
4F12	70.34	4.72	12.02	61.02	0.71	0.60	0.90	1.02
4F37	68.02	6.90	19.92	94.95	1.73	0.72	0.72	.00
4F38	67.46	7.00	19.91	95.28	1.75	0.72	0.72	.00

- (1) Bone Phosphate of Lime Ca₃(PO₄)₂
- (2) Acid Insoluble Material
- (3) 100 (Concentrate weight)/(Feed weight)
- (4) 100 (Concentrate units)/(Feed units)
- (5) Pounds Reagent Per Short Ton of Feed
- (6) No. 5 fuel oil

Laboratory report sheets for collector R1 on present feed are presented in Volume II of this report.

4.4 **Reagent R2**

Anionic collector R2 is a blend of tall oil fatty acids. Refer to Table 2-3 for specific data for collector R2.

4.4.1 Present Feed

The Plackett-Burman experimental test design for collector R2 on present feed is presented in Table 4-4 and the statistical design analysis is presented in Appendix 'B'. Results from the 12 tests are summarized in Table 4-5.

The data indicate that increasing the collector dosage is significant in improving the BPL recovery. Increasing the collector dosage is also significant in increasing the percent AI of the concentrate. The addition of sodium silicate is shown to reduce the AI in the concentrate while not significantly affecting the BPL recovery to the concentrate. The significant variables and their effects on BPL recovery and concentrate percent AI are shown below.

<u>Variable</u>	<u>Change</u>	<u>Effect</u>
Collector dosage	from 0.53 to 0.65 lb/T	increased BPL recovery by 40.5% and increased AI by 1.6%
Na ₂ SiO ₃ addition	from 0 to 1 lb/T	reduced AI by 1.0%

Laboratory report sheets for collector R2 on present feed are presented in Volume II of this report.

4.4.2 Future Feed

The Plackett-Burman experimental test design for collector R2 on future feed is presented in Table 4-6 and the statistical design analyses is presented in Appendix 'B'. Results from the 12 tests are summarized in Table 4-7.

Table 4-4
Test Design
Collector R2 - Present Feed
(Task 4)

<u>Test</u>		<u>Conditioning</u>		<u>Cleaning</u>	<u>Size Cell</u>	<u>Collector</u>	<u>Collector to</u>	<u>Conditioner</u>	<u>Na₂SiO₃⁽²⁾</u>
<u>No.</u>	<u>Sequence</u>	<u>Time(min)</u>	<u>% Solids</u>	<u>Stages</u>	<u>(liters)</u>	<u>(lb/T)</u>	<u>FO⁽¹⁾ Ratio</u>	<u>pH</u>	<u>(lb/T)</u>
4F16	4	3.5	70	1	5	0.65	1:1.0	8.05	None
4F17	5	2.5	70	2	3	0.65	1:1.0	8.05	1.0
4F20	8	3.5	65	2	5	0.65	1:1.5	8.05	None
4F22	10	2.5	70	1	5	0.53	1:1.5	9.05	None
4F24	12	2.5	65	2	3	0.65	1:1.5	9.05	None
4F15	3	2.5	65	1	5	0.65	1:1.0	9.05	1.0
4F23	11	3.5	65	1	3	0.53	1:1.5	8.05	1.0
4F18	6	3.5	70	1	3	0.65	1:1.5	9.05	1.0
4F19	7	3.5	70	2	3	0.53	1:1.0	9.05	None
4F21	9	2.5	70	2	5	0.53	1:1.5	8.05	1.0
4F13	1	3.5	65	2	5	0.53	1:1.0	9.05	1.0
4F14	2	2.5	65	1	3	0.53	1:1.0	8.05	None

(1) No. 5 fuel oil

(2) When used, Na₂SiO₃ is to be added to conditioner.

Table 4-5

Test Results Summary
Collector R2 - Present Feed
(Task 4)

Test	Concentrate		% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾			
	%BPL ⁽²⁾	%AI ⁽²⁾	Wt. ⁽³⁾	BPL	AI	R2	FO ⁽⁶⁾	Na ₂ SiO ₃	NaOH
4F13	71.60	2.00	1.27	6.56	0.03	0.54	0.54	1.02	0.23
4F14	70.95	4.23	10.90	55.61	0.57	0.53	0.53	.00	0.18
4F15	71.32	3.42	9.53	46.86	0.41	0.67	0.67	1.02	0.33
4F16	69.75	5.28	19.14	93.22	1.26	0.65	0.65	.00	0.20
4F17	71.70	3.23	14.22	70.84	0.57	0.65	0.65	1.00	0.20
4F18	69.66	5.20	17.77	87.10	1.15	0.65	0.98	1.00	0.40
4F19	71.23	3.06	13.81	67.73	0.53	0.53	0.53	.00	0.40
4F20	70.12	4.66	19.12	93.94	1.11	0.66	0.99	.00	0.21
4F21	72.43	2.50	2.62	15.08	0.08	0.55	0.83	1.04	0.21
4F22	72.59	2.81	3.71	19.40	0.13	0.54	0.80	.00	0.41
4F23	72.43	2.99	1.89	9.75	0.07	0.56	0.84	1.05	0.21
4F24	69.84	5.56	4.95	25.04	0.35	0.65	0.98	.00	0.41

4100

- (1) Bone Phosphate of Lime Ca₃(PO₄)₂
- (2) Acid Insoluble Material
- (3) 100 (Concentrate weight)/(Feed weight)
- (4) 100 (Concentrate units)/(Feed units)
- (5) Pounds Reagent Per Short Ton of Feed
- (6) No. 5 fuel oil

Table 4-6

Test Design
Collector R2 - Future Feed
(Task 4)

Test		Conditioning		Cleaning	Cell Size	Collector	Collector to	Conditioner	Na ₂ SiO ₃ ⁽²⁾
No.	Sequence	Time(min)	% Solids	Stages	(liters)	(lb/T)	FO ⁽¹⁾ Ratio	pH	(lb/T)
4F44	6	3.5	70	1	5	0.56	1:1.0	8.50	None
4F46	8	2.5	70	2	3	0.56	1:1.0	8.50	1.0
4F50	12	3.5	65	2	5	0.56	1:1.5	8.50	None
4F42	4	2.5	70	1	5	0.46	1:1.5	9.50	None
4F40	2	2.5	65	2	3	0.56	1:1.5	9.50	None
4F41	3	2.5	65	1	5	0.56	1:1.0	9.50	1.0
4F45	7	3.5	65	1	3	0.46	1:1.5	8.50	1.0
4F47	9	3.5	70	1	3	0.56	1:1.5	9.50	1.0
4F49	11	3.5	70	2	3	0.46	1:1.0	9.50	None
4F43	5	2.5	70	2	5	0.46	1:1.5	8.50	1.0
4F39	1	3.5	65	2	5	0.46	1:1.0	9.50	1.0
4F48	10	2.5	65	1	3	0.46	1:1.0	8.50	None

4-9

(1) No. 5 fuel oil

(2) When used, Na₂SiO₃ is to be added to conditioner.

Table 4-7

Test Results Summary
Collector R2 - Future Feed
(Task 4)

Test	Concentrate			% Recovery ⁽⁴⁾				Reagent, lb/T ⁽⁵⁾			
	%BPL ⁽¹⁾	%AI ⁽²⁾	%MgO	Wt. ⁽³⁾	BPL	AI	MgO	R2	FO ⁽⁶⁾	Na ₂ SiO ₃	NaOH
4F39	58.56	5.24	2.92	21.30	87.86	1.51	48.77	0.47	0.47	1.02	0.39
4F40	47.04	21.54	2.78	27.72	92.61	8.12	58.38	0.57	0.86	.00	0.40
4F41	45.91	22.20	3.00	28.65	92.43	8.69	63.25	0.57	0.57	1.02	0.40
4F42	50.79	16.75	2.76	25.50	92.79	5.78	54.46	0.46	0.69	.00	0.37
4F43	62.68	3.54	1.98	18.18	80.23	0.87	31.42	0.47	0.71	1.03	0.27
4F44	53.03	11.20	3.12	24.25	91.05	3.70	56.16	0.57	0.57	.00	0.28
4F45	56.87	7.65	2.78	23.08	87.89	2.45	46.75	0.49	0.73	1.06	0.27
4F46	56.59	6.17	3.14	22.07	88.27	1.86	50.84	0.57	0.57	1.02	0.27
4F47	46.57	24.79	2.32	28.08	93.66	9.46	47.03	0.57	0.85	1.01	0.39
4F48	45.82	22.44	3.00	28.44	92.44	8.69	62.35	0.47	0.47	.00	0.25
4F49	51.25	15.27	3.06	19.96	74.51	4.09	46.44	0.46	0.46	.00	0.39
4F50	58.93	5.78	2.58	21.55	89.84	1.69	45.18	0.57	0.86	.00	0.27

4-10

(1) Bone Phosphate of Lime Ca₃(PO₄)₂
 (2) Acid Insoluble Material
 (3) 100 (Concentrate Weight)/Feed Weight
 (4) 100 (Concentrate Units)/(Feed Units)
 (5) Pounds Reagent Per Short Ton of Feed
 (6) No. 5 fuel oil

The data indicate that changing from one to two cleaning stages is significant in reducing the AI in the concentrate and increasing conditioning pulp pH is significant in increasing the AI in the concentrate. The significant variables and their effects on BPL recovery and concentrate percent AI are shown below.

<u>Variable</u>	<u>Change</u>	<u>Effect</u>
Cleaning Stages	from 1 to 2	reduced AI by 7.9%
Conditioning Pulp pH	from 8.5 to 9.5	increased AI by 8.2%

Laboratory report sheets for collector R2 on future feed are presented in Volume II of this report.

4.5 **Reagent R3**

Anionic collector R3 is a blend of tall oil fatty acids. Refer to Table 2-3 for specific data for collector R3.

4.5.1 Present Feed

The Plackett-Burman experimental test design for collector R3 on present feed is presented in Table 4-8 and the statistical design analysis is presented in Appendix 'B'. Results from the 12 tests are summarized in Table 4-9.

The data indicate that increasing the conditioning time and increasing the conditioner pulp percent solids are significant in improving the BPL recovery. The addition of sodium silicate is shown to reduce the concentrate percent AI while not significantly affecting the BPL recovery. The significant variables and their effects on BPL recovery and concentrate AI are shown below.

<u>Variable</u>	<u>Change</u>	<u>Effect</u>
Conditioning time	from 2.5 to 3.5 minutes	increase BPL recovery by 43.5%
Conditioner % Solids	from 65 to 70%	increase BPL recovery by 32.6%
Na ₂ SiO ₃ addition	from 0 to 1 lb/T	reduce AI by 7.6%

Table 4-8

Test Design
Collector R3 - Present Feed
(Task 4)

Test		Conditioning		Cleaning Stages	Cell Size (liters)	Collector (lb/T)	Collector to FO(1) Ratio	Conditioner pH	Na ₂ SiO ₃ (2) (lb/T)
No.	Sequence	Time(min)	% Solids						
4F26	2	3.5	70	1	5	0.67	1:1.0	8.20	None
4F32	8	2.5	70	2	3	0.67	1:1.0	8.20	1.0
4F36	12	3.5	65	2	5	0.67	1:1.5	8.20	None
4F29	5	2.5	70	1	5	0.55	1:1.5	9.20	None
4F30	6	2.5	65	2	3	0.67	1:1.5	9.20	None
4F33	9	2.5	65	1	5	0.67	1:1.0	9.20	1.0
4F35	11	3.5	65	1	3	0.55	1:1.5	8.20	1.0
4F28	4	3.5	70	1	3	0.67	1:1.5	9.20	1.0
4F25	1	3.5	70	2	3	0.55	1:1.0	9.20	None
4F31	7	2.5	70	2	5	0.55	1:1.5	8.20	1.0
4F34	10	3.5	65	2	5	0.55	1:1.0	9.20	1.0
4F27	3	2.5	65	1	3	0.55	1:1.0	8.20	None

4-12

(1) No. 5 fuel oil

(2) When used, Na₂SiO₃ is to be added to conditioner.

Table 4-9

Test Results Summary
Collector R3 - Present Feed
(Task 4)

Test	Concentrate		% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾			
	%BPL ⁽¹⁾	%AI ⁽²⁾	Wt. ⁽³⁾	BPL	AI	R3	FO ⁽⁶⁾	Na ₂ SiO ₃	NaOH
4F25	68.55	6.68	18.32	87.58	1.53	0.56	0.56	.00	0.48
4F26	64.12	12.77	20.76	93.44	3.31	0.68	0.68	.00	0.31
4F27	62.65	14.74	5.36	23.78	0.98	0.55	0.56	.00	0.30
4F28	67.35	8.38	18.63	88.27	1.95	0.67	1.01	1.01	0.47
4F29	58.41	21.26	13.06	51.81	3.47	0.57	0.86	.00	0.49
4F30	57.02	23.74	2.99	12.19	0.88	0.67	1.01	.00	0.53
4F31	72.60	1.50	1.23	6.16	0.02	0.56	0.84	1.02	0.34
4F32	70.85	3.63	10.34	51.20	0.47	0.69	0.69	1.03	0.36
4F33	66.98	8.94	1.05	5.09	0.12	0.68	0.68	1.01	0.55
4F34	66.74	9.48	0.09	0.43	0.01	0.55	0.55	1.00	0.50
4F35	64.86	11.42	15.32	70.59	2.19	0.55	0.83	1.01	0.37
4F36	65.60	9.84	15.05	70.69	1.85	0.69	1.04	.00	0.36

- (1) Bone Phosphate of Lime Ca₃(PO₄)₂
- (2) Acid Insoluble Material
- (3) 100 (Concentrate weight)/(Feed weight)
- (4) 100 (Concentrate units)/(Feed units)
- (5) Pounds Reagent Per Short Ton of Feed
- (6) No. 5 fuel oil

The laboratory report sheets for collector R3 on present feed are presented in Volume II of this report.

4.5.2 Future Feed

The Plackett-Burman experimental test design for collector R3 on future feed is presented in Table 4-10 and the statistical design analysis is presented in Appendix 'B'. Results from the 12 tests are summarized in Table 4-11.

The data indicate that increasing the collector dosage is significant in improving the BPL recovery, whereas decreasing the cell pulp density, adding sodium silicate, and adding a second cleaning stage are significant in reducing the BPL recovery. The addition of a second cleaning stage and decreasing the cell pulp density are shown to be significant in reducing the AI in the concentrate. The significant variables and their effects on BPL recovery and concentrate percent AI are shown below.

<u>Variable</u>	<u>Change</u>	<u>Effect</u>
Cleaning stages	from 1 to 2	reduced BPL recovery by 4.9% and reduced AI by 3.8%
Flotation cell water content	from 68 to 80% (from 3 liter cell to 5 liter cell)	reduced BPL recovery by 5.3% and reduced AI by 4.3%
Collector dosage	from 0.36 to 0.44 lb/T	increased BPL recovery by 3.7%
Na ₂ SiO ₄ addition	from 0 to 1 lb/T	reduced BPL recovery by 2.6%

The laboratory report sheets for collector R3 on future feed are presented in Volume II of this report.

4.6 **Reagent R4**

Anionic collector R4 is a commercial petroleum sulfonate produced by American Cyanamid Company. Refer to Table 2-3 for specific data for collector R4.

Table 4-10
Test Design
Collector R3 - Future Feed
(Task 4)

<u>Test</u>		<u>Conditioning</u>		<u>Cleaning Stages</u>	<u>Cell Size (liters)</u>	<u>Collector (lb/T)</u>	<u>Collector to FO⁽¹⁾ Ratio</u>	<u>Conditioner pH</u>	<u>Na₂SiO₃⁽²⁾ (lb/T)</u>
<u>No.</u>	<u>Sequence</u>	<u>Time(min)</u>	<u>% Solids</u>						
4F54	4	3.5	70	1	5	0.44	1:1.0	8.45	None
4F55	5	2.5	70	2	3	0.44	1:1.0	8.45	1.0
4F58	8	3.5	65	2	5	0.44	1:1.5	8.45	None
4F62	12	2.5	70	1	5	0.36	1:1.5	9.45	None
4F60	10	2.5	65	2	3	0.44	1:1.5	9.45	None
4F52	2	2.5	65	1	5	0.44	1:1.0	9.45	1.0
4F51	1	3.5	65	1	3	0.36	1:1.5	8.45	1.0
4F57	7	3.5	70	1	3	0.44	1:1.5	9.45	1.0
4F53	3	3.5	70	2	3	0.36	1:1.0	9.45	None
4F56	6	2.5	70	2	5	0.36	1:1.5	8.45	1.0
4F59	9	3.5	65	2	5	0.36	1:1.0	9.45	1.0
4F61	11	2.5	65	1	3	0.36	1:1.0	8.45	None

(1) No. 5 fuel oil

(2) When used, Na₂SiO₃ is to be added to conditioner.

Table 4-11

Test Results Summary
Collector R3 - Future Feed
(Task 4)

Test	Concentrate			% Recovery ⁽⁴⁾				Reagent, lb/T ⁽⁵⁾			
	%BPL ⁽¹⁾	% AI ⁽²⁾	%MgO	Wt. ⁽³⁾	BPL	AI	MgO	R3	FO ⁽⁶⁾	Na ₂ SiO ₃	NaOH
4F51	50.45	19.30	2.44	22.31	78.37	5.83	43.24	0.36	0.54	1.00	0.25
4F52	52.81	13.16	3.06	18.36	68.79	3.25	45.33	0.45	0.45	1.02	0.33
4F53	51.01	15.84	2.86	19.10	68.03	4.10	43.70	0.37	0.37	.00	0.31
4F54	55.35	11.64	2.50	21.10	81.32	3.33	41.82	0.44	0.44	.00	0.24
4F55	59.41	4.35	1.71	16.97	74.03	0.99	27.98	0.45	0.44	1.01	0.23
4F56	64.60	2.45	2.00	8.76	39.52	0.29	13.79	0.36	0.54	1.01	0.23
4F57	48.27	20.77	2.52	26.45	89.58	7.41	52.50	0.45	0.67	1.02	0.28
4F58	58.75	3.36	2.98	13.01	53.22	0.59	30.19	0.45	0.68	.00	0.22
4F59	60.83	2.63	2.74	6.26	27.97	0.22	13.61	0.37	0.37	1.01	0.27
4F60	49.31	19.22	2.52	25.10	87.88	6.52	47.59	0.45	0.67	.00	0.29
4F61	46.95	21.02	2.90	23.43	76.80	6.66	54.86	0.37	0.37	.00	0.23
4F62	57.34	11.23	1.92	18.32	74.38	2.77	27.95	0.37	0.55	.00	0.26

- (1) Bone phosphate of lime Ca₃(PO₄)₂
- (2) Acid insoluble material
- (3) 100 (Concentrate weight)/(Feed weight)
- (4) 100 (Concentrate units)/(Feed units)
- (5) Pounds reagent per short ton of feed
- (6) No. 5 fuel oil

4.6.1 Future Feed

The Plackett-Burman experimental test design for collector R4 on future feed is presented in Table 4-12 and the statistical design analysis is presented in Appendix 'B'. Results from the 12 tests are summarized in Table 4-13.

The data indicate that increasing the conditioner percent solids and using No. 5 fuel oil instead of No 2 fuel oil are significant in increasing the BPL recovery; whereas, decreasing the cell pulp density, increasing the conditioning and flotation cell pH and feed scrubbing with sodium silicate are significant in reducing the BPL recovery. Using No. 5 fuel oil instead of No. 2 fuel oil also resulted in an increase in the concentrate AI and MgO contents. The significant variables and their effects on BPL recovery and concentrate AI and MgO are shown below.

<u>Variable</u>	<u>Change</u>	<u>Effect</u>
Conditioner % solids	from 65 to 70%	increased BPL recovery by 10.3%
Flotation cell water content	from 68 to 80% (from 3 liter cell to 5 liter cell)	reduced BPL recovery by 16.7%
Fuel oil	from No. 2 fuel oil to No. 5 fuel oil	increased BPL recovery by 1.3% increased AI by 3.9% and increased MgO by 0.5%
Conditioning and flotation pH	from 4.0 to 9.0	reduce BPL recovery by 6.5%, increased AI by 6.7%
Feed scrubbing with Na ₂ SiO ₃	without to with	reduced BPL recovery by 7.5%

Laboratory report sheets for collector R4 on future feed are presented in Volume II of this report.

4.7 Discussion of Results

The best performances for each feed/reagent combination in Task 4 are given in Table 4-14. The tabulated performances represent the best performance obtained during the evaluation of the test variables; they do not, however, necessarily reflect optimums.

Table 4-12

Test Design
Collector R4 - Future Feed
(Task 4)

Test		Conditioning		Cleaning	Cell Size	Collector	Fuel Oil	Conditioner	Feed
No.	Sequence	Time(min)	% Solids	Stages	(liters)	(lb/T)	Type ⁽¹⁾	pH ⁽²⁾	Scrub ⁽³⁾
4F72	10	3.5	70	1	5	2.27	No.2	Acid (4.0)	None
4F66	4	2.5	70	2	3	2.27	No.2	Acid (4.0)	Yes
4F64	2	3.5	65	2	5	2.27	No.5	Acid (4.0)	None
4F74	12	2.5	70	1	5	1.85	No.5	Base (9.0)	None
4F67	5	2.5	65	2	3	2.27	No.5	Base (9.0)	None
4F70	8	2.5	65	1	5	2.27	No.2	Base (9.0)	Yes
4F73	11	3.5	65	1	3	1.85	No.5	Acid (4.0)	Yes
4F65	3	3.5	70	1	3	2.27	No.5	Base (9.0)	Yes
4F63	1	3.5	70	2	3	1.85	No.2	Base (9.0)	None
4F69	7	2.5	70	2	5	1.85	No.5	Acid (4.0)	Yes
4F71	9	3.5	65	2	5	1.85	No.2	Base (9.0)	Yes
4F68	6	2.5	65	1	3	1.85	No.2	Acid (4.0)	None

(1) Ratio will be constant at 1.0 part collector to 0.2 part fuel oil.

(2) Acid pH and cell pH should be at 4.0; both adjusted with H₂SO₄. Also, Aero 65 frother addition to the cell is required with acid pH. Base pH = 9.0 is adjusted with NaOH; cell requires no pH adjustment or frother addition.

(3) Feed scrub at 65% solids for 2.0 minutes with 1.0 lb Na₂SiO₃/ton feed followed by jet rinse.

Table 4-13

Test Results Summary
Collector R-4 - Future Feed
(Task 4)

Test	Concentrate			% Recovery ⁽⁴⁾				Reagent, lb/T				
	%BPL ⁽¹⁾	%AI ⁽²⁾	%MgO	Wt/ ⁽³⁾	BPL	AI	MgO	R4	FO No. 2	FO No. 5	Aero 65	Na ₂ SiO ₃
4F 63	61.66	6.68	1.77	21.04	90.57	1.89	33.41	1.90	0.38	0	0	0
4F 64	68.20	2.66	0.95	16.78	80.17	0.60	13.19	2.33	0	0.47	0.16	0
4F 65	51.57	20.21	1.86	26.17	93.79	7.13	41.22	2.31	0	0.46	0	1.02
4F 66	66.52	2.90	1.20	18.45	87.60	0.72	18.83	2.33	0.47	0	0.19	1.03
4F 67	58.58	10.63	1.81	21.64	91.20	3.10	32.25	2.31	0	0.46	0	0
4F 68	66.14	3.43	1.26	18.80	87.49	0.87	19.17	1.89	0.38	0	0.12	0
4F 69	65.02	3.12	1.58	18.70	85.97	0.79	24.49	1.89	0	0.38	0.24	1.02
4F 70	64.18	7.84	0.94	10.44	47.13	1.11	8.00	2.32	0.46	0	0	1.02
4F 71	61.10	3.04	1.04	13.90	63.54	0.56	11.68	1.85	0.37	0	0	1.00
4F 72	67.17	2.78	0.97	16.69	78.99	0.63	13.65	2.32	0.46	0	0.12	0
4F 73	62.50	3.56	2.08	20.27	88.67	0.97	37.01	1.88	0	0.38	0.12	1.02
4F 74	58.30	10.12	1.92	20.26	83.35	2.78	31.12	1.89	0	0.38	0	0

4-19

- (1) Bone phosphate of lime Ca₃(PO₄)₂
- (2) Acid insoluble material
- (3) 100 (Concentrate weight)/(Feed weight)
- (4) 100 (Concentrate units)/(Feed units)
- (5) Pounds reagent per short ton of feed

Table 4-14

Task 4 Best Flotation Performance

<u>Test</u>	<u>Feed</u>	<u>Reagent</u>	<u>% BPL Recovery</u>	<u>Concentrate</u>		
				<u>% BPL</u>	<u>% AI</u>	<u>% MgO</u>
4F7	P	R1	93.95	69.3	5.27	NA
4F8	P	R1	96.09	68.4	6.96	NA
4F2	P	R1	94.15	69.4	6.39	NA
4F4	P	R1	93.40	69.7	5.71	NA
4F16	P	R2	93.22	69.8	5.28	NA
4F20	P	R2	93.94	70.1	4.66	NA
4F25	P	R3	87.58	68.6	6.68	NA
4F46	F	R2	88.27	56.6	6.17	3.14
4F50	F	R2	89.84	58.9	5.78	2.58
4F39	F	R2	87.86	58.6	5.24	2.92
4F55	F	R3	74.03	59.4	4.35	1.71
4F73	F	R4	88.67	62.5	3.56	2.08
4F63	F	R4	90.57	61.7	6.68	1.77
4F69	F	R4	85.97	65.0	3.12	1.58
4F68	F	R4	87.49	66.1	3.43	1.26

Feed Identification

P - Present

F - Future

For present feed, it is clear that reagents R1 and R2 are best. Reagent R3 gave 6.36% and 8.51% lower BPL recovery than reagent R2 and R1, respectively. Concentrate grade for reagent R1 and R2 was slightly higher than for reagent R3.

For future feed, reagents R2 and R4 were superior. In addition, reagent R4 showed some selectivity with regard to MgO rejection.

Based on the above, the two reagents recommended for process optimization testing with present feed in Task 5 are R1 and R2. The two reagents recommended for process optimization testing with future feed in Task 5 are R2 and R4. It is recognized that R4 is more expensive than R2 and R3, however, it is felt that the potential for MgO rejection coupled with comparable BPL recovery and grade justified the inclusion of reagent R4 in the Task 5 test program.

SECTION 5

PROCESS OPTIMIZATION

5.1 **Objective**

The objective of Task 5 was to determine optimum performance for the anionic rougher-cleaner process and the Crago process in tests of present and future feed using the anionic collectors selected in the process definition phase (Task 4).

5.2 **Test Program**5.2.1 Summary

The formal anionic rougher-cleaner test program on present and future feed was carried out in two phases. The initial phase consisted of tests to identify process variables that significantly influenced flotation performance. After the critical variables were identified, the second phase of tests were conducted to optimize the process performance. A similar parallel series of tests were conducted to optimize the Crago process performance. The test program is outlined as follows:

<u>Test</u>	<u>Feed</u>	<u>Collector</u>	<u>Test Description</u>	<u>Tests</u>
Anionic	Present	R1	Variable Identification	8
			Optimization	6
	Future	R2	Variable Identification	8
			Optimization	6
		R2	Variable Identification	8
			Optimization	6
	R4	Variable Identification	16	
		Optimization	12	
Crago	Present	R2	Variable Identification	8
			Optimization	6
	Future	R2	Variable Identification	8
			Optimization	4
Misc.	Present	R2	Water Recycle	3
			Gangue Rejection	<u>3</u>

Total Formal Tests - 102

Locked cycle tests were conducted to simulate the effect of recycle streams and to provide a data base for comparing the anionic rougher-cleaner and the Crago processes. The test program is outlined as follows:

<u>Test</u>	<u>Feed</u>	<u>Collector</u>	<u>Test Description</u>	<u>Tests</u>
Anionic	Present	R2	Locked Cycle - 1	10
			Locked Cycle - 2	10
Crago	Present	R2	Intermediate	<u>4</u>
Total Formal Tests				24

In accordance with the amended scope of work, additional testing was carried out to evaluate anionic rougher-cleaner flotation of 14 x 150 mesh, 14 x 35 mesh and 35 x 150 mesh size fractions of feed obtained from present ore. Again, the testwork was carried out in two phases. The initial phase identified critical process variables and the second phase optimized the performances of the anionic rougher-cleaner and Crago processes. The test program is outlined as follows:

<u>Test</u>	<u>Feed</u>	<u>Collector</u>	<u>Test Description</u>	<u>Tests</u>
Anionic	14 x 150	R2	Variable Identification	8
			Optimization	6
	14 x 35	R2	Variable Identification	8
			Optimization	5
	35 x 150	R2	Variable Identification	8
			Optimization	5
Crago	14 x 150	R2	Optimization	6
	14 x 35	R2	Optimization	4
	35 x 150	R2	Optimization	<u>4</u>
Total Formal Tests				54

The process optimization test program (Task 5) contained a total of 180 formal flotation tests

5.2.2 Procedures

The two reagents tested with present feed in Task 5 were R1 and R2. The two reagents tested with future feed in Task 5 were R2 and R4.

The reagents selected for testing present and future feed in Task 5 were evaluated using one of two statistical experimental designs. One design, a 1/16 replicate of a seven factor experiment (Box-Wilson), consists of eight tests to determine the steepest ascent line and six additional tests to seek the optimum. The second design is a three factor-two level factorial experiment and also consists of eight tests. Following the eight tests are six more tests to seek the optimum performance.

Specific ZW procedures followed in the execution of Task 5 are listed below and are presented in Appendix 'A' of this report.

<u>Procedure</u>	<u>Title</u>
002.2	Ore Washing
003.1	Anionic Rougher Flotation - Bench Testing
004.0	Bench Test Mass Balance Calculation
005.1	Feed Scrubbing
006.0	Anionic Cleaner Flotation - Bench Testing
007.1	Reagent Preparation and Use
008.0	Cationic Flotation - Bench Testing
009.0	Locked Cycle Flotation Tests

5.2.3 Results

For present feed, collectors R1 and R2 gave similar optimum recovery and grade using the anionic rougher-cleaner process. For future feed, collector R2 gave better optimum performance than collector R4, using the anionic rougher-cleaner process. As a result, collector R2 was selected for the subsequent Crago, locked cycle, and sized feed flotation testwork.

The bench scale locked cycle tests showed that, with tailings recycle, the anionic rougher-cleaner process gave over a 2 percent BPL recovery improvement compared to the Crago process. Without tailings recycle, the BPL recovery improvement was less than 2 percent.

Test results showed that flotation of sized feed fractions, using the anionic rougher-cleaner process, gives a recovery and concentrate grade improvement over unsized feed flotation. The Crago process demonstrated a similar recovery advantage for sized feed flotation, however, the concentrate grade was slightly lower for sized flotation.

Relative to the Crago process, the anionic rougher-cleaner process gave lower grade concentrate, higher phosphate recovery and lower reagent cost for 28 x 150 present feed.

5.3 **Reagent R1**

Anionic collector R1 is a blend of fatty acid soaps and sulfates. Refer to Table 2-3 for specific data for collector R1.

5.3.1 Present Feed

The Box-Wilson experimental test design for collector R1 on present feed is presented in Table 5-1 and the statistical design analysis is presented in Appendix 'B'. Results from the eight tests (5F01-5F08) are summarized in Table 5-2.

For the test conditions, the data indicate that collector dosage and the number of cleaning stages are the most significant variables affecting BPL recovery and concentrate AI. Testing to determine optimum performance was accomplished by using the Box-Wilson method of steepest ascent to select six additional tests located on the line of expected maximum response.

Table 5-1
Test Design
Collector R1 - Present Feed
(Task 5)

<u>Test</u>		<u>Conditioning</u>		<u>Cleaning</u>	<u>Cell Size</u>	<u>Collector</u>	<u>Collector to</u>
<u>No.</u>	<u>Sequence</u>	<u>Time (min.)</u>	<u>% Solids</u>	<u>Stages</u>	<u>(liter)</u>	<u>(lb/T)</u>	<u>FO⁽¹⁾ Ratio</u>
5F05	5	3	68	1	3	0.61	1:1.1
5F08	8	3	72	2	3	0.69	1:1.1
5F01	1	3	72	1	3	0.69	1:1.3
5F03	3	3	68	2	3	0.61	1:1.3
5F04	4	3	72	1	5	0.61	1:1.3
5F06	6	3	68	2	5	0.69	1:1.3
5F02	2	3	68	1	5	0.69	1:1.1
5F07	7	3	72	2	5	0.61	1:1.1

(1) No. 5 fuel oil

Comments: (a) No feed scrubbing
 (b) Na₂SiO₃ not used
 (c) Conditioner pH at ambient

Table 5-2

Test Results Summary
Collector R1 - Present Feed
(Task 5)

Test	Concentrate		% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾		
	%BPL ⁽¹⁾	%AI ⁽²⁾	Wt. ⁽³⁾	BPL	AI	R1	FO ⁽⁶⁾	Na ₂ SiO ₃
5F01	67.78	7.72	18.18	86.46	1.74	0.70	0.91	.00
5F02	67.69	7.48	18.54	89.73	1.72	0.70	0.77	.00
5F03	70.10	3.77	9.31	47.37	0.43	0.60	0.78	.00
5F04	70.29	3.58	12.07	61.68	0.53	0.61	0.79	.00
5F05	68.94	5.53	14.63	71.80	1.01	0.61	0.67	.00
5F06	68.94	5.88	17.74	87.24	1.30	0.70	0.90	.00
5F07	69.33	4.69	9.31	47.18	0.54	0.61	0.67	.00
5F08	69.23	4.53	15.75	79.42	0.89	0.68	0.75	.00
5F68	66.97	7.37	20.38	93.73	1.90	0.71	0.85	.00
5F69	66.24	7.70	20.67	95.09	2.00	0.73	0.88	.00
5F70	67.06	7.77	20.54	95.05	2.00	0.75	0.90	.00
5F71	68.62	5.69	19.03	90.53	1.36	0.70	0.85	.00
5F72	68.16	6.06	19.86	93.09	1.51	0.71	0.85	0.20
5F73	68.81	5.25	18.79	89.93	1.23	0.65	0.78	.00

- (1) Bone phosphate of lime Ca₃(PO₄)₂
- (2) Acid insoluble material
- (3) 100 (Concentrate Weight)/(Feed Weight)
- (4) 100 (Concentrate Unit)/(Feed Units)
- (5) Pounds reagent per short ton of feed
- (6) No. 5 fuel oil

Comments: (a) Conditioning time = 3 minutes

The six test optimization design for collector R1 on present feed is presented in Table 5-3. Results from the six tests (5F68-5F73) are summarized in Table 5-2. The best performance for collector R1 on present feed is from test 5F69 and is summarized as follows:

<u>Test</u>	<u>Concentrate</u>		<u>% Recovery</u>		
	<u>% BPL</u>	<u>% AI</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>
5F69	66.84	7.70	20.67	95.13	2.00

Laboratory report sheets for collector R1 on present feed are presented in Volume II of this report.

5.4 **Reagent R2**

Anionic collector R2 is a blend of tall oil fatty acids. Refer to Table 2-3 for specific data for collector R2.

5.4.1 Present Feed

The factorial test design for collector R2 on present feed is presented in Table 5-4 and the statistical design analysis is presented in Appendix 'B'. Results from the eight tests (5F9-5F16) are summarized in Table 5-5.

The data indicate, for the test conditions, that conditioning pH and number of cleaning stages are the most significant variables affecting BPL recovery and concentrate AI. The six test optimization design for collector R2 on present feed is presented in Table 5-6. Results from the six tests (5F62-5F67) are summarized in Table 5-5. The best performance for collector R-2 on present feed is from tests 5F64 and 5F65 and is summarized below:

<u>Test</u>	<u>Concentrate</u>		<u>% Recovery</u>		
	<u>% BPL</u>	<u>% AI</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>
5F64	67.67	6.68	20.14	94.02	1.70
5F65	67.30	8.00	20.75	95.62	2.09

Laboratory report sheets for collector R2 on present feed are presented in Volume II of this report.

Table 5-3

Test Design
Collector R1 - Present Feed
(Task 5 Optimization)

No	Conditioning		Cleaning Stages	Cell Size (liters)	Collector (lb/T)	Collector to FO(1) Ratio
	Time(min)	% Solids				
5F68	3	70	1	5	0.70	1:1.20
5F69	3	70	1	5	0.72	1:1.20
5F70	3	70	1	5	0.74	1:1.20
5F71	3	70	2	5	0.70	1:1.20
5F72	3	70	2	5	0.70	1:1.20
5F73	3	70	2	5	0.65	1:1.20

67
1
00

(1) No. 5 fuel oil

Comments:(a) Conditioner pH at ambient

Table 5-4
Test Design
Collector R2 - Present Feed
(Task 5)

<u>Test</u>		<u>Conditioning</u>		<u>Cleaning</u>	<u>Cell Size</u>	<u>Collector</u>	<u>Collector to</u>	<u>Conditioner</u>
<u>No.</u>	<u>Sequence</u>	<u>Time(min)</u>	<u>% Solids</u>	<u>Stages</u>	<u>(liters)</u>	<u>(lb/T)</u>	<u>FO(1) Ratio</u>	<u>pH</u>
5F12	4	3	68	1	5	0.60	1:1.25	8.0
5F15	7	3	68	2	5	0.60	1:1.25	8.0
5F09	1	3	68	1	5	0.70	1:1.25	8.0
5F14	6	3	68	2	5	0.70	1:1.25	8.0
5F16	8	3	68	1	5	0.60	1:1.25	8.6
5F11	3	3	68	2	5	0.60	1:1.25	8.6
5F13	5	3	68	1	5	0.70	1:1.25	8.6
5F10	2	3	68	2	5	0.70	1:1.25	8.6

(1) No. 5 fuel oil

Table 5-5

Test Results Summary
Collector R2 - Present Feed
(Task 5)

Test	Concentrate		% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾		
	%BPL ⁽¹⁾	%AI ⁽²⁾	Wt. ⁽³⁾	BPL	AI	R2	FO ⁽⁶⁾	NaOH
5F09	70.29	4.24	10.79	54.06	0.57	0.69	0.87	0.15
5F10	69.03	5.09	17.90	88.45	1.13	0.69	0.86	0.20
5F11	70.58	3.79	13.04	65.78	0.61	0.59	0.74	0.17
5F12	69.91	4.74	12.61	63.06	0.74	0.60	0.75	0.11
5F13	68.56	6.25	18.83	91.81	1.47	0.71	0.89	0.15
5F14	70.00	4.40	14.09	71.26	0.77	0.70	0.88	0.11
5F15	69.71	3.95	11.55	57.94	0.57	0.61	0.76	0.11
5F16	69.23	4.40	15.02	74.78	0.82	0.59	0.74	0.14
5F62	70.08	3.59	3.86	18.81	0.17	0.71	0.88	0.06
5F63	67.39	6.51	19.35	90.24	1.58	0.71	0.89	0.08
5F64	67.67	6.68	20.14	94.02	1.70	0.70	0.88	0.10
5F65	67.30	8.00	20.75	95.62	2.09	0.70	0.88	0.12
5F66	67.95	5.66	17.98	84.23	1.28	0.66	0.83	0.09
5F67	70.08	3.97	13.55	65.40	0.68	0.60	0.76	0.09

- (1) Bone phosphate of lime Ca₃(PO₄)₂
- (2) Acid insoluble material
- (3) 100 (concentrate weight)/(feed weight)
- (4) 100 (concentrate units)/(feed units)
- (5) Pounds reagent per short ton of feed
- (6) No. 5 fuel oil

Comments:(a) Conditioning: 3 minutes at 68% solids

Table 5-6
Test Design
Collector R2 - Present Feed
(Task 5 - Optimization)

<u>No.</u>	<u>Conditioning</u>		<u>Cleaning</u> <u>Stages</u>	<u>Cell Size</u> <u>(liters)</u>	<u>Collector</u> <u>(lb/T)</u>	<u>Collector to</u> <u>FO⁽¹⁾ Ratio</u>	<u>Conditioner</u> <u>pH</u>
	<u>Time(min)</u>	<u>% Solids</u>					
5F 62	3	68	1	5	0.70	1:1.25	8.4
5F 63	3	68	1	5	0.70	1:1.25	8.6
5F 64	3	68	1	5	0.70	1:1.25	8.8
5F 65	3	68	1	5	0.70	1:1.25	9.0
5F 66	3	68	1	5	0.65	1:1.25	8.8
5F 67	3	68	2	5	0.60	1:1.25	8.8

(1) No. 5 fuel oil

5.4.2 Future Feed

The factorial test design for collector R2 on future feed is presented in Table 5-7 and the statistical design analysis is presented in Appendix 'B'. Results from the eight tests (5F25-5F32) are summarized in Table 5-8.

For the test conditions, the data indicate that collector dosage and number of cleaning stages are the most significant variables affecting BPL recovery and concentrate AI. The six test optimization design for collector R2 on future feed is presented in Table 5-9. Results from the six tests (5F56-5F61) are summarized in Table 5-8. The best performance for collector R2 on future feed is from test 5F59 and is summarized below:

<u>Test</u>	<u>Concentrate</u>			<u>% Recovery</u>			
	<u>%BPL</u>	<u>%AI</u>	<u>% MgO</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>	<u>MgO</u>
5F59	56.18	4.76	3.25	23.68	94.04	1.55	56.39

Laboratory report sheets for collector R2 on future feed are presented in Volume II of this report.

5.5 **Reagent R4**

Anionic collector R4 is a petroleum sulfonate product supplied by American Cyanamid Company. Refer to Table 2-3 for specific data for collector R4.

5.5.1 Future Feed

The Box-Wilxon experimental test design for collector R4 on future feed is presented in Table 5-10 and the statistical design analysis is presented in Appendix 'B'. Results from the eight tests (5F17-5F24) are summarized in Table 5-10.

Table 5-7
Test Design
Collector R2 - Future Feed
(Task 5)

<u>Test</u>		<u>Conditioning</u>		<u>Cleaning</u>	<u>Cell Size</u>	<u>Collector</u>	<u>Conditioner</u>	<u>Conditioner</u>
<u>No</u>	<u>Sequence</u>	<u>Time(min)</u>	<u>% Solids</u>	<u>Stages</u>	<u>(liters)</u>	<u>(lb/T)</u>	<u>FO(1) Ratio</u>	<u>pH</u>
5F27	3	3	68	1	5	0.50	1:1.25	8.4
5F28	4	3	68	2	5	0.50	1:1.25	8.4
5F31	7	3	68	1	5	0.60	1:1.25	8.4
5F26	2	3	68	2	5	0.60	1:1.25	8.4
5F32	8	3	68	1	5	0.50	1:1.25	9.0
5F29	5	3	68	2	5	0.50	1:1.25	9.0
5F30	6	3	68	1	5	0.60	1:1.25	9.0
5F25	1	3	68	2	5	0.60	1:1.25	9.0

(1) No 5 fuel oil

Table 5-8

Test Results Summary
Collector R2 - Future Feed
(Task 5)

Test	Concentrate			% Recovery ⁽⁴⁾				Reagent, lb/T ⁽⁵⁾		
	%BPL ⁽¹⁾	%AI ⁽²⁾	%MgO	Wt. ⁽³⁾	BPL	AI	MgO	R2	FO ⁽⁶⁾	NaOH
5F25	57.09	4.13	3.40	22.88	93.14	1.28	59.38	0.60	0.76	0.08
5F26	57.92	3.69	3.20	22.28	92.48	1.11	57.72	0.60	0.75	0.06
5F27	57.18	3.88	3.20	22.68	93.29	1.19	56.98	0.50	0.62	0.04
5F28	58.65	3.64	3.30	21.15	88.76	1.05	50.48	0.50	0.63	0.04
5F29	57.45	3.84	3.10	22.13	91.84	1.16	53.58	0.50	0.63	0.06
5F30	56.90	4.73	3.30	23.16	94.42	1.49	60.13	0.60	0.75	0.07
5F31	57.27	4.42	3.00	22.87	94.19	1.38	55.30	0.60	0.75	0.04
5F32	56.44	4.67	3.20	23.21	94.12	1.47	59.58	0.50	0.63	0.08
5F56	56.18	4.38	3.60	22.58	88.55	1.36	58.36	0.55	0.69	0.07
5F57	56.92	4.39	3.40	22.60	91.12	1.36	55.49	0.61	0.76	0.07
5F58	55.81	4.95	3.45	23.76	92.75	1.62	60.96	0.67	0.84	0.07
5F59	56.18	4.76	3.25	23.68	94.04	1.55	56.39	0.71	0.89	0.07
5F60	57.57	3.67	3.45	21.44	87.11	1.08	53.55	0.57	0.71	0.07
5F61	56.82	3.10	3.40	21.51	86.56	0.92	55.70	0.56	0.70	0.05

5-14

- (1) Bone phosphate of lime $\text{Ca}_3(\text{PO}_4)_2$
(2) Acid insoluble material
(3) 100 (concentrate weight)/(feed weight)
(4) 100 (concentrate units)/(feed units)
(5) Pounds reagent per short ton of feed
(6) No. 5 fuel oil

Comments: (a) Conditioning: 3 minutes at 68% solids

Table 5-9
Test Design
Collector R2 - Future Feed
(Task 5 Optimization)

Test No.	Conditioning		Cleaning Stages	Cell Size (liters)	Collector (lb/T)	Collector to FO ⁽¹⁾ Ratio	Conditioner pH
	Time(min)	% Solids					
5F56	3	68	1	5	0.55	1:1.25	8.7
5F57	3	68	1	5	0.60	1:1.25	8.7
5F58	3	68	1	5	0.65	1:1.25	8.7
5F59	3	68	1	5	0.70	1:1.25	8.7
5F60	3	68	2	5	0.55	1:1.25	8.7
5F61	3	68	2	5	0.55	1:1.25	8.4

(1) No. 5 fuel oil

Table 5-10

Test Design
Collector R4 - Future Feed
(Task 5)

<u>Test</u>		<u>Conditioning</u>		<u>Cleaning</u>	<u>Cell Size</u>	<u>Collector</u>	<u>Collector to</u>	<u>Conditioner</u>	<u>Feed</u>
<u>No.</u>	<u>Sequence</u>	<u>Time(min)</u>	<u>% Solids</u>	<u>Stages</u>	<u>(liters)</u>	<u>(lb/T)</u>	<u>FO⁽¹⁾ Ratio</u>	<u>pH</u>	<u>Scrub⁽²⁾</u>
5F21	5	3	68	1	3	1.8	1:0.1	3.5	none
5F18	2	3	72	2	3	1.8	1:0.3	4.5	none
5F24	8	3	72	1	5	1.8	1:0.3	3.5	yes
5F19	3	3	68	2	5	1.8	1:0.1	4.5	yes
5F22	6	3	72	1	3	2.0	1:0.1	4.5	yes
5F23	7	3	68	2	3	2.0	1:0.3	3.5	yes
5F17	1	3	68	1	5	2.0	1:0.3	4.5	none
5F20	4	3	72	2	5	2.0	1:0.1	3.5	none

(1) No. 2 fuel oil

(2) Na₂SiO₃ was not used for feed scrub

For the test conditions, the data indicate that conditioning percent solids, collector dosage and collector to fuel oil ratio are the most significant variables affecting BPL recovery and concentrate AI and MgO. Testing to determine optimum performance was accomplished by using the Box-Wilson method of steepest ascent to select seven additional tests located on the line of expected maximum response. The test optimization design for collector R4 on future feed is presented in Table 5-12. Results from the seven tests (5F49-5F55) are summarized in Table 5-11. The best performance for collector R4 on future feed is from test 5F51 and is summarized below.

<u>Test</u>	<u>Concentrate</u>			<u>% Recovery</u>			
	<u>% BPL</u>	<u>% AI</u>	<u>% MgO</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>	<u>MgO</u>
5F51	66.93	2.38	1.10	12.79	62.60	0.41	10.63

It can be seen that the above “optimized” test resulted in inferior performance when compared to earlier tests using collector R4 on future feed in Task 4. For example, test 4F68 gave the following performance:

<u>Test</u>	<u>Concentrate</u>			<u>% Recovery</u>			
	<u>% BPL</u>	<u>% AI</u>	<u>% MgO</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>	<u>MgO</u>
4F68	66.10	3.43	1.26	18.80	87.49	0.87	19.17

As a result, the experimental test design was re-run. The test design for the eight Box-Wilson tests and the six optimization tests are presented in Tables 5-13 and 5-14, respectively. Results from both test series (tests 5F94-5F101 and 5F105-5F110) are summarized in Table 5-11. The best rerun performance for collector R4 on future feed is from test 5F107 and is summarized below.

<u>Test</u>	<u>Concentrate</u>			<u>% Recovery</u>			
	<u>% BPL</u>	<u>% AI</u>	<u>% MgO</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>	<u>MgO</u>
5F107	59.43	3.31	2.85	21.41	88.07	0.97	51.95

Although the BPL recovery for test 5F107 was improved over test 5F51 and approximately equivalent to test 5F68, the MgO recovery to the concentrate was significantly higher.

Table 5-11

**Test Results Summary
Collector R4 - Future Feed
(Task 5)**

Test	Concentrate			% Recovery ⁽⁴⁾				Reagent, lb/T ⁽⁵⁾			
	%BPL (1)	%AI ⁽²⁾	%MgO	Wt. ⁽³⁾	BPL	AI	MgO	R4	FO ⁽⁶⁾	H ₂ SO ₄	Aero 65
5F17	65.66	2.64	1.55	14.63	67.12	0.52	18.44	1.98	0.59	3.39	0.06
5F18	65.37	3.18	1.48	8.95	41.44	0.39	10.66	1.81	0.54	2.67	0.08
5F19	67.98	2.92	0.96	7.35	35.18	0.29	5.98	1.81	0.18	3.69	0.08
5F20	69.31	2.20	0.83	9.60	47.22	0.28	6.60	2.00	0.20	5.96	0.08
5F21	68.46	2.63	1.11	10.37	50.48	0.37	9.76	1.83	0.18	4.72	0.06
5F22	65.08	3.09	1.53	15.10	69.86	0.63	19.31	2.01	0.20	3.39	0.06
5F23	68.07	2.35	0.90	8.16	39.80	0.26	6.32	2.01	0.60	4.87	0.08
5F24	66.43	3.27	1.15	13.53	64.73	0.60	12.83	1.82	0.54	5.06	0.06
5F49	65.82	2.42	1.35	6.70	31.73	0.22	7.31	2.06	0.36	4.98	0.08
5F50	66.00	2.26	1.30	8.58	40.80	0.27	8.20	2.06	0.33	4.95	0.08
5F51	66.93	2.38	1.10	12.79	62.60	0.41	10.63	2.12	0.30	5.57	0.08
5F52	65.82	2.48	1.55	8.98	42.49	0.30	10.54	2.21	0.27	5.63	0.08
5F53	68.04	2.26	0.90	10.53	52.52	0.32	7.39	2.09	0.31	5.63	0.08
5F54	67.20	1.36	1.20	9.42	45.90	0.17	8.67	2.15	0.28	5.64	0.08
5F55	67.48	2.10	1.00	10.17	50.38	0.29	7.96	2.18	0.30	5.75	0.08
5F94	59.93	3.56	2.75	18.65	79.59	0.90	40.69	2.06	0.62	1.70	0.08
5F95	58.45	3.40	3.15	15.80	65.72	0.73	40.77	1.81	0.54	1.90	0.08
5F96	59.28	4.08	3.00	15.57	65.89	0.86	37.29	1.84	0.18	1.02	0.06
5F97	59.37	4.04	3.00	19.55	81.77	1.07	47.04	1.86	0.56	1.70	0.06
5F98	62.15	4.02	2.40	18.74	82.41	1.01	36.41	2.02	0.61	1.51	0.06
5F99	64.01	2.91	2.02	15.09	67.88	0.59	25.14	1.85	0.18	2.21	0.08
5F100	59.00	3.70	3.05	17.05	71.21	0.85	42.10	2.07	0.21	2.17	0.06
5F101	60.30	3.34	2.80	19.89	84.76	0.90	44.50	2.03	0.20	1.42	0.08
5F105	60.45	3.02	2.80	19.85	82.65	0.83	44.31	2.00	0.40	1.28	0.06
5F106	59.62	3.15	2.75	20.76	85.81	0.90	47.54	2.03	0.39	1.36	0.06
5F107	59.43	3.31	2.85	21.41	88.07	0.97	51.95	2.08	0.39	1.47	0.06
5F108	60.17	3.59	2.80	20.67	85.95	1.01	47.54	2.14	0.39	1.50	0.06
5F110	63.04	2.65	1.90	14.93	66.04	0.54	24.05	2.15	0.40	1.51	0.08

- (1) Bone phosphate of lime Ca₃(PO₄)₂
(2) Acid insoluble material
(3) 100 (concentrate weight)/(feed weight)
(4) 100 (concentrate units)/(feed units)
(5) Pounds reagent per short ton of feed
(6) No. 2 fuel oil

Comments: (a) Conditioning: 3 minutes at pH level 3.50
(b) Cleaning stages: 2 (no feed scrub)

Table 5-12

Test Design
Collector R4 - Future Feed
(Task 5 Optimization)

No.	Conditioning		Cleaning Stages	Cell Size (liters)	Collector (lb/T)	Collector to FO ⁽¹⁾ Ratio	Conditioner pH	Feed Scrub
	Time (min)	% Solids						
5F49	3	71.2	2	5	1.96	1:0.18	3.5	no
5F50	3	72.4	2	5	2.02	1:0.16	3.5	no
5F51	3	73.5	2	5	2.08	1:0.14	3.5	no
5F52	3	74.7	2	5	2.14	1:0.12	3.5	no
5F53	3	72.9	2	5	2.05	1:0.15	3.5	no
5F54	3	74.1	2	5	2.11	1:0.13	3.5	no
5F55	3	73.5	2	5	2.14	1:0.14	3.5	no

(1) No. 2 fuel oil

Table 5-13

Test Design
Collector R4 - Future Feed
(Task 5 Re-Run)

Test		Conditioning			Cleaning	Cleaner	Collector	Collector to	Rougher
No.	Sequence	Time(min)	% Solids	pH ⁽¹⁾	Stages	Cell pH	(lb/T)	FO ⁽²⁾ Ratio	Cell pH
5F96	3	3	68	5	1	ambient	1.8	1:0.1	ambient
5F98	5	3	72	4	1	ambient	2.0	1:0.3	ambient
5F101	8	3	72	5	2	ambient	2.0	1:0.1	4.0
5F95	2	3	68	4	2	ambient	1.8	1:0.3	4.0
5F97	4	3	72	5	1	4.0	1.8	1:0.3	4.0
5F100	7	3	68	4	1	4.0	2.0	1:0.1	4.0
5F94	1	3	68	5	2	4.0	2.0	1:0.3	ambient
5F99	6	3	72	4	2	4.0	1.8	1:0.1	ambient

(1) pH adjusted with H₂SO₄
(2) No. 2 fuel oil

Comments:(a) 3 liter cell used for all tests

Table 5-14

Test Design
Collector R4 - Future Feed
(Task 5 Optimization Re-Run)

<u>Test</u>	<u>Conditioning</u>			<u>Cell Size (liter)</u>	<u>Cleaning Stages</u>	<u>Collector (lb/T)</u>	<u>Collector to FO(2) Ratio</u>
	<u>Time(min)</u>	<u>% Solids</u>	<u>pH(1)</u>				
5F105	2.5	73.5	4.4	3	1	2.00	1:0.2
5F106	2.5	73.5	4.1	3	1	2.06	
5F107	2.5	73.5	3.9	3	1	2.11	
5F108	2.5	73.5	3.7	3	1	2.17	
5F110	2.5	65.0	3.8	3	2	2.17	

(1) pH adjusted with H₂SO₄

(2) Maintain same dosage of No. 2 fuel oil for tests
5F106-5F110 as for Test 5F105.

Comments: (a) No cell pH adjustment

Since no formal procedural changes had occurred to account for the change in performance of collector R4, it was decided to return a sample of R4 to American Cyanamid for analysis to determine if its specification had changed during the test program.

Tests run by American Cyanamid confirmed that the original sample of R4 was chemically unstable. The degree of change that occurred during Tasks 3 and 4 is not known. The earlier test results were not reproducible and further testing of R4 was curtailed.

Laboratory report sheets for collector R4 on future feed are presented in Volume II of this report.

5.6 **Reagent R2 - Crago Tests**

Crago flotation tests were performed on present and future feed in order to permit comparative evaluation of the anionic rougher-cleaner and Crago processes. Reagent R2 was selected as the anionic collector for the Crago testing. The cationic collector selected was Custamine 710. Refer to Table 2-3 for specific collector data.

5.6.1 Present Feed

The Box-Wilson experimental test design for collector R2 on present feed is presented in Table 5-15 and the statistical design analysis is presented in Appendix 'B'. Results from the eight tests (5F41-5F48) are summarized in Table 5-16.

The data indicate, for the laboratory tests, that conditioning pH, collector to fuel oil ratio, acid scrub time and amine dosage are the most significant variables affecting BPL recovery and concentrate AI. Six additional tests, located on the line of maximum response, were run in order to determine optimum performance. The six test optimization design for collector R2 on

Table 5-15

Test Design
Collector R2 - Present Feed
(Task 5 Crago)

Test		Conditioning			Collector	Collector to	Acid Scrub			Amine	
No.	Sequence	Time(min)	% Solids	pH	(lb/T)	FO ⁽¹⁾ Ratio	Time(min)	% Solids	H ₂ SO ₄ ⁽²⁾	(lb/T)	Kerosene ⁽³⁾
5F44	4	3	68	8.0	0.60	1:1.0	2	65	1.2	.20	none
5F45	5	3	68	8.6	0.70	1:1.0	2	65	1.4	.30	none
5F41	1	3	68	8.6	0.60	1:1.5	2	65	1.4	.20	yes
5F47	7	3	68	8.0	0.70	1:1.5	2	65	1.2	.30	yes
5F48	8	3	68	8.6	0.60	1:1.0	3	65	1.2	.30	yes
5F46	6	3	68	8.0	0.70	1:1.0	3	65	1.4	.20	yes
5F43	3	3	68	8.0	0.60	1:1.5	3	65	1.4	.30	none
5F42	2	3	68	8.6	0.70	1:1.5	3	65	1.2	.20	none

(1) No. 5 fuel oil

(2) lb. H₂SO₄ per ton rougher feed

(3) Kerosene usage: amine/kerosene = 4/1

Comments:(a) Cell size = 5 liters

Table 5-16

Test Results Summary
Collector R2 - Present Feed
(Task 5 Crago)

<u>Test</u>	<u>Concentrate</u>		<u>% Recovery⁽⁴⁾</u>			<u>Reagent, lb/T⁽⁵⁾</u>					
	<u>% BPL⁽¹⁾</u>	<u>% AI⁽²⁾</u>	<u>Wt.⁽³⁾</u>	<u>BPL</u>	<u>AI</u>	<u>R2</u>	<u>FO⁽⁶⁾</u>	<u>NaOH</u>	<u>H₂SO₄</u>	<u>Amine</u>	<u>Kerosene</u>
5F41	71.53	1.76	10.31	51.92	0.23	0.62	0.92	0.10	1.44	0.21	0.05
5F42	71.53	2.57	17.27	85.86	0.56	0.72	1.08	0.11	1.23	0.21	.00
5F43	71.62	2.26	10.53	53.28	0.30	0.61	0.92	0.06	1.43	0.31	.00
5F44	70.89	3.14	13.14	64.46	0.52	0.61	0.61	0.06	1.22	0.20	.00
5F45	72.08	2.19	15.24	76.92	0.42	0.72	0.72	0.10	1.43	0.31	.00
5F46	72.08	2.18	13.67	68.78	0.37	0.71	0.71	0.06	1.42	0.20	0.05
5F47	72.17	1.58	5.77	29.24	0.11	0.71	1.07	0.06	1.23	0.31	0.08
5F48	71.99	2.11	15.43	77.10	0.41	0.60	0.60	0.10	1.20	0.30	0.08
5F74	70.71	2.45	16.02	80.27	0.49	0.71	0.71	0.06	1.32	0.23	.00
5F75	70.80	2.92	18.79	93.76	0.69	0.71	0.67	0.07	1.31	0.22	.00
5F76	70.06	3.13	19.10	95.07	0.75	0.71	0.57	0.09	1.32	0.20	.00
5F77	70.52	2.96	19.03	95.02	0.71	0.71	0.46	0.11	1.32	0.19	.00
5F82	71.52	2.73	9.85	49.90	0.34	0.71	0.25	0.15	1.32	0.15	.00
5F83	70.78	3.73	19.20	93.79	0.90	0.71	0.35	0.13	1.32	0.17	.00

5-24

- (1) Bone phosphate of lime $\text{Ca}_3(\text{PO}_4)_2$
(2) Acid insoluble material
(3) 100 (concentrate weight)/(feed weight)
(4) 100 (concentrate units)/(feed units)
(5) Pounds reagent per short ton of feed
(6) No. 5 fuel oil

feed is presented in Table 5-17. Results from the six tests (5F74-5F77, 5F82-5F83) are summarized in Table 5-16. The best performance for collector R2 on present feed is from tests 5F76 and 5F77 and is summarized below.

<u>Test</u>	<u>Concentrate</u>		<u>% Recovery</u>		
	<u>% BPL</u>	<u>% AI</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>
5F76	70.06	3.13	19.10	95.07	0.75
5F77	70.52	2.96	19.03	95.02	0.71

Laboratory report sheets for collector R2 on present feed using the Crago process are presented in Volume II of this report.

5.6.2 Future Feed

The Box-Wilxon experimental test design for collector R2 on future feed is presented in Table 5-18 and the statistical design analysis is presented in Appendix 'B'. Results from the eight tests (5F33-5F40) are summarized in Table 5-19.

For the experimental design, the data indicate that R2 dosage, collector to fuel oil ratio, sulfuric acid dosage and amine dosage are the most significant variables affecting BPL recovery and concentrate AI. The test optimization design for collector R2 on future feed is presented in Table 5-20. Results from the four optimization tests (5F78-5F81) are summarized in Table 5-19. The best performance for collector R2 on future feed is from test 5F79 and is summarized below.

<u>Test</u>	<u>Concentrate</u>			<u>% Recovery</u>			
	<u>% BPL</u>	<u>% AI</u>	<u>% MgO</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>	<u>MgO</u>
5F79	57.35	2.76	3.55	23.53	94.39	0.88	67.83

Laboratory report sheets for collector R2 on future feed using the Crago process are presented in Volume II of this report.

Table 5-17

**Test Design
Collector R2 - Present Feed
(Task 5 Crago Optimization)**

No.	Conditioning			Cell Size (liters)	Collector (lb/T)	Collector to FO ⁽¹⁾ Ratio	Acid Scrub			Amine (lb/T)
	Time(min)	% Solids	pH				Time(min)	% Solids	H ₂ SO ₄ (lb/T)	
5F74	3	68	8.5	5	0.7	1:1.00	2.78	65	1.3	0.23
5F75	3	68	8.7	5	0.7	1:0.95	3.06	65	1.3	0.22
5F76	3	68	8.9	5	0.7	1:0.80	3.34	65	1.3	0.20
5F77	3	68	9.1	5	0.7	1:0.65	3.62	65	1.3	0.19
5F83	3	68	9.3	5	0.7	1:0.50	3.90	65	1.3	0.17
5F82	3	68	9.5	5	0.7	1:0.35	4.20	65	1.3	0.15

5-26

(1) No. 5 fuel oil

Comments: (a) No kerosene used for tests

Table 5-18

Test Design
Collector R2 - Future Feed
(Task 5 Crago)

Test		Conditioning			Cell Size	Collector	Collector to	Acid Scrub			Amine	
No.	Sequence	Time(min)	% Solids	pH	(liters)	(lb/T)	FO ⁽¹⁾ Ratio	Time(min)	% Solids	H ₂ SO ₄ (lb/T)	(lb/T)	Kerosene ⁽²⁾
5F39	7	3	68	8.4	5	0.5	1:1.0	2	65	1.2	0.20	none
5F36	4	3	68	9.0	5	0.6	1:1.0	2	65	1.4	0.30	none
5F35	3	3	68	9.0	5	0.5	1:1.5	2	65	1.4	0.20	yes
5F33	1	3	68	8.4	5	0.6	1:1.5	2	65	1.2	0.30	yes
5F40	8	3	68	9.0	5	0.5	1:1.0	3	65	1.2	0.30	yes
5F34	2	3	68	8.4	5	0.6	1:1.0	3	65	1.4	0.20	yes
5F37	5	3	68	8.4	5	0.5	1:1.5	3	65	1.4	0.30	none
5F38	6	3	68	9.0	5	0.6	1:1.5	3	65	1.2	0.20	none

(1) No. 5 fuel oil

(2) Kerosene usage: amine/kerosene = 4/1

5-27

Table 5-19

Test Results Summary
Collector R2 - Future Feed
(Task 5 Crago)

Test	Concentrate			% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾						
	%BPL ⁽¹⁾	%AI ⁽²⁾	%MgO	Wt. ⁽³⁾	BPL	AI	MgO	R2	FO ⁽⁶⁾	NaOH	H ₂ SO ₄	Amine	Kerosene
5F33	58.09	2.05	3.30	21.18	84.99	0.59	57.61	0.60	0.91	0.06	1.21	0.30	0.08
5F34	57.08	2.78	3.40	23.30	92.33	0.88	65.46	0.61	0.61	0.06	1.42	0.20	0.05
5F35	58.28	2.52	3.10	22.30	90.72	0.76	55.87	0.51	0.76	0.08	1.42	0.20	0.05
5F36	56.71	2.43	3.60	23.78	93.45	0.79	70.59	0.60	0.60	0.08	1.41	0.30	.00
5F37	58.64	2.45	3.30	22.08	89.54	0.74	57.35	0.51	0.76	0.06	1.42	0.30	.00
5F38	59.38	2.78	3.00	22.27	90.74	0.85	53.64	0.61	0.91	0.08	1.21	0.30	.00
5F39	57.08	2.25	3.40	23.37	91.75	0.72	62.13	0.51	0.51	0.06	1.22	0.20	.00
4F40	57.81	2.18	3.55	21.29	84.22	0.64	60.91	0.51	0.51	0.07	1.22	0.30	0.08
5F78	57.72	2.55	3.55	23.36	91.47	0.81	67.87	0.67	0.80	0.06	1.36	0.23	.00
5F79	57.35	2.76	3.55	23.53	94.39	0.88	67.83	0.67	0.77	0.06	1.40	0.20	.00
5F80	57.17	2.84	3.60	23.88	94.79	0.93	68.64	0.68	0.75	0.06	1.45	0.18	.00
5F81	56.61	3.08	3.70	24.13	94.58	1.02	73.28	0.69	0.73	0.06	1.51	0.16	.00

- (1) Bone phosphate of lime Ca₃(PO₄)₂
- (2) Acid insoluble material
- (3) 100 (concentrate weight)/(feed weight)
- (4) 100 (concentrate units)/(feed units)
- (5) Pounds reagent per short ton of feed
- (6) No. 5 fuel oil

Table 5-20

Test Design
 Collector R2 - Future Feed
 (Task 5 Crago Optimization)

No	Conditioning			Cell Size (liters)	Collector (lb/T)	Collector to FO ⁽¹⁾ Ratio	Acid Scrub			Amine (lb/T)	Kerosene
	Time(min)	% Solids	pH				Time(min)	% Solids	H ₂ SO ₄ (lb/T)		
5F78	3	68	8.3	5	0.66	1:1.20	2.5	65	1.35	0.23	no
5F79	3	68	8.3	5	0.67	1:1.15	2.5	65	1.40	0.20	no
5F80	3	68	8.3	5	0.68	1:1.10	2.5	65	1.45	0.18	no
5F81	3	68	8.3	5	0.69	1:1.05	2.5	65	1.50	0.16	no

(1) No. 5 fuel oil

5.7 Miscellaneous Tests

5.7.1 Water Recycle

Three anionic rougher-cleaner bench tests were performed on present feed to determine the effect of recycle water on BPL recovery and concentrate AI. The tests were performed using collector R2 under identical test conditions except as shown below.

<u>Test</u>	<u>Type of Water Used For</u>	
	<u>Rougher Flotation</u>	<u>Cleaner Flotation</u>
5F102	tap	recycle*
5F103	tap	tap
5F104	recycle**	recycle* *

* from rougher cell water

**from locked cycle test cell water (tests 5F84-5F93)

Results from the three tests are summarized as follows:

	<u>Test</u>		
	<u>5F102</u>	<u>5F103</u>	<u>5F104</u>
Flotation Feed			
% BPL	14.6	14.5	14.6
% AI	79.3	79.6	79.7
Rougher Concentrate			
% BPL	65.1	65.5	65.1
Cleaner Concentrate			
% BPL	68.5	70.1	69.4
% AI	6.5	5.0	6.0
Yield - % weight	19.1	17.2	18.4
BPL Distribution			
Rougher Tails	7.4	7.8	8.3
Cleaner Tails	2.7	9.4	4.0
Cleaner Concentrate	89.9	82.8	87.7

The data indicate no significant difference in rougher flotation performance from the use of tap or recycle water and that the use of recycle water in cleaner flotation may improve flotation recovery and reduce concentrate grade. Chemical analysis of the laboratory tap and recycle water is discussed in Section 2.3.7.

5.7.2 Gangue Rejection Using Sodium Metasilicate

Three anionic rougher-cleaner bench tests were performed on future feed to determine the effect of sodium metasilicate on cleaner flotation BPL recovery, and concentrate AI and MgO analyses. The tests were performed using collector R2 under identical test conditions, except as shown below. Test 5F122 was used as a “base case” test.

<u>Test</u>	<u>Sodium Metasilicate Dosage (lb/T feed)</u>		
	<u>Cleaner #1</u>	<u>Cleaner #2</u>	<u>Cleaner #3</u>
5F122	0	0	0
5F123	0.25	0.25	0.25
5F124	0.49	0.49	0.49

Sodium metasilicate was not used during rougher conditioning or rougher flotation. Analyses were performed on the final cleaner concentrates and are summarized as follows:

<u>Test</u>	<u>Concentrate</u>			
	<u>% Weight</u>	<u>% BPL</u>	<u>% AI</u>	<u>MgO</u>
5F122	25.11	56.19	4.17	3.35
5F123	24.83	56.10	3.93	3.40
5F124	24.21	56.85	3.94	3.15

The data indicate that under the test conditions the use of sodium metasilicate in cleaner flotation, at one to two pounds per ton of cleaner concentrate, does not significantly lower the concentrate AI and MgO.

5.8 Locked Cycle Flotation Test

The purpose of a locked cycle test is to simulate the effect of recycle streams in a continuous process by batch testing.

ZW procedure 009.0, using present feed and collector R2, was followed for the locked cycle flotation tests. The test was specifically designed to evaluate the effects of using recycled water for cleaner flotation and adding the cleaner tailings to the subsequent test conditioner feed. The recycle of the cleaner tailings was conducted for nine tests. Results from all nine tests (5F84-5F93) are summarized in Table 5-21 and are illustrated in Figure 5-1. For the cycle test, the concentrate grade averaged 68.3 BPL at 92.1% BPL recovery. The cleaner tailing (middling) from the last test analyzed 1.4 BPL, but amounted to less than two percent of the new feed weight.

The locked cycle test using future feed and collector R4 was not performed because of the previously discussed problems (see section 5.5). Instead, a second locked cycle test using present feed and collector R2 was performed. The second locked cycle test differed from the first in that four separate Crago tests were performed between floats of the locked cycle test in order to provide a better data base for comparing flotation performance.

Results from the nine locked cycle tests and the intermediate Crago tests (5F126-5F138) are summarized in Table 5-22 and Table 5-23, respectively. The results are illustrated in Figure 5-2. For the cycle test, the concentrate grade averaged 66.91 BPL at 95.85 % BPL recovery. The cleaner tailings from the last cycle analyzed 11.54 BPL and represented about 2.3 percent of the weight of new flotation feed. For the intermediate Crago tests, the concentrate grade averaged 70.59 BPL at 93.73 % BPL recovery.

FIG. 5-1

TASK 5: PRESENT FEED LOCKED CYCLE SERIES 1

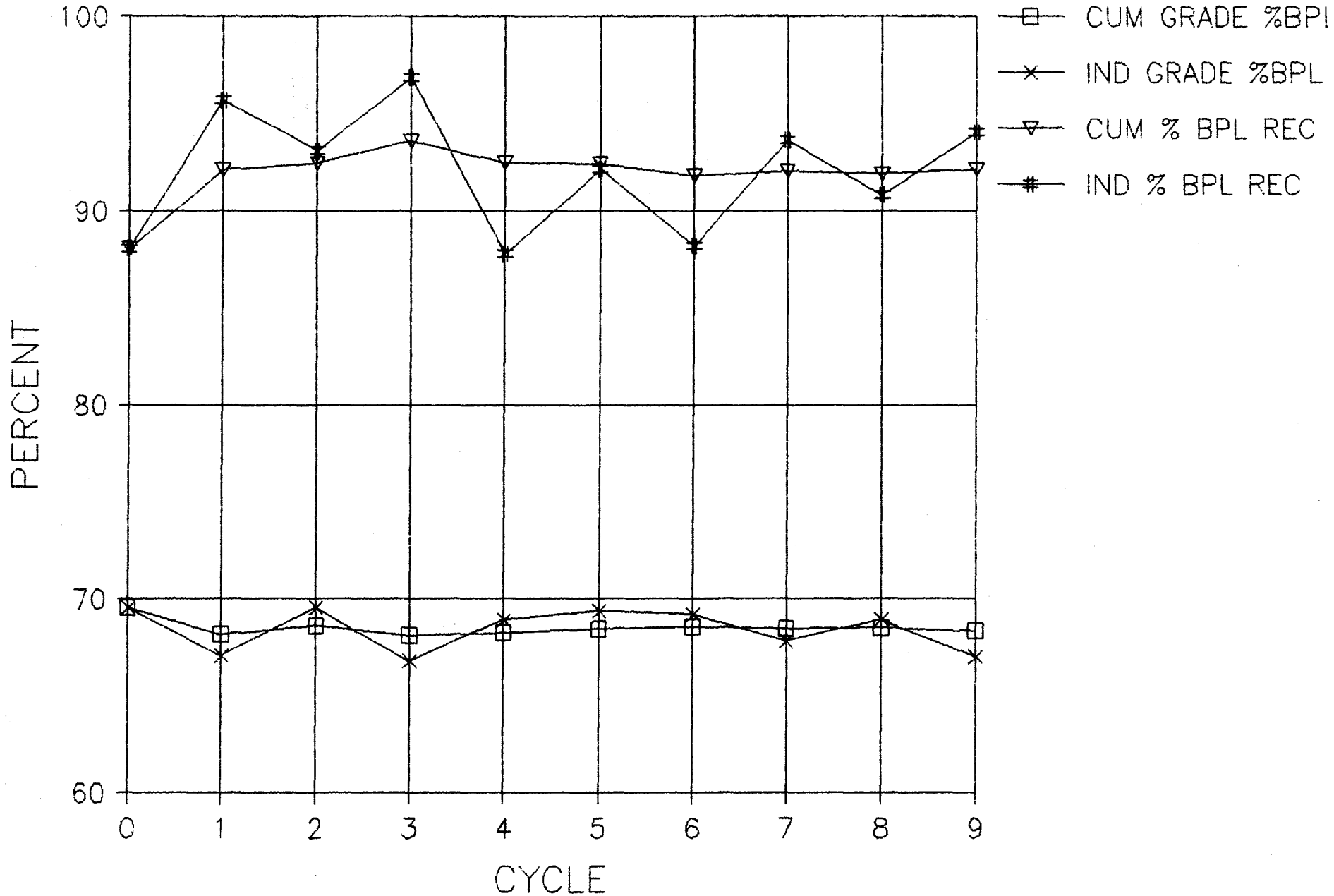


TABLE 5-21
SUMMARY DATA - LOCKED CYCLE TEST NO. 1
PRESENT 28/150 FEED

* * * * * ANALYSES OF TEST PRODUCTS * * * * *

TEST NO.	ROUGHER TAILS			CLEANER CONC			TOTAL		
	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.
5F85	791.2	.83	98.14	218.5	67.04	8.31	1009.7	15.16	78.70
5F86	795.1	1.21	97.05	186.5	69.55	6.53	981.6	14.19	79.85
5F87	787.5	.60	97.77	219.1	66.76	8.19	1006.6	15.00	78.27
5F88	809.4	2.05	95.74	172.9	68.90	5.92	982.3	13.82	79.93
5F89	801.5	1.40	96.92	190.1	69.37	5.38	991.6	14.43	79.37
5F90	816.1	2.13	95.44	187.2	69.20	4.87	1003.3	14.64	78.54
5F91	789.1	1.18	97.77	202.2	67.81	6.86	991.3	14.77	79.23
5F92	811.3	1.59	97.09	185.4	68.92	5.36	996.7	14.11	80.03
5F93	781.1	.85	98.27	211.8	66.97	7.56	992.9	14.95	78.92
AVERAGE	798.0	1.32	97.13	197.1	68.28	6.55	995.1	14.56	79.20
VARIANCE	130.71	.25	.89	240.66	1.14	1.43	88.09	.18	.37

* * * * * DISTRIBUTION OF COMPONENTS * * * * *

TEST NO.	ROUGHER TAILS			CLEANER CONC			TOTAL		
	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.
5F85	78.36	4.29	97.72	21.64	95.71	2.28	100.0	100.0	100.0
5F86	81.00	6.90	98.45	19.00	93.10	1.55	100.0	100.0	100.0
5F87	78.23	3.13	97.72	21.77	96.87	2.28	100.0	100.0	100.0
5F88	82.40	12.23	98.70	17.60	87.77	1.30	100.0	100.0	100.0
5F89	80.83	7.84	98.70	19.17	92.16	1.30	100.0	100.0	100.0
5F90	81.34	11.83	98.84	18.66	88.17	1.16	100.0	100.0	100.0
5F91	79.60	6.36	98.23	20.40	93.64	1.77	100.0	100.0	100.0
5F92	81.40	9.17	98.75	18.60	90.83	1.25	100.0	100.0	100.0
5F93	78.67	4.47	97.96	21.33	95.53	2.04	100.0	100.0	100.0
AVERAGE	80.2	7.36	98.34	19.8	92.64	1.66	100.0	100.00	100.00
VARIANCE	2.06	9.34	.18	2.06	9.34	.18	.00	.00	.00

* * * * * TEST CONDITIONS * * * * *

	MINUTES	%SOLIDS	PH
RGH CONDITIONING	3.00	68.0	8.80
RGH FLOTATION	1.30	20.0	8.20
CLNR FLOTATION	1.00	8.0	8.10

* * * * * REAGENTS * * * * *

	DOSE (MLS)	GRAMS/LITER	LE/TON FEED	CONC
R-2	.38	920	.70	3.55
NO. 5 FUEL OIL	.19	920	.35	1.77
NAOH	1.26	50	.13	.64

TABLE 5-22
SUMMARY DATA - LOCKED CYCLE TEST NO. 2
PRESENT 28/150 FEED

***** ANALYSES OF TEST PRODUCTS *****

TEST NO.	ROUGHER TAILS			CLEANER CONC			TOTAL		
	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.
5F127	791.8	.85	98.07	207.1	67.56	6.86	998.9	14.68	79.16
5F128	799.5	.69	98.17	213.0	67.47	6.73	1012.5	14.74	78.93
5F130	784.2	.58	98.50	218.2	65.81	8.43	1002.4	14.78	78.89
5F131	785.5	.69	98.30	213.9	66.18	8.05	999.4	14.71	78.98
5F132	791.2	.80	98.15	206.5	67.47	7.22	997.7	14.60	79.33
5F134	799.9	.72	98.29	213.2	67.1	7.46	1013.1	14.69	79.18
5F135	795.5	1.03	97.77	204.7	67.29	7.79	1000.2	14.59	79.35
5F136	791.9	.69	98.50	215.1	67.1	8.14	1007.0	14.88	79.20
5F137	792.9	.87	98.04	203.0	66.27	9.07	995.9	14.20	79.90
AVERAGE	792.5	.77	98.20	210.5	66.92	7.75	1003.0	14.65	79.21
VARIANCE	25.93	.02	.05	24.78	.38	.52	36.08	.03	.08

***** DISTRIBUTION OF COMPONENTS *****

TEST NO.	ROUGHER TAILS			CLEANER CONC			TOTAL		
	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.
5F127	79.27	4.59	98.20	20.73	95.41	1.80	100.0	100.0	100.0
5F128	78.96	3.70	98.21	21.04	96.30	1.79	100.0	100.0	100.0
5F130	78.23	3.07	97.67	21.77	96.93	2.33	100.0	100.0	100.0
5F131	78.60	3.69	97.82	21.40	96.31	2.18	100.0	100.0	100.0
5F132	79.30	4.35	98.12	20.70	95.65	1.88	100.0	100.0	100.0
5F134	78.96	3.87	98.02	21.04	96.13	1.98	100.0	100.0	100.0
5F135	79.53	5.61	97.99	20.47	94.39	2.01	100.0	100.0	100.0
5F136	78.64	3.65	97.80	21.36	96.35	2.20	100.0	100.0	100.0
5F137	79.62	4.88	97.69	20.38	95.12	2.31	100.0	100.0	100.0
AVERAGE	79.0	4.16	97.95	21.0	95.84	2.05	100.0	100.00	100.00
VARIANCE	.19	.54	.04	.19	.54	.04			

***** TEST CONDITIONS *****

	MINUTES	XSOLIDS	PH
RGH CONDITIONING	2.00	68.0	8.70
RGH FLOTATION	1.40	20.0	8.17
CLNR FLOTATION	1.20	8	8.15

***** REAGENTS *****

	DOSE (MLS)	GRAMS/LITER	LB/TON FEED	CONC
R-2	.40	920	.73	3.50
NO. 5 FUEL OIL	.24	920	.44	2.10
NAOH	1.20	50	.12	.57

TABLE 5-23
 SUMMARY DATA -LOCKED CYCLE TEST NO.2
 PRESENT 28/150 FEED
 INTERMEDIATE CRAGO TESTS

***** ANALYSES OF TEST PRODUCTS *****

TEST NO.	ROUGHER TAILS			AMINE TAILS			AMINE CONC			TOTAL		
	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.
5F125	784.5	.93	97.96	25.2	10.16	82.23	193.5	70.43	3.02	1003.2	14.57	79.25
5F129	780.9	.66	98.35	23.6	9.05	84.94	200.1	70.34	3.06	1004.6	14.74	79.05
5F133	785.7	1.06	97.55	24.6	10.25	84.13	195.2	70.80	3.00	1005.5	14.82	78.87
5F138	781.8	.72	98.05	25.2	13.67	81.23	196.4	70.80	3.12	1003.4	14.76	79.05
AVERAGE	783.2	.84	97.98	24.7	10.78	83.13	196.3	70.59	3.05	1004.2	14.72	79.06
VARIANCE	3.80	.03	.08	.43	3.00	2.17	5.88	.04	.00	.87	.01	.02

***** DISTRIBUTION OF COMPONENTS *****

TEST NO.	ROUGHER TAIL			AMINE TAILS			AMINE CONC			TOTAL		
	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.	WEIGHT	BPL	A. I.
5F125	78.20	4.99	96.66	2.51	1.75	2.61	19.29	93.26	.73	100.0	100.0	100.0
5F129	77.73	3.48	96.70	2.35	1.44	2.52	19.92	95.08	.77	100.0	100.0	100.0
5F133	78.14	5.59	96.65	2.45	1.69	2.61	19.41	92.72	.74	100.0	100.0	100.0
5F137	77.92	3.80	96.65	2.51	2.33	2.58	19.57	93.87	.77	100.0	100.0	100.0
AVERAGE	78.0	4.47	96.67	2.5	1.80	2.58	19.5	93.73	.75	100.0	100.0	100.0
VARIANCE	.03	.74	.00	.00	.10	.00	.06	.77	.00			

***** TEST CONDITIONS *****

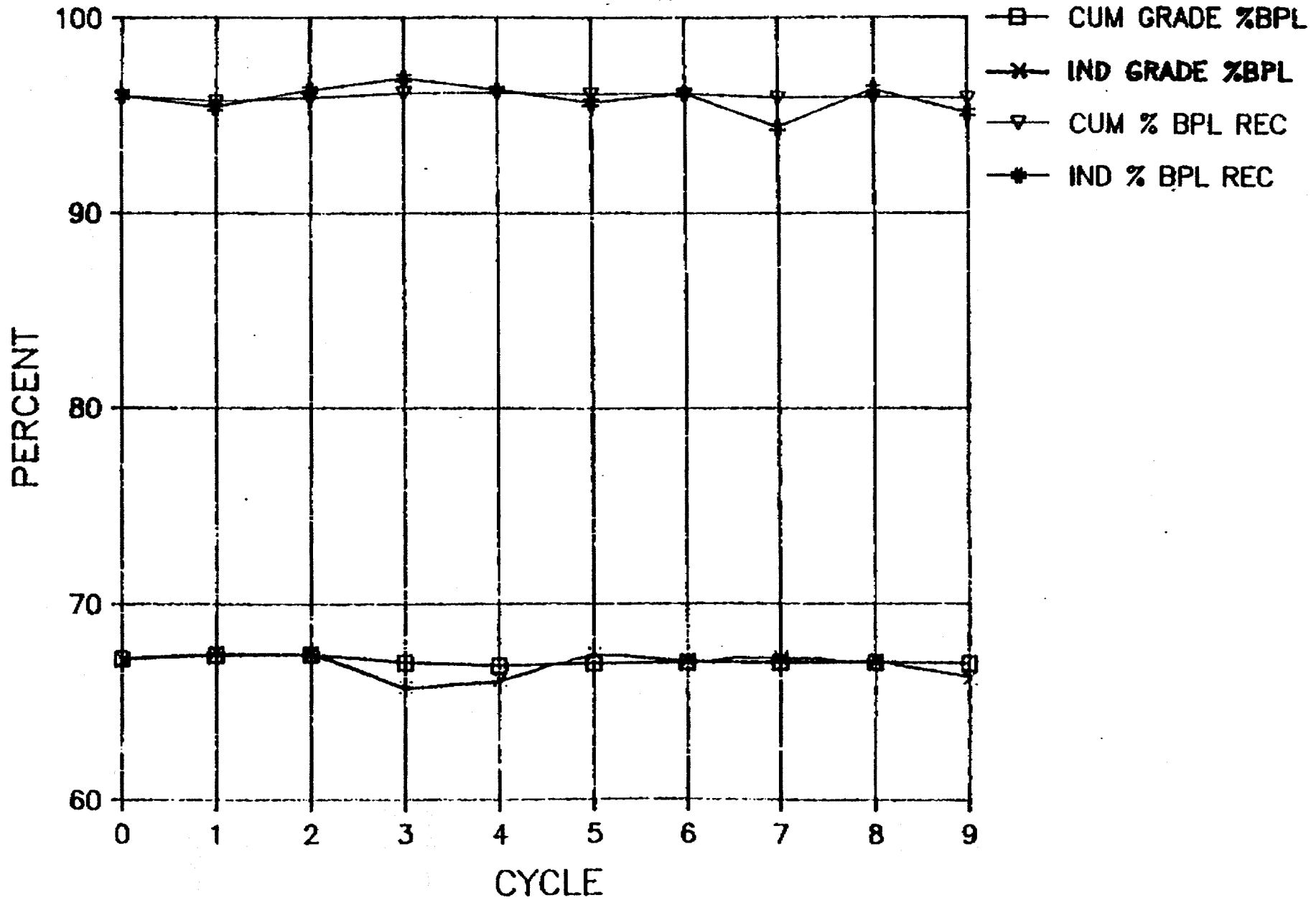
	MINUTES	%SOLIDS	PH
RGH CONDITIONING	2.0	68.0	8.70
RGH FLOTATION	1.4	20.0	8.17
ACID SCRUB	3.0	65.0	4.12
AMINE FLOTATION	1.1	8.4	7.66

***** REAGENTS *****

	DOSE (ML)	GRAMS/LITER	FEED	LB/TON CONC
R-2	.40	920.0	.73	3.75
NO. 5 FUEL OIL	.24	920.0	.44	2.25
NAOH	1.20	50.0	.12	.61
H2SO4	13.00	50.0	1.29	6.62
CUSTAMINE 710	1.60	50.0	.16	.82

FIG. 5-2

TASK 5: PRESENT FEED LOCKED CYCLE SERIES 2



5.9 Flotation of Feed Fractions (Task 5A)

Task 5A was authorized by contract Amendment No. 1. The purpose of this work was to address the flotation of coarse particles which is a priority item for beneficiation research sponsored by FIPR.

The additional work was inserted into the program after Task 5 so that the information gained from previous testing could be applied to the anionic rougher-cleaner flotation of feeds of different particle size consist. The test program consists of:

- (a) Flotation of feed fraction - The anionic collector found to give the best flotation performance at the lowest cost for present feed in Task 5 was tested on three feed fractions. The present feed was prepared as 14 x 150 mesh (unsized feed), and as 14 x 35 mesh (coarse feed) and 35 x 150 mesh (fine feed).

The anionic rougher-cleaner process developed in Tasks 4 and 5 was tested on each feed fraction, using a 1/16 replicate of a seven factor experimental design (Box-Wilson). The design consisted of eight rougher-cleaner tests and six more tests to seek the optimum performance for each feed fraction. The variables examined, and their base levels, were determined from the results of Task 5.

- (b) The Crago flotation process was tested on each feed fraction with a series of six tests. Test conditions were determined from the results of Crago flotation tests in Task 5 and modified as appropriate.

5.9.1 Unsize Feed

The Box-Wilson experimental test design for collector R2 on present 14 x 150 mesh feed is presented in Table 5-24 and the statistical design analysis is presented in Appendix 'B'. Results from the eight tests (5AF12-5AF19) are summarized in Table 5-25. Test optimization designs for collector R2 on

Table 5-24

Test Design
 Collector R2 - Present Feed
 (Task 5A Anionic - 14 x 150 Feed)

<u>Test</u>		<u>Conditioning</u>			<u>Cell Size</u>	<u>Collector</u>	<u>Collector to</u>	<u>RPM</u>	
<u>No.</u>	<u>Sequence</u>	<u>Time(min)</u>	<u>% Solids</u>	<u>Ratio⁽¹⁾</u>	<u>(liters)</u>	<u>(lb/T)</u>	<u>FO⁽²⁾ Ratio</u>	<u>Cell</u>	<u>Conditioner</u>
5AF19	8	2	68	3.50	3	0.8	1:1.0	1200	700
5AF14	3	3	72	3.50	3	1.0	1:1.5	1200	700
5AF17	6	3	68	3.90	3	1.0	1:1.0	1400	700
5AF13	2	2	72	3.90	3	0.8	1:1.5	1400	700
5AF18	7	3	68	3.50	5	0.8	1:1.5	1400	700
5AF15	4	2	72	3.50	5	1.0	1:1.0	1400	700
5AF16	5	2	68	3.90	5	1.0	1:1.5	1200	700
5AF12	1	3	72	3.90	5	0.8	1:1.0	1200	700

(1) NaOH to collector ratio

(2) No. 5 fuel oil

Table 5-25

Test Results Summary
Anionic and Crago Tests on Present 14 x 150 Feed
(Task 5A)

Test	Concentrate		% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾						
	%BPL ⁽¹⁾	%AI ⁽²⁾	Wt. ⁽³⁾	BPL	AI	R2	FO ⁽⁶⁾	NaOH	H ₂ SO ₄	Amine	Kerosene	
5AF12	66.47	8.10	22.45	65.20	2.69	0.82	0.82	0.17				
5AF13	65.18	10.30	27.68	76.56	4.27	0.81	1.21	0.17				
5AF14	64.53	11.05	29.80	81.95	4.91	1.00	1.49	0.19				
5AF15	63.80	11.94	27.80	75.90	4.96	1.01	1.01	0.19				
5AF16	64.90	9.47	28.65	81.15	4.05	1.00	1.49	0.21				
5AF17	61.22	15.42	28.67	74.88	6.61	1.00	1.00	0.21				
5AF18	64.99	6.90	28.58	82.35	2.95	0.83	1.24	0.15				
5AF19	65.82	8.93	23.09	65.68	3.05	0.80	0.80	0.15				
5AF36	66.76	7.49	32.70	90.80	3.70	1.00	1.70	0.15				
5AF37	67.22	7.28	29.66	82.72	3.29	1.06	2.01	0.15				
5AF38	67.59	7.39	31.45	87.75	3.53	1.11	2.32	0.15				
5AF39	66.21	8.48	34.13	93.77	4.40	1.11	1.88	0.15				
5AF40	66.96	8.11	27.93	77.80	3.43	1.10	1.66	0.15				
5AF41	67.03	7.08	31.50	88.82	3.35	1.15	1.96	0.15				
5AF52	70.59	3.67	30.91	93.79	1.68	1.10	1.87	0.15	2.00	0.25		.00
5AF53	69.85	4.10	31.28	94.47	1.90	1.11	1.88	0.15	2.01	0.20		.00
5AF54	68.94	5.45	31.95	94.67	2.58	1.11	1.88	0.15	2.02	0.15		.00
5AF55	70.04	3.80	30.04	91.11	1.68	1.10	1.87	0.15	2.00	0.25		0.06
5AF56	70.04	4.11	30.98	94.19	1.88	1.10	1.88	0.15	2.01	0.20		0.05
5AF57	69.21	5.02	30.91	92.41	2.29	1.10	1.87	0.15	2.01	0.15		0.04

(1) Bone phosphate of lime Ca₃(PO₄)₂

(2) Acid insoluble material

(3) 100 (concentrate weight)/(feed weight)

(4) 100 (concentrate units)/(feed units)

(5) Pounds reagent per short ton of feed

(6) No. 5 fuel oil

present 14 x 150 mesh feed using the anionic rougher-cleaner process (test 5AF36-5AF41) and the Crago process (test 5AF52-5AF57) are presented in Tables 5-26 and 5-27, respectively. Results from the optimization tests are summarized in Table 5-25. The best performance for collector R2 on present 14 x 150 mesh feed for both processes is summarized below.

<u>Test</u>	<u>Process</u>	<u>Concentrate</u>		<u>% Recovery</u>		
		<u>% BPL</u>	<u>% AI</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>
5AF39	Anionic	66.21	8.48	34.13	93.77	4.40
5AF56	Crago	70.04	4.11	30.98	94.19	1.88

Laboratory report sheets for collector R2 on present -14 + 150 mesh feed are presented in Volume II of this report.

5.9.2 Coarse Feed

The Box-Wilson experimental test design for collector R2 on present 14 x 35 mesh feed is presented in Table 5-28 and the statistical design analysis is presented in Appendix 'B'. Results from the eight tests (5AF20-5AF27) are summarized in Table 5-29. Test optimization designs for collector R2 on present 14 x 35 mesh feed using the rougher-cleaner process (test 5AF42-5AF46) and the Crago process (test 5AF58-5AF61) are presented in Tables 5-30 and 5-31, respectively. Results from the optimization tests are summarized in Table 5-29. The best performance for collector R2 on present 14 x 35 mesh feed for both processes is summarized below.

<u>Test</u>	<u>Process</u>	<u>Concentrate</u>		<u>% Recovery</u>		
		<u>% BPL</u>	<u>% AI</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>
5AF45	Anionic	67.43	6.22	57.36	96.96	8.09
5AF60	Crago	68.92	4.70	53.57	98.18	5.29

Laboratory report sheets for collector R2 on present 14 x 150 mesh feed are presented in Volume II of this report.

Table 5-26

Test Design
 Collector R2 - Present Feed
 (Task 5A Anionic Optimization - 14 x 150 Feed)

<u>No.</u>	<u>Conditioning</u>			<u>Cell Size</u> <u>(liter)</u>	<u>Collector</u> <u>(lb/T)</u>	<u>Collector to</u> <u>FO⁽¹⁾ Ratio</u>	<u>Cell</u> <u>RPM</u>	<u>Cleaning</u> <u>Stages</u>
	<u>Time(min)</u>	<u>% Solids</u>	<u>pH</u>					
5AF36	3	68	8.75	5	1.00	1:1.7	1200	1
5AF37	3	68	8.65	5	1.05	1:1.9	1200	1
5AF38	3	68	8.65	5	1.10	1:2.1	1200	1
5AF39	3	68	8.40	5	1.10	1:1.7	1200	1
5AF40	3	68	8.45	5	1.10	1:1.5	1200	1
5AF41	3	68	8.40	5	1.15	1:1.7	1200	2

(1) No. 5 fuel oil

Table 5-27

Test Design
 Collector R2 - Present Feed
 (Task 5A Crago Optimization - 14 x 150 Feed)

Test	Conditioning			Cell Size (liters)	Collector (lb/T)	Collector to Cell FO ⁽¹⁾ Ratio	Cell RPM	Acid Scrub			Amine	
	Time(min)	% Solids	pH					Time(min)	% Solids	H ₂ SO ₄ ⁽²⁾	(lb/T)	Kerosene ⁽³⁾
5AF52	3	68	8.5	5	1.1	1:1.7	1200	3	65	2.0	25	no
5AF53	3	68	8.5	5	1.1	1:1.7	1200	3	65	2.0	20	no
5AF54	3	68	8.6	5	1.1	1:1.7	1200	3	65	2.0	.15	no
5AF55	3	68	8.6	5	1.1	1:1.7	1200	3	65	2.0	.25	yes
5AF56	3	68	8.5	5	1.1	1:1.7	1200	3	65	2.0	.20	yes
5AF57	3	68	8.4	5	1.1	1:1.7	1200	3	65	2.0	.15	yes

5-43

- (1) No. 5 fuel oil
- (2) lb H₂SO₄/ton rougher feed
- (3) Amine/kerosene ratio = 4/1

Comments: (a) Optimum rougher based on test 5AF39

Table 5-28

Test Design
 Collector R2 - Present Feed
 (Task 5A Anionic - 14 x 35 Feed)

Test		Conditioning			Cell Size	Collector	Collector to	Cell	Conditioner
No	Sequence	Time(min)	% Solids	Ratio ⁽¹⁾	(liters)	(lb/T)	FO ⁽²⁾ Ratio	RPM	RPM ⁽³⁾
5AF23	4	2	68	2.0	3	1.0	1:1.5	1200	850
5AF26	7	3	72	2.0	3	1.4	1:2.0	1200	850
5AF20	1	3	68	2.7	3	1.4	1:1.5	1400	850
5AF25	6	2	72	2.7	3	1.0	1:2.0	1400	850
5AF24	5	3	68	2.0	5	1.0	1:2.0	1400	850
5AF27	8	2	72	2.0	5	1.4	1:1.5	1400	850
5AF21	2	2	68	2.7	5	1.4	1:2.0	1200	850
5AF22	3	3	72	2.7	5	1.0	1:1.5	1200	850

(1) Ratio of NaOH to collector

(2) No. 5 fuel oil

(3) To suspend coarse material

Table 5-29

Test Results Summary
Anionic and Crago Tests on Present 14 x 35 Feed
(Task 5A)

Test	Concentrate		% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾					
	%BPL ⁽¹⁾	%AI ⁽²⁾	Wt. ⁽³⁾	BPL	AI	R2	FO ⁽⁶⁾	NaOH	H ₂ SO ₄	Amine	Kerosene
5AF20	67.15	7.19	52.70	93.14	8.01	1.43	2.15	0.21			
5AF21	67.61	6.55	54.38	96.77	7.51	1.36	2.73	0.20			
5AF22	71.57	2.44	3.41	6.33	0.18	1.02	1.53	0.15			
5AF23	68.62	4.76	45.81	82.62	4.64	0.99	1.49	0.11			
5AF24	68.44	5.02	50.23	91.06	5.32	1.02	2.04	0.11			
5AF25	69.36	4.27	35.56	65.58	3.19	1.02	2.04	0.15			
5AF26	68.72	4.89	50.02	90.37	5.20	1.40	2.81	0.15			
5AF27	67.80	6.02	53.11	94.25	6.85	1.40	2.10	0.15			
5AF42	67.98	5.48	56.06	95.39	6.95	1.39	2.09	0.15			
5AF43	68.16	5.11	55.67	95.32	6.42	1.40	2.46	0.15			
5AF44	67.98	5.42	56.04	95.37	6.89	1.39	2.79	0.15			
5AF45	67.43	6.22	57.36	96.96	8.09	1.40	2.10	0.21			
5AF46	67.33	6.42	58.02	96.30	8.44	1.41	2.44	0.21			
5AF58	68.28	4.75	53.51	98.03	5.34	1.42	2.13	0.21	3.04	0.35	.00
5AF59	69.10	4.77	53.51	98.24	5.34	1.42	2.13	0.21	3.05	0.36	0.09
5AF60	68.92	4.70	53.57	98.18	5.29	1.41	2.11	0.21	3.02	0.40	.00
5AF61	66.63	6.28	54.25	98.43	7.10	1.42	2.14	0.21	3.05	0.31	.00

- (1) Bone phosphate of lime Ca₃(PO₄)₂
- (2) Acid insoluble material
- (3) 100 (concentrate weight)/(feed weight)
- (4) 100 (concentrate units)/(feed units)
- (5) Pounds reagent per short ton of feed
- (6) No. 5 fuel oil

Table 5-30

**Test Design
Collector R2 - Present Feed
(Task 5A Anionic Optimization - 14x35 Feed)**

<u>Test</u>	<u>Conditioning</u>			<u>Cell Size (liter)</u>	<u>Collector (lb/T)</u>	<u>Collector to FO⁽²⁾ Ratio</u>	<u>Cell RPM</u>	<u>Cleaning Stages</u>
	<u>Time(min)</u>	<u>% Solids</u>	<u>Ratio⁽¹⁾</u>					
5AF42	2	68	2.0	5	1.4	1:1.50	1400	1
5AF43	2	68	2.0	5	1.4	1:1.75	1400	1
5AF44	2	68	2.0	5	1.4	1:2.00	1400	1
5AF45	2	68	2.7	5	1.4	1:1.50	1400	1
5AF46	2	68	2.7	5	1.4	1:1.75	1400	1

(1) Ratio of NaOH to collector

(2) No. 5 fuel oil

Table 5-31

Test Design
 Collector R2 - Present Feed
 (Task 5A Crago Optimization - 14 x 35 Feed)

Test	Conditioning			Cell Size (liters)	Collector (lb/T)	Collector to FO ⁽²⁾ Ratio	Cell RPM	Acid Scrub			Amine	
	Time(min)	% Solids	Ratio ⁽¹⁾					Time(min)	% Solids	H ₂ SO ₄ ⁽³⁾	(lb/T)	Kerosene ⁽⁴⁾
5AF58	2	68	2.7	5	1.4	1:1.50	1400	3	65	3.0	0.35	no
5AF59	2	68	2.7	5	1.4	1:1.50	1400	3	65	3.0	0.35	yes
5AF60	2	68	2.7	5	1.4	1:1.50	1400	3	65	3.0	0.40	no
5AF61	2	68	2.7	5	1.4	1:1.50	1400	3	65	3.0	0.30	no

- (1) Ratio of NaOH to collector
- (2) No. 5 fuel oil
- (3) lb H₂SO₄/ton rougher feed
- (4) Amine/kerosene = 4/1

Comments: (a) Optimum rougher based on test 5AF45
 (b) Conditioner RPM = 850

5.9.3 Fine Feed

The Box-Wilson experimental test design for collector R2 on present 35 x 150 mesh feed is presented in Table 5-32 and the statistical design analysis is presented in Appendix 'B'.

Results from the eight tests (5AF28-5AF35) are summarized in Table 5-33. Test optimization designs for collector R2 on present 35 x 150 mesh feed using the rougher-cleaner process (test 5AF47-5AF51) and the Crago process (test 5AF62-5AF65) are presented in Tables 5-34 and 5-35, respectively. Results from the optimization tests are summarized in Table 5-33. The best performance for collector R2 on present 35 x 150 mesh feed for both processes is summarized below.

<u>Test</u>	<u>Process</u>	<u>Concentrate</u>		<u>% Recovery</u>		
		<u>% BPL</u>	<u>% AI</u>	<u>Weight</u>	<u>BPL</u>	<u>AI</u>
5AF48	Anionic	68.00	7.44	18.30	96.69	1.66
5AF64	Crago	70.20	3.40	17.31	95.56	0.72

Laboratory report sheets for collector R2 on present -35 + 150 mesh feed are presented in Volume II of this report.

5.10 **Discussion of Results**

For present feed, collectors R1 and R2 gave similar optimum recovery and grade using the anionic rougher-cleaner process. For future feed, collector R2 gave better optimum performance than collector R4 using the anionic rougher-cleaner process. Collector R2 gave about 6 percent higher BPL recovery with a 3.2 percent lower concentrate grade. Testwork using collector R4 was inconclusive due to the aforementioned reagent stability problems.

Table 5-32

Test Design
 Collector R2 - Present Feed
 (Task 5A Anionic - 35 x 150 Feed)

Test		Conditioning			Cell Size	Collector	Collector to	Cell	Conditioner
No.	Sequence	Time(min)	% Solids	Ratio ⁽¹⁾	(liters)	(lb/T)	FO ⁽²⁾ Ratio	RPM	RPM
5AF30	3	2	68	3.2	3	0.7	1:0.6	1200	700
5AF31	4	3	72	3.2	3	0.8	1:1.0	1200	700
5AF28	1	3	68	3.5	3	0.8	1:0.6	1400	700
5AF35	8	2	72	3.5	3	0.7	1:1.0	1400	700
5AF34	7	3	68	3.2	5	0.7	1:1.0	1400	700
5AF33	6	2	72	3.2	5	0.8	1:0.6	1400	700
5AF29	2	2	68	3.5	5	0.8	1:1.0	1200	700
5AF52	5	3	72	3.5	5	0.7	1:0.6	1200	700

(1) Ratio of NaOH to collector

(2) No 5 fuel oil

Table 5-33

Test Results Summary
Anionic and Crago Tests on Present 35 x 150 Feed
(Task 5A)

Test	Concentrate		% Recovery ⁽⁴⁾			Reagent, lb/T ⁽⁵⁾						
	%BPL ⁽¹⁾	%AI ⁽²⁾	Wt. ⁽³⁾	BPL	AI	R2	FO ⁽⁶⁾	NaOH	H ₂ SO ₄	Amine	Kerosene	
5AF28	62.84	13.24	19.66	95.28	3.18	0.81	0.49	0.15				
5AF29	66.62	8.69	18.68	95.26	2.01	0.81	0.81	0.15				
5AF30	66.44	8.64	18.53	94.79	1.97	0.70	0.42	0.12				
5AF31	64.19	12.11	19.57	95.78	2.90	0.81	0.81	0.14				
5AF32	68.42	6.21	17.92	94.08	1.38	0.70	0.42	0.13				
5AF33	66.71	10.12	19.13	95.83	2.37	0.80	0.48	0.14				
5AF34	65.63	9.19	19.11	95.76	2.15	0.70	0.70	0.12				
5AF35	63.38	12.03	19.64	95.72	2.89	0.70	0.70	0.12				
5AF47	66.79	8.31	18.47	95.75	1.87	0.74	0.44	0.12				
5AF48	68.00	7.44	18.30	96.69	1.66	0.70	0.42	0.11				
5AF49	68.18	7.13	17.92	95.35	1.55	0.66	0.40	0.11				
5AF50	67.72	8.07	18.38	96.31	1.81	0.74	0.37	0.12				
5AF51	69.00	6.95	17.69	94.63	1.50	0.70	0.35	0.11				
5AF62	71.66	2.20	16.68	95.93	0.45	0.70	0.42	0.11	1.30	0.20		.00
5AF63	70.65	2.87	17.17	95.33	0.60	0.70	0.42	0.11	1.30	0.17		.00
5AF64	70.20	3.40	17.31	95.56	0.72	0.70	0.42	0.11	1.30	0.15		.00
5AF65	70.10	3.77	17.19	95.25	0.79	0.70	0.42	0.11	1.31	0.15		0.04

- Notes: (1) Bone phosphate of lime $\text{Ca}_3(\text{PO}_4)_2$
(2) Acid insoluble material
(3) $100 (\text{concentrate weight}) / (\text{feed weight})$
(4) $100 (\text{concentrate units}) / (\text{feed units})$
(5) Pounds reagent per short ton of feed
(6) No. 5 fuel oil

Table 5-34

Test Design
 Collector R2 - Present Feed
 (Task 5A Anionic Optimization - 35 x 150 feed)

Test	Conditioning			Cell Size (liters)	Collector (lb/T)	Collector to FO ⁽²⁾ Ratio	Cell RPM	Cleaning Stages
	Time(min)	% Solids	Ratio ⁽¹⁾					
5AF47	2	68	3.0	5	0.74	1:0.6	1200	1
5AF48	2	68	3.0	5	0.70	1:0.6	1200	1
5AF49	2	68	3.0	5	0.66	1:0.6	1200	1
5AF50	2	72	3.0	5	0.74	1:0.5	1200	1
5AF51	2	72	3.0	5	0.70	1:0.5	1200	1

(1) Ratio of NaOH to collector

(2) No. 5 fuel oil

Table 5-35

Test Design
 Collector R2 - Present Feed
 (Task 5A Crago Optimization - 35 x 150 Feed)

Test	Conditioning			Cell Size (liters)	Collector (lb/T)	Collector to FO ⁽²⁾ Ratio	Cell RPM	Acid Scrub			Amine (lb/T)	Kerosene ⁽⁴⁾
	Time(min)	% Solids	Ratio ⁽¹⁾					Time(min)	% Solids	H ₂ SO ₄ ⁽³⁾		
5AF62	2	68	3.0	5	0.7	1:0.6	1200	3	65	1.3	0.20	no
5AF63	2	68	3.0	5	0.7	1:0.6	1200	3	65	1.3	0.17	no
5AF64	2	68	3.0	5	0.7	1:0.6	1200	3	65	1.3	0.15	no
5AF65	2	68	3.0	5	0.7	1:0.6	1200	3	65	1.3	0.15	yes

- (1) Ratio of NaOH to collector
- (2) No. 5 fuel oil
- (3) lb H₂SO₄/ton rougher feed
- (4) Amine/kerosene ratio = 4/1

Comments: (a) Optimum rougher based on test 5AF

For each feed/reagent combination tested in Task 5, an optimum performance was established independent of testing other feed/reagent combinations. A performance comparison between the anionic rougher-cleaner and the Crago processes based on optimum tests is not straightforward because the flotation conditions, although optimized, were not necessarily comparable with respect to anionic reagent consumption. Anionic rougher-cleaner and Crago process comparisons are discussed in Section 6.

Bench scale locked cycle tests were conducted to evaluate the effect of adding the cleaner tailings to the subsequent test conditioner feed. Two series of tests that examined tailings recycle for 28 x 150 mesh present feed were presented in Section 5.8. The second test series was conducted in parallel to four Crago tests. Comparative results, as given below, show about the same performance with tailings recycle as was obtained without recycle.

Process Comparison

	<u>Anionic</u> ⁽¹⁾	<u>Crago</u> ⁽²⁾
Flotation Concentrate		
% BPL	66.9	70.6
% AI	8.0	3.1
Yield - % weight	21.0	19.6
BPL Distribution		
Rougher Tailings	4.2	4.5
Cleaner Tailings	0	1.8
Final Concentrate	95.8	93.7
Reagent Consumption		
R2	0.73	0.73
No. 5 fuel oil	0.44	0.44
NaOH	0.12	0.12
Amine	0	1.29
Kerosene	0	0.16

(1) averaged from 9 locked cycle tests (Lock Cycle Test No. 2)

(2) averaged from 4 tests (Locked Cycle Test No. 2 - Intermediate Crago Tests)

With tailings recycle, the anionic rougher-cleaner process gave a 2.1 percent BPL recovery improvement over the Crago process. Without tailings recycle, the BPL recovery improvement was 1.8 percent.

The purpose of Task 5A was to address the flotation of unsized (14 x 150 mesh), coarse (14 x 35 mesh), and fine (35 x 150 mesh) feed fractions using the anionic rougher-cleaner and the Crago processes. The results of the testwork are presented in Table 5-36. The “reconstituted” 14 x 150 mesh results were calculated from the weight distribution and flotation data from the 14 x 35 and 35 x 150 mesh feeds. The calculation was performed to compare the overall performance of sized flotation versus unsized (bulk) flotation.

In Task 5A, most of the test designs incorporated a caustic to collector ratio instead of separate caustic and collector dosages. This was done to minimize the interaction between caustic, collector and fuel oil.

Comparative results, as shown in Table 5-36, show that flotation of sized feed fractions using the anionic rougher-cleaner process gives a 3.0 percent BPL recovery increase and a 1.5 BPL concentrate grade improvement over unsized feed flotation. The Crago process demonstrated a similar recovery advantage for sized feed flotation, however, the concentrate grade was lower by 0.4 BPL for sized flotation.

Relative to the Crago process, anionic rougher-cleaner flotations gave lower grade concentrates and lower reagent consumption for all feeds. The BPL recovery was similar for both processes.

Table 5-36

**Bench Scale Process Comparison
Present Feed**

	<u>14 x 35 Mesh</u>		<u>35 x 150 Mesh</u>		<u>Reconstituted 14 x 150 Mesh</u>		<u>Actual 14 x 150 Mesh</u>	
	<u>Anionic</u>	<u>Crago</u>	<u>Anionic</u>	<u>Crago</u>	<u>Anionic</u>	<u>Crago</u>	<u>Anionic</u>	<u>Crago</u>
Flotation Feed								
% BPL	39.9	37.6	12.9	12.7	24.3	23.3	24.1	23.0
% AI	44.1	47.6	82.1	82.0	65.9	67.4	65.8	67.8
Weight	42.5	42.5	57.5	100.0	100.0	100.0	100.0	100.0
Flotation Concentrate								
% BPL	67.4	68.9	68.0	70.2	67.7	69.6	66.2	70.0
% AI	6.2	4.7	7.4	3.4	6.9	4.0	8.5	4.1
Yield, % weight	57.4	53.6	18.3	17.3	34.9	32.7	34.1	31.0
BPL Distribution in Products								
Rougher Tailings	1.8	1.5	3.1	3.2	2.5	2.5	5.5	4.1
Cleaner Tailings	1.2	0.3	0.2	1.3	0.6	0.9	0.7	1.7
Final Concentrate	97.0	98.2	96.7	95.5	96.8	96.6	93.8	94.2
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Reagent Consumption (lb/T feed)								
Anionic Collector (R2)	1.40	1.41	0.70	0.70	1.00	1.00	1.11	1.10
Anionic Extender (No. 5 FO)	2.10	2.11	0.42	0.42	1.13	1.14	1.88	1.88
NaOH	0.21	0.21	0.11	0.11	0.15	0.15	0.15	0.15
H ₂ SO ₄	.00	3.02	.00	1.30	.00	2.03	.00	2.01
Cationic Collector	.00	0.40	.00	0.15	.00	0.26	.00	0.20
Cationic Extender	.00	.00	.00	.00	.00	.00	.00	0.05
Test Identification	5AF45	5AF60	5AF48	5AF64			5AF39	5AF56

SECTION 6

ROUGHER-CLEANER PROCESS VS. CRAGO PROCESS

6.1 Background

Phosphate rock produced in Florida is mostly converted to fertilizer products via the manufacture of wet process phosphoric acid. Triple superphosphate (TSP) is produced by reacting high grade phosphate rock with phosphoric acid. The more popular granular ammonium phosphates (DAP and MAP) are produced by reacting ammonia with phosphoric acid.

Phosphate rock of high BPL content may be used advantageously to:

- o produce TSP
- o reduce shipping costs (per unit of BPL)
- o blend off low grade phosphate rock.

Few high grade pebble deposits remain and high grade rock (+68% BPL) is typically obtained as a flotation concentrate. The use of flotation concentrate to produce phosphoric acid in Florida has advantages that must be balanced against the costs associated with flotation.

The cost of phosphate rock and sulfuric acid are major contributors to the cost of phosphoric acid. The following comparisons of anionic rougher-cleaner flotation to Crago flotation consider process differences as well as the potential impact of the concentrates on phosphoric acid manufacture by the wet process.

6.2 Concentrate Grade

The objective of the two flotation processes is to separate acid insoluble material (quartz) from the phosphate. A low acid insoluble content is necessary to obtain the BPL concentration required for TSP manufacture. In phosphoric acid manufacture, the insoluble material increases solids loading

on the gypsum filter and abrasion; however, quantification of these minor effects for acid insoluble contents of 4 to 12 percent requires controlled testing.

Detailed chemical analyses of concentrates produced from present and future feed by the two flotation processes are shown on Table 6.1. Acid insoluble (AI) analyses are markedly higher for the anionic concentrates. The reported oxides of calcium, magnesium, iron, and aluminum are indicative of the primary contaminants effecting rock quality. The ratios of these metal oxides to P_2O_5 are essentially identical for the anionic and Crago concentrates obtained from present and future feed. Except for the acid insoluble content, the concentrates produced by anionic rougher-cleaner flotation are comparable in quality to conventional concentrates.

Nominal criteria for the chemical quality of phosphate rock are listed below:

P_2O_5	28 percent, lower limit
MgO	1 percent upper limit
CaO: P_2O_5	1.6 upper limit
Fe_2O_3 & $Al_2O_3:P_2O_5$	0.10 upper limit

The anionic concentrate from present feed has chemical characteristics suitable for commercial acidulation. Neither of the concentrates from future feed are suitable for commercial acidulation because of excessive quantities of CaO and MgO, and marginal quantities of Fe_2O_3 and Al_2O_3 . Dolomite (a calcium-magnesium carbonate) and ankerite (a calcium-magnesium-iron carbonate) are the root source of the contaminants.

The Florida Institute of Phosphate Research conducted x-ray diffraction analyses of several concentrate samples. The concentrates from present feed contained a mixture of fluorapatite, carbonate fluorapatite, and quartz, while the concentrates from future feed contained these minerals plus dolomite/ankerite. Polarized light microscopy confirmed the mineral identification and found that small quantities of the dolomitic material existed as inclusions in the apatite, even after fine grinding.

Table 6-1
Concentrate Quality Comparison
(28 x 150 mesh flotation feed)

	<u>Present Feed</u>		<u>Future Feed</u>	
	<u>Anionic</u> (1)	<u>Crago</u> (2)	<u>Anionic</u> (3)	<u>Crago</u> (4)
% P ₂ O ₅	30.80	32.40	25.51	26.33
% CaO	45.87	47.61	46.69	47.39
% MgO	0.35	0.37	3.50	3.60
% Al	8.00	2.92	4.93	2.57
% Fe ₂ O ₃	1.19	1.29	1.71	1.83
% Al ₂ O ₃	1.20	1.22	0.80	0.80
% F	3.68	3.85	3.12	3.20
% SO ₄	1.41	1.50	1.16	1.27
CaO:P ₂ O ₅ *	1.463	1.442	1.804	1.772
MgO:P ₂ O ₅	0.0114	0.0114	0.1372	0.1367
Fe ₂ O ₃ & Al ₂ O ₃ :P ₂ O ₅	0.0776	0.0775	0.0984	0.0999

*Corrected for SO₄

	<u>Test No.</u>	<u>Sample No.</u>
(1)	5F65	87-11-19-12
(2)	5F75	87-12-01-06
(3)	5F58	87-11-18-09
(4)	5F78	87-12-02-03

Comparative concentrate grade data for different feed fractions are shown on Table 6.2. On the average, the concentrate analysis is reduced by 2.6 percent BPL and increased by 3.5 percent AI when the anionic rougher-cleaner process is compared to the Crago process.

6.3 Phosphate Recovery

Anionic rougher-cleaner flotation and Crago flotation each yield three process streams; rougher tailings, cleaner tailings, and final concentrate. The distributions of BPL values obtained in the three process streams by the two processes, from present feed fractions and future feed, are compared on Table 6-2.

The averaged distributions from Table 6.2 for the three streams are as follows:

	<u>Average % BPL Distribution</u>	
	<u>Anionic</u>	<u>Crago</u>
rougher tailings	4.1	3.7
cleaner tailings	0.7	1.6
final concentrate	95.2	94.6

Rougher flotation conditions were maintained essentially identical for both processes, therefore the difference between the rougher tailings distributions is not process related. In retrospect, the sequence of testing may have created a minor bias in favor of the Crago tests. Crago tests always followed the rougher-cleaner tests and possibly benefited slightly by the experience gained from the preceding tests.

The cleaner tailings BPL distribution was lower from the anionic rougher-cleaner process for all feeds except for the 14 x 35 mesh. Improved cleaner flotation recovery is the only avenue for the anionic rougher-cleaner process to yield improved recovery over the Crago process. The improvement for the five feeds averaged 0.9 percent, without recycle of the cleaner tailing.

Table 6-2

Bench Scale Process Comparison
(without recycle of anionic cleaner tailings)

	Present Feed								Future Feed	
	28 x 150 mesh		14 x 150 mesh		14 x 35 mesh		35 x 150 mesh		28 x 150 mesh	
	Anionic	Crago	Anionic	Crago	Anionic	Crago	Anionic	Crago	Anionic	Crago
<u>Flotation Feed</u>										
% BPL	14.6	14.2	24.1	23.0	39.9	37.6	12.9	12.7	14.3	14.7
% AI	79.5	80.0	65.9	67.9	44.1	47.6	82.1	82.0	72.8	73.1
<u>Flotation Concentrate</u>										
% BPL	67.3	70.8	66.2	70.0	67.4	68.9	68.0	70.2	55.8	57.7
% AI	8.0	2.9	8.5	4.1	6.2	4.7	7.4	3.4	5.0	2.6
Yield %weight	20.8	18.8	34.1	31.0	57.4	53.6	18.3	17.3	23.8	23.4
<u>BPL Distribution in Products</u>										
Rougher tailings	4.0	3.4	5.5	4.1	1.8	1.5	3.1	3.2	6.3	6.5
Cleaner tailings	0.4	2.8	0.7	1.7	1.2	0.3	0.2	1.3	0.9	2.0
Final concentrate	95.6	93.8	93.8	94.2	97.0	98.2	96.7	95.5	92.8	91.5
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
<u>Reagent Consumption (lb/T)</u>										
Anionic Collector (R2)	0.70	0.71	1.11	1.10	1.40	1.41	0.70	0.70	0.67	0.67
Anionic Extender (No. 5 FO)	0.88	0.67	1.88	1.88	2.10	2.11	0.42	0.42	0.84	0.80
NaOH	0.12	0.07	0.15	0.15	0.21	0.21	0.11	0.11	0.07	0.06
H ₂ SO ₄	0	1.31	0	2.01	0	3.02	0	1.30	0	1.36
Cationic Collector	0	0.22	0	0.20	0	0.40	0	0.15	0	0.23
Cationic Extender	0	0	0	0.05	0	0	0	0	0	0
Test Identification	5F65	5F75	5AF39	5AF56	5AF45	5AF60	5AF48	5AF64	5F58	5F78

The final concentrate obtained from the anionic rougher-cleaner process recovered slightly more of the BPL than the Crago process. If the bias of the rougher tailings were removed, the average recovery increase for the five feeds would be equivalent to the gain in cleaner flotation, or about one percent.

The preceding discussion applies to tests where the anionic cleaner tailing was discarded. The cleaner tailing may be recycled to rougher flotation in the anionic process; however, it is always discarded in the Crago process. Two series of tests that examined tailings recycle for 28 x 150 mesh present feed were presented in Section 5.8. The second test series was conducted in parallel to four Crago tests. Comparative results, as given on Table 6.3, show about the same performance with tailings recycle as was obtained previously without recycle. With tailings recycle, the anionic rougher-cleaner process gave a BPL recovery improvement of 2.1 percent over the Crago process, while without tailings recycle, the BPL recovery improvement for 28 x 150 mesh feed was 1.8 percent.

Tailings recycle does not appear to be necessary for optimized laboratory tests. Flotation results are less favorable in full scale continuous operation and tailings recycle may be more beneficial under plant conditions.

Differences between flowsheets for the anionic rougher-cleaner process and the Crago process may provide additional improvements in concentrate recovery. For example, the cyclones that dewater rougher concentrate and deoil rougher concentrate have some loss of BPL values. Similarly, the acid rinse wash boxes may have some BPL losses. These losses which are associated with the Crago process vary from shift to shift and from plant to plant. For this study, the BPL losses associated with material handling losses in the Crago process acid rinse circuit are estimated at two percent.

The potential BPL recovery improvement of four percent for the anionic rougher-cleaner process results from higher flotation recovery (two percent) and reduced material handling losses (two percent).

Table 6-3

**Bench Scale Process Comparison
(with recycle of anionic cleaner tailings)**

	<u>Anionic</u> ⁽¹⁾	<u>Crago</u> ⁽²⁾
<u>Flotation Feed</u>		
% BPL	14.65	14.72
% AI	79.26	79.05
<u>Flotation Concentrate</u>		
% BPL	66.9	70.6
% AI	8.0	3.1
Yield - % weight	21.0	19.6
<u>BPL Distribution of Products</u>		
Rougher Tailing	4.2	4.5
Cleaner Tailing	0	1.8
Final Concentrate	95.8	93.7
<u>Reagent Consumption (lb/T feed)</u>		
Anionic collector (R2)	.73	.73
Anionic extender (No. 5 FO)	.44	.44
NaOH	.12	.12
Cationic collector	0	1.29
Cationic extender	0	0.16

(1) averaged values from nine locked cycle tests of present 28 x 150 mesh feed.

(2) averaged values from four tests of present 28 x 150 mesh feed.

6.4 Reagent Consumption

The Crago process utilizes the same anionic reagents as the anionic rougher-cleaner process and in addition H_2SO_4 , cationic collector, and sometimes a cationic extender. Reagent costs for the anionic process are reduced by the elimination of H_2SO_4 and the cationic reagent suite. Comparative reagent consumption data for various feed fractions are given on Table 6.2. Similarly, Table 6.3 shows comparative reagent consumption data for 28 x 150 mesh present feed. The resultant reagent costs, shown on Table 6.4, were obtained by summing the products of reagent consumption (lb/T) and reagent unit cost (\$/lb) for each process.

Table 6-4

Bench Scale Reagent Cost Comparison

<u>Present feed fraction</u>	<u>Reagent Cost (\$/T feed)</u>		<u>Savings (\$/T feed)</u>
	<u>Anionic</u>	<u>Crago</u>	
28 x 150 mesh (Table 6.3)	0.099	0.182	0.083
28 x 150 mesh (Table 6.2)	0.121	0.205	0.084
14 x 150 mesh (Table 6.2)	0.216	0.335	0.119
14 x 35 mesh (Table 6.2)	0.259	0.461	0.202
35 x 150 mesh (Table 6.2)	0.095	0.175	0.080
<u>Future feed fraction</u>			
28 x 150 mesh (Table 6.2)	0.108	0.209	0.101

Reagent savings of \$0.08 to \$0.20 per feed ton are realized when the anionic rougher-cleaner process is used. Relative to the Crago process reagent costs, the savings range from 36 to 48 percent.

6.5 Other Factors

The elimination of the acid rinse circuitry would reduce electrical consumption, maintenance costs, process water and well water make-up requirements. The costs of electricity, process water, and well water make-up are developed in Section 7.

SECTION 7

PLANT DESIGN AND OPERATING CONSIDERATIONS

7.1 **Introduction**

7.1.1 Objective

The purpose of examining plant design and plant operation is to establish the preliminary feasibility of phosphate flotation with the anionic rougher-cleaner process.

7.1.2 Methodology

A field investigation of six phosphate plants was undertaken to review the practical considerations of retrofitting these plants to utilize the anionic rougher-cleaner process. The cost of retrofit was estimated from a conceptual engineering study of required modifications for a plant having 1100 tpy feed rate.

A similar study of the net difference between grass-roots plants utilizing the Crago and anionic rougher-cleaner processes was performed. The net difference in capital cost for the two 1100 tph grass-roots plants was estimated.

The impact of the anionic rougher-cleaner process on cash operating costs was examined with the ZW cost model. Cash costs were computed with model input from each process.

A preliminary risk/benefit analysis of the anionic rougher-cleaner process examined beneficiation and also the acidulation of the anionic concentrate.

7.2 Summary

The modifications to permit the adoption of the anionic rougher-cleaner process into a conventional flotation plant, having a nominal capacity of 1100 TPH feed, range in cost from \$30,000 to \$120,000. The lower cost assumes simple piping changes and no recycle of anionic cleaner tailings. The higher cost includes installation of a pump and pump box to recycle cleaner tailings.

From the investigation of six plants, it became apparent that the incorporation of the anionic rougher-cleaner process into an existing plant would be both possible and inexpensive provided that:

- (1) The existing cationic cleaner cells had adequate capacity to function as anionic cleaners.
- (2) No intermediate treatment of the anionic rougher concentrate was required.
- (3) The anionic rougher concentrate could be pumped and refloated in an anionic cleaner circuit without significant recovery losses.

The capital cost saving for a grass-roots plant utilizing the anionic rougher-cleaner process was estimated as 1.3 million dollars. Anionic rougher-cleaner flotation has operating cost impacts due to increased recovery and also lower consumption of reagents and electrical power. The reduction in cash costs amounted to \$2.50 per ton of concentrate.

Additional testing is required to quantify the risks and more accurately quantify the benefits of anionic rougher-cleaner flotation. No environmental risks were identified.

7.3 **Plant Retrofit**

7.3.1 Hypothetical Plant Configuration

The hypothetical flotation plant selected for comparison of the Crago and anionic rougher-cleaner processes has the following characteristics:

o Annual Concentrate Tonnage	2,000,000
o Operating Factor	85%
o Hourly Rougher Feed Rate	1100 tons
- Fine Rougher Feed Rate	800 TPH
- Coarse Rougher Feed Rate	300 TPH
o Hourly Final Concentrate Rate	270 tons
- Fine Concentrate Rate	180 TPH
- Coarse Concentrate Rate	90 TPH
o Overall Ratio of Concentration	4.07

These plant parameters were assigned to provide a basis for equipment selection and comparison of the two flotation processes.

The hypothetical plant was configured with three flowsheets representing common variations of the Crago process. These variations are:

- (1) **Bulk Flotation** - Flotation feed nominally -20 x 150 mesh, is processed in 24 rougher flotation cells. The cells are arranged into three parallel trains, each having two rows of four cells. The rougher concentrates are combined for deoiling and washing. Cleaner circuit consists of three parallel rows of four flotation cells.
- (2) **Separate Rougher Separate Cleaner (SRSC)** - Flotation feed is sized at 35 mesh and the coarse feed is handled in one train having two rows of four cells and the fine feed is handled in two parallel trains each having two rows of four cells. The coarse and fine rougher concentrate are maintained separately throughout deoiling and cleaner flotation.

- (3) **Separate Rougher Combined Cleaner (SRCC)** - Sized feed is processed in the roughers, with one train for coarse feed and two trains for fine feed. However, coarse and fine rougher concentrate are combined for the deoiling and subsequent cleaner flotation.

7.3.2 Process Considerations

The requirements for implementing the anionic rougher-cleaner process at a conventional plant are discussed for each unit operation in the balance of this section.

Anionic Rougher Circuit

The anionic rougher circuit, which includes conditioning and flotation, is identical to the Crago process. The types and capacities of equipment would be unchanged for the anionic rougher-cleaner process except that the recycle of anionic cleaner tailings would affect the tonnage of material processed.

Deoiling and Washing

The bench scale testwork indicated that no intermediate treatment of rougher concentrate is required for the anionic rougher-cleaner process. Since the anionic-rougher cleaner process maintains the coating of anionic reagents for its cleaner flotation step, the deoiling and subsequent removal of spent anionic reagents between the rougher and cleaner flotation steps is not required. To retrofit the anionic rougher-cleaner process into any of the inspected plants, it would be necessary to pump the rougher concentrate to the anionic cleaner circuit. The existing rougher concentrate pump boxes and pumps could be utilized for this purpose. All of the plants visited had deoiling configurations which could be modified by one of the following:

- o Rougher concentrate pumped to the deoiling circuit - for this case the existing rougher concentrate pumps could be connected to the cleaner circuit directly. The inspected plants had adequate space for a relocated rougher concentrate pipeline.

- o Rougher concentrate flows by gravity to the deoiling circuit - in this case an existing deoiling tank could be used as a flow-through device with the impeller taken out of service. If a static deoiler is used, it could be replaced with a pipe spool or operated without sulfuric acid. The pump and pump box which had been used to feed the deoiling cyclones and wash boxes/screw classifiers, could be used to feed the existing cleaner cells.

Cleaner Flotation

The flotation time for the anionic cleaner bench scale flotation testing is equal to or less than the flotation time for cationic cleaner bench scale flotation of equivalent rougher concentrates. Based on bench scale flotation retention times, there appears to be no need for additional cleaner capacity.

The froth discharge of the cationic cleaner will become the product stream in the anionic cleaner and the cationic product discharge will become the anionic cleaner tailings discharge. There appears to be adequate headroom in the plants visited to repipe the product and tailings discharges for the new service. The conversion from cationic to anionic cleaner flotation in continuous full scale operation may encounter limitations not evident from bench scale flotation. For example, assuming a cleaner cell ratio of concentration of 1.3, the froth product would increase from 0.3 to 1.0 on a relative basis. Rate of froth removal may impose a capacity limitation.

Recycle of anionic cleaner tailings in bench scale flotation is not essential to maintain high recovery. This finding may well change in continuous full scale operation. The need for tailings recycle will also be investigated in the proposed pilot testing to be conducted in Phase II.

In the event that anionic cleaner tailings must be recycled, the individual plant configuration will dictate the cost of the modification as follows:

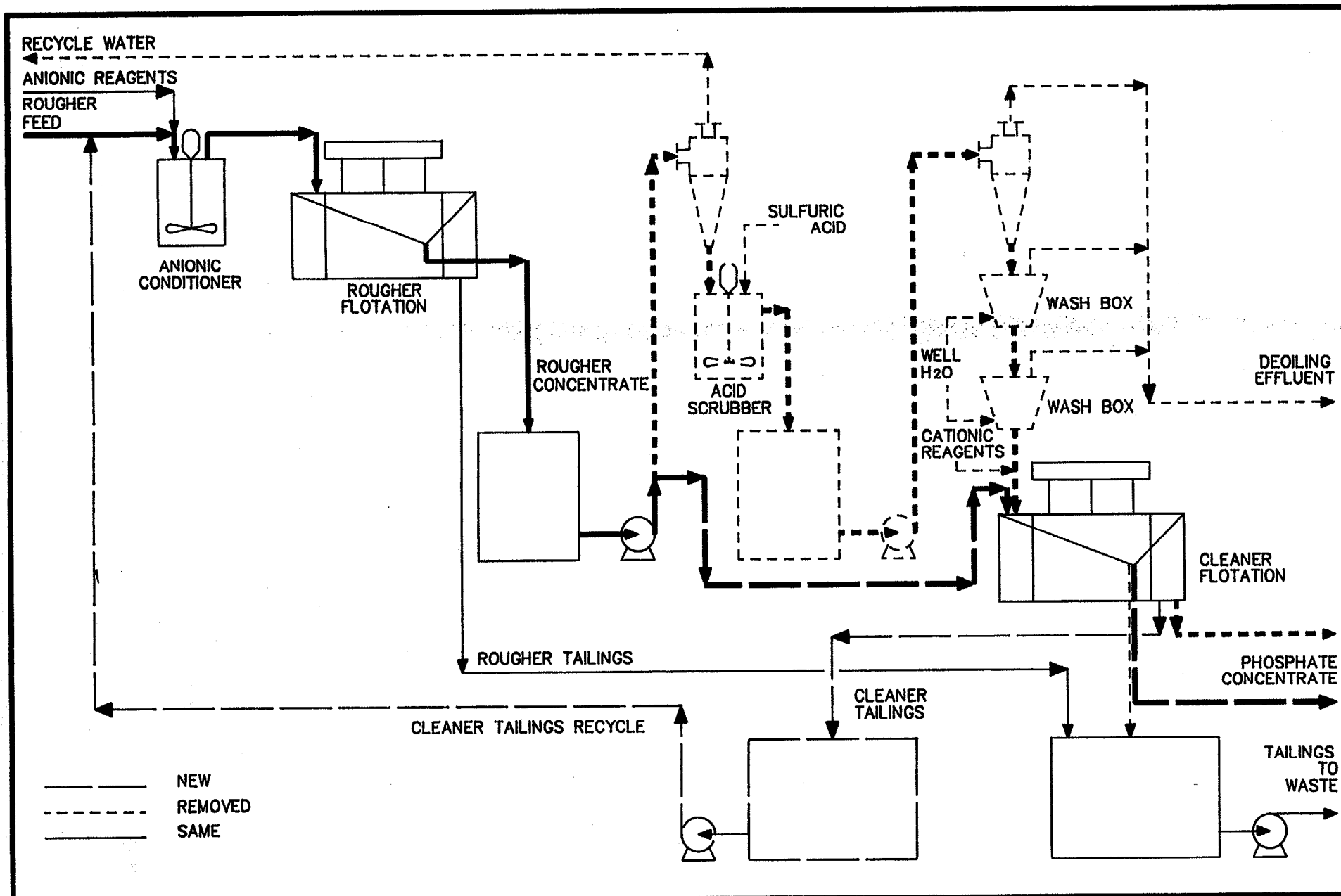
- o Plants where cationic tailings are currently being pumped - after repiping the tailing discharge, reroute the cleaner tailings pipeline to a rougher feed bin. If the plant has separate fine and coarse rougher circuits and a combined cleaner, it will probably be advantageous to direct the recycle stream to the coarse rougher circuit to maximize recovery of coarse particles.
- o Plants in which cationic tailings flow by gravity and are mixed into the general mill tailings - this plant will require a slurry pumping step to return the anionic cleaner tailings to a bin ahead of the rougher flotation circuit.

Product Handling

The presence of residual anionic reagents on the flotation concentrate could impact the dewatering, storage, and loading of phosphate concentrates. At this time, we have not included any new provisions for this eventuality.

7.3.3 Modifications to Existing Plants

The hypothetical flowsheet shown in Figure 7-1 illustrates the modifications required to convert an existing plant to the anionic rougher-cleaner process. As shown, the rougher concentrate pump box and pump are utilized to pump rougher concentrate directly to the cleaner flotation cells. The cleaner cell underflow and launder discharges are repiped and a new pump and pump box are provided to recycle the cleaner tailings to flotation feed bins.



7-7

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CRAGO REVERSE FLOTATION
 MODIFIED TO ANIONIC ROUGHER - CLEANER
 FIGURE 7-1

Table 7-1 presents the estimated cost of retrofitting anionic rougher cleaner flotation into existing Crago plants having nominal 1100 tons per hour flotation feed capacity. The three flowsheet variations described in Section 7.3 and listed below were examined in the study.

- o Bulk Flotation Plant (Bulk)
- o Separate Rougher - Separate Cleaner (SRSC)
- o Separate Rougher - Combined Cleaner (SRCC)

Each plant estimate shows the cost of modifications with and without recycle of the anionic cleaner tailings. In the case of recycle, the estimate allows for a new pump and pump box. No credit was deducted for equipment taken out of service. The modification cost excludes any costs associated with automatic sampler relocation and/or addition.

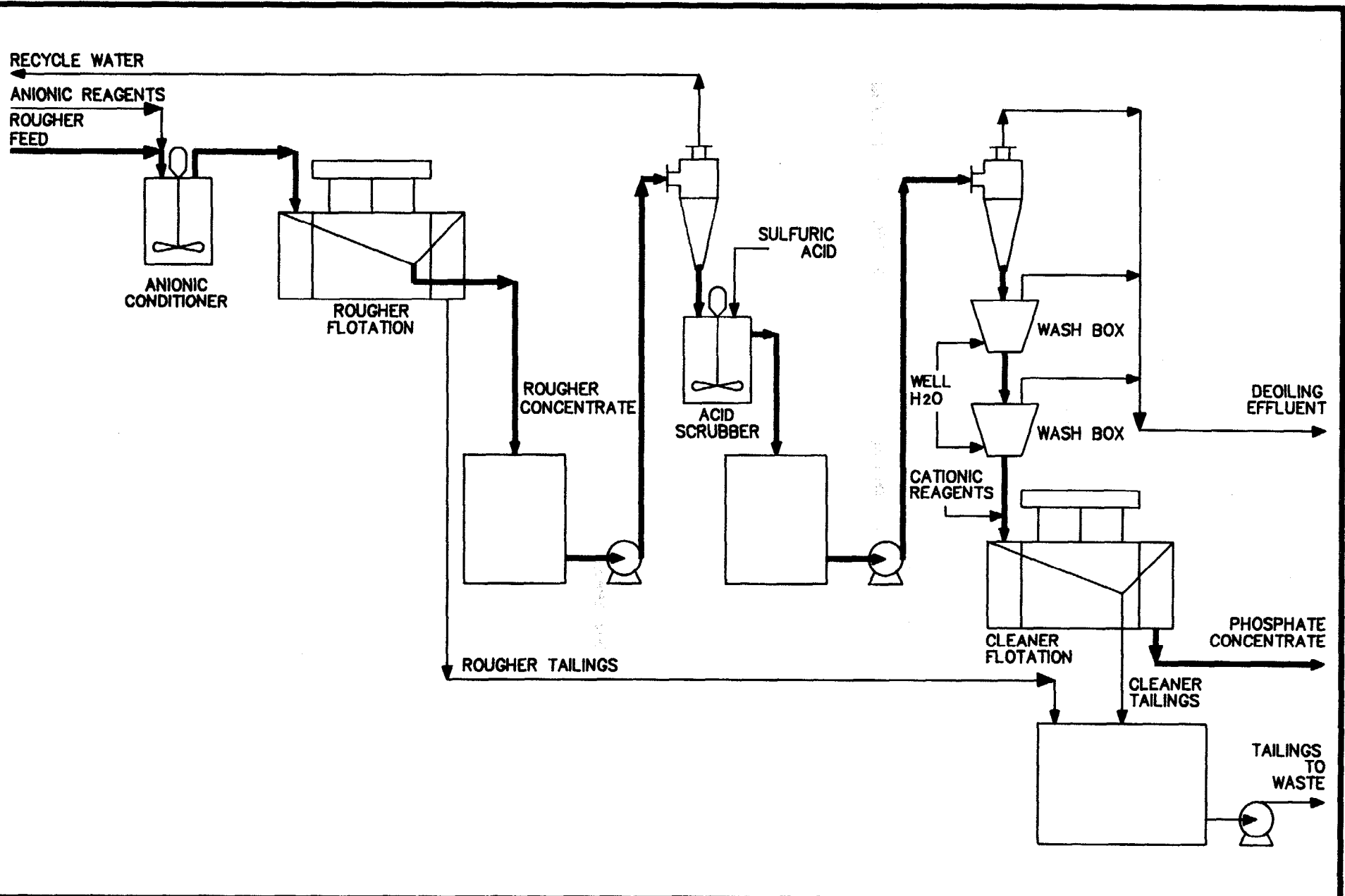
7.4 **Grass-roots Plants**

A grass-roots anionic rougher-cleaner plant will require less capital investment than a Crago plant. The capital savings result from the elimination of several equipment items. Figures 7-2 and 7-3 show the flowsheets for the Crago process and the anionic rougher-cleaner process. A comparison of these flowsheets reveals that the deoiling and washing circuit may be eliminated. Comparable equipment lists for the two processes are given in Appendix 'C'.

An estimate of the total capital costs of a mine and beneficiation plant for each process is beyond the scope of this study. However, estimates based on the comparable equipment lists show a direct capital cost savings of about \$800,000 for the anionic rougher-cleaner process. The estimates of direct capital are presented in Appendix 'C'.

Table 7-1
Estimated Installed Capital Cost of Modifications
(values in dollars)

<u>Circuit</u>	<u>Item</u>	<u>Bulk</u>	<u>SRSC</u>	<u>SCCC</u>
- Anionic Rougher		0	0	0
- Deoiling & Washing				
	Cyclones	0	0	0
	Piping	7,200	13,700	7,200
- Anionic Cleaner				
	Piping	<u>19,000</u>	<u>19,000</u>	<u>19,000</u>
Total w/o Recycle		26,200	32,700	26,200
Tailings Recycle				
	Pump & Pump Box	40,000	60,000	40,000
	Piping	<u>7,100</u>	<u>10,200</u>	<u>7,100</u>
Total with Recycle		73,300	102,900	73,300
Allowance for unforeseen		<u>14,660</u>	<u>20,580</u>	<u>14,660</u>
Estimated Capital Cost		87,960	123,480	87,960



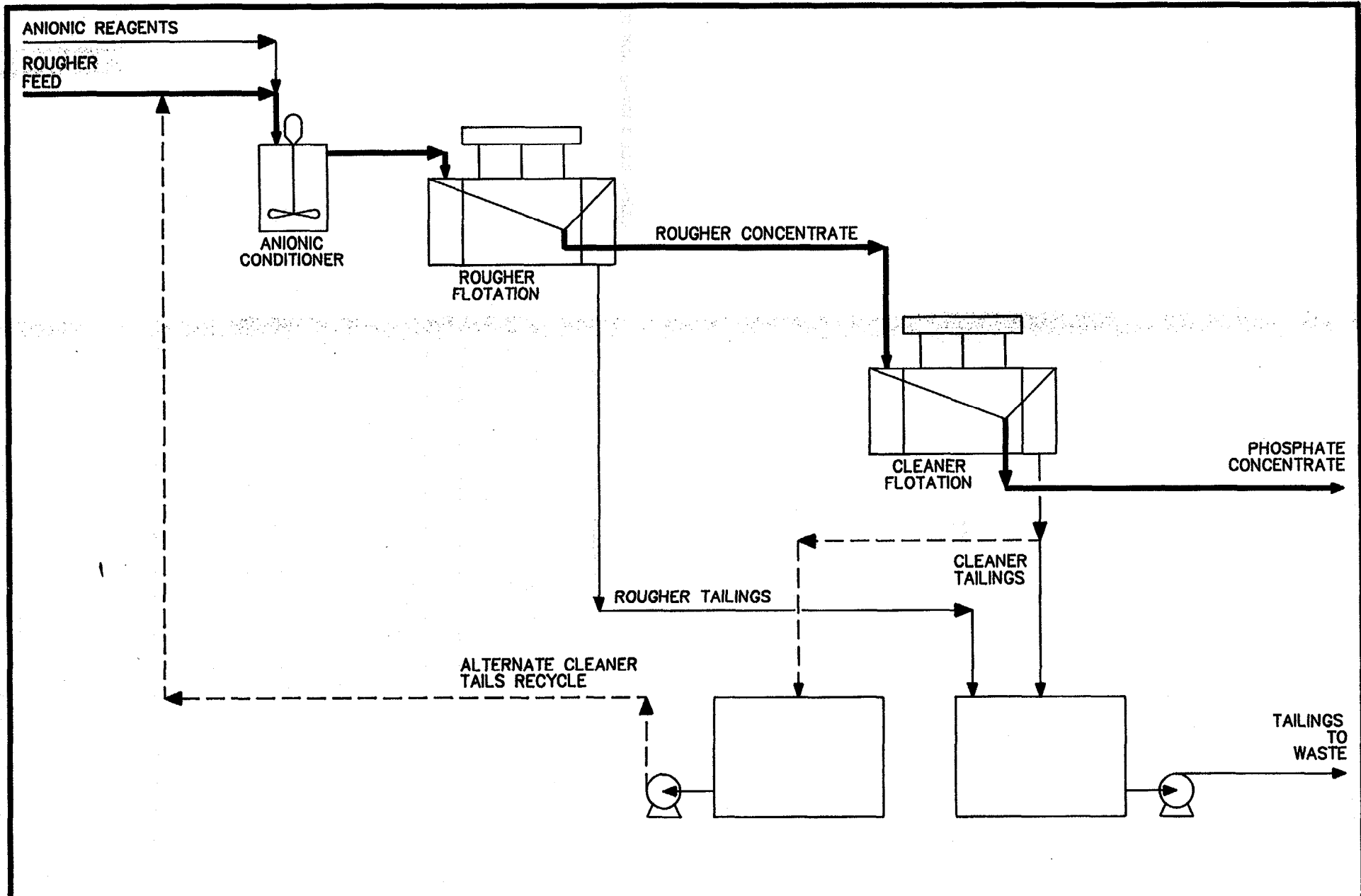
7-10

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CRAGO REVERSE FLOTATION
 FIGURE 7-2



7-11

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ROUGHER-CLEANER FLOTATION
 FIGURE 7-3

The total capital saving for an anionic rougher-cleaner grass-root plant, having the characteristics outlined in Section 7.3, is estimated as follows:

	<u>Saving (\$1,000)</u>
Direct capital	800
Indirect capital @ 30% of directs	240
Fixed capital	1,040
Startup expense @ 6% of fixed	62
Working capital @ 15% of total	<u>195</u>
TOTAL SAVING	1,297

The total capital saving from utilizing the anionic rougher-cleaner process instead of the Crago process in a grass-roots plant is about 1.3 million dollars.

7.5 Operating Cost Impacts

The operating cost savings from the adoption of the anionic rougher-cleaner process has been estimated as about \$2.50 per ton of flotation concentrate. The comparison of operating costs between the rougher-cleaner and the Crago processes is shown in Table 7-2. The operating cost comparison was made by utilizing the ZW cost model (File No. 7R) developed for FIPR Project 86-04-031. The cost model was modified to reflect the flotation feed characteristics and metallurgical performance from the test program. Reagent consumption and unit costs were also modified to be consistent with the test program. The cost analysis considered the following:

- (1) A greater yield of flotation concentrate for the rougher-cleaner process due to: (a) elimination of the deoiling and washing steps and (b) a flotation recovery improvement of 2 percent.
- (2) Lower process water requirements for the rougher-cleaner process.

Table 7-2

Operating Cost Comparison

<u>Area</u>	<u>Operating Costs - \$1000/yr</u>	
	<u>Anionic</u>	<u>Crago</u>
Mining	7,020	7,020
Beneficiation	10,327	11,056
Waste, Reclaim & Water	6,271	6,347
Product Management	1,426	1,362
Administration	<u>4,056</u>	<u>3,874</u>
Total Costs	29,100	29,659
Pebble ktpa	1660	1660
Concentrate ktpa ⁽¹⁾	<u>1460</u>	<u>1320</u>
Product ktpa	3120	2980
\$/ton concentrate	19.94	22.46
\$/ton product	9.33	9.95

(1) Metallurgical performance data are shown in Section 6, and tonnage data are from the cost model.

- (3) Lower reagent cost for the rougher-cleaner process due to the elimination of sulfuric acid and amine.
- (4) Lower power consumption for the rougher-cleaner process due to the elimination of the deoiling and washing steps.

No credit for reduced maintenance was taken. Additional details of the operating cost estimates are provided in Appendix 'C'. The operating costs per ton of product for each area is shown on Table 7-3.

Table 7-3
Operating Cost Comparison

<u>Area</u>	<u>Cost (\$/ton product)</u>	
	<u>Anionic</u>	<u>Crago</u>
Mining	2.25	2.35
Beneficiation	3.31	3.71
Waste, Reclaim & Water	2.01	2.13
Product Management	0.46	0.46
Administration	<u>1.30</u>	<u>1.30</u>
Total Operating Cost:	9.33	9.95

7.6 Preliminary Risk/Benefit Analysis

Anionic rougher-cleaner flotation has associated risks that may be categorized as beneficiation risks and acidulation risks. Risks in beneficiation focus on the probability of failure of the process to realize the anticipated benefits in an operating plant. In acidulation, the risks are that the lower grade reagentized concentrate from anionic rougher-cleaner flotation will incur penalties. Cost penalties, although not expected to be

significant, may result due to increases in defoamer consumption, abrasion, and power consumption. Similarly, a small P_2O_5 recovery penalty may result from changes in rock grindability and/or gypsum filtration.

Phase I bench scale test results confirmed the potential benefits of the anionic rougher-cleaner process that were identified in Zellars-Williams revised proposal of December 15, 1986. The anticipated potential benefits are compared below to the benefits determined in Phase I.

	<u>“Anticipated” Benefits</u>	<u>“Phase I” Potential</u>
BPL recovery improvement	4 to 8%	2 to 4%
Reagent cost reduction	20 to 40%	36 to 48%
Cash cost reduction/T concentrate	\$1.0 to 1.5	\$2.5
Reduction in well water make-up	yes	yes
Reduction in energy	yes	yes
Existing plant retrofit	easy	easy
Grass-roots plant capital cost	reduced	reduced

Pilot plant testing and full scale plant tests are required to quantify the beneficiation risks.

The anionic rougher-cleaner process is not expected to generate any benefits in acidulation other than providing less expensive phosphate rock. The risks involving acidulation of the lower grade reagentized concentrate can only be quantified by comparative testing.

Relative to the Crago process, the anionic rougher-cleaner process has environmental benefits and no identified environmental risks. The elimination of reagents such as sulfuric acid, amine, and kerosene is expected to be environmentally beneficial. Also, the anionic rougher-cleaner process requires less well water make-up than the Crago process.

SECTION 8

RECOMMENDATIONS

8.1 Potential of Process

The preliminary evaluation (Phase I) confirmed the potential of anionic rougher-cleaner flotation. The potential benefits are substantial and more extensive studies of flotation and acidulation of flotation concentrates are warranted.

8.2 Proposed Program

A Phase II program was recommended to the Florida Institute of Phosphate Research by the Zellars-Williams Company on December 31, 1987. The proposal entitled "Pilot Plant Evaluation of Anionic Rougher-Cleaner Flotation on Florida Phosphate" included beneficiation testing, acidulation testing, and techno-economic analysis.

8.2.1 Beneficiation Testing

The potential benefits of the anionic rougher-cleaner process demonstrated by Phase I bench scale testing warrant the testing of additional feeds in a pilot scale flotation test program. Zellars-Williams recommends that the proposed Phase II program be approved. The proposed beneficiation testing includes:

- o anionic rougher-cleaner pilot scale flotation of four flotation feeds.
 - Each feed to be examined at three dosages of reagent to develop grade vs recovery vs cost information.
 - test sequencing to be conducted to examine the effects of recycle water.
- o Crago pilot scale flotation of the same flotation feeds
- o bench scale flotation testing to support the pilot scale anionic and Crago flotation testing.

8.2.2 Acidulation Testing

The total impact of the anionic rougher-cleaner process on acid plant performance is not predictable from the concentrate chemical composition alone. The ratios of CaO, MgO, Fe₂O₃ and Al₂O₃ to P₂O₅ are useful well known indices; however, defoamer consumption, abrasion, and filtration rate are determined by testing. Zellars-Williams recommends that the proposed Phase II program be approved. The proposed acidulation testing includes:

- o acidulation testing of selected final concentrate from anionic flotation
- o acidulation testing of a parallel concentrate from Crago flotation
- o supplemental testing and analysis of results to examine the following factors on a relative basis:
 - rock grindability
 - corrosion/abrasion
 - defoamer consumption
 - filtration rate
- o The following information will also be determined:
 - H₂SO₄ consumption
 - classification of P₂O₅ losses
 - distribution of impurities
 - calculated dry basis P₂O₅ of 28% acids
 - effect of residual reagent on rubber lining

8.2.3 Techno-Economic Analysis

The potential benefits of anionic rougher-cleaner flotation may be offset by the potential disadvantages of its lower grade reagentized concentrate. High grade rock is typically used for export and for the manufacture of TSP. Lower grade rock is normally used for phosphoric acid manufacture. The

proposed Phase II beneficiation and acidulation testing will provide information to quantify the potential benefits and disadvantages of anionic rougher-cleaner flotation on the overall economics of producing filter acid. Included in the techno-economic analysis are:

- o An update of the Phase I estimated cash operating costs based on continuous pilot scale flotation test results
- o An update of Phase I estimated costs to retrofit a hypothetical flotation plant with the anionic rougher-cleaner process
- o An update of Phase I estimated net capital cost difference between grass-roots plants having the Crago process and the anionic rougher-cleaner process.
- o An estimate of the net effect on the overall economics of producing filter acid from anionic concentrate.

8.3 Discussion

The three facets of the program; beneficiation testing, acidulation testing, and techno-economic analysis are complementary and necessary to examine the overall impacts of producing filter acid from concentrate produced by the anionic rougher-cleaner process. Beneficiation testing will establish the continuous flotation performance of the anionic rougher-cleaner process and the Crago process for four plant prepared feeds. Acidulation testing will produce filter acid from Crago and anionic concentrates under controlled conditions and without the external interferences that are common to full scale plant tests. The techno-economic analysis will incorporate the results of beneficiation and acidulation testing to evaluate capital and operating cost impacts.