

Publication No. 02-151-200

# ANIONIC ROUGHER-CLEANER FLOTATION

*Prepared by*  
Jacobs Engineering Inc.

*under a grant sponsored by*



May 2003

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ANIONIC ROUGHER-CLEANER FLOTATION

FINAL REPORT

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May 2003

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## PERSPECTIVE

Patrick Zhang, Research Director - Beneficiation & Mining

In the Crago “double float” process, sized flotation feed is dewatered and conditioned at about 70% solids with fatty acid/fuel oil at about 9 pH for three minutes. The phosphate is then floated to produce a rougher concentrate and a sand tailing. The rougher concentrate goes through a dewatering cyclone, an acid scrubber, and a wash box to remove the reagents from the phosphate surfaces. After rinsing, the deoiled rougher concentrate is transported into flotation cells where amine is added. The silica is finally floated at neutral pH.

In this conventional process, about 30-40% by weight of the sands in the feed are floated twice, first by fatty acid, and then by amine. This Crago process is, therefore, inefficient in terms of collector utilization. One of the major drawbacks of this process is the deoiling process. Deoiling consumes a significant amount of sulfuric acid, which calls for special safety cautions and equipment maintenance. Insufficient deoiling, which happens frequently, often causes poor concentrate grade. Deoiling also causes loss of fine phosphate particles, amounting to more than 1% phosphate recovery in most operations. Another problem with the Crago process is with the amine flotation step. Not only are amines more expensive than fatty acids, but they are also very sensitive to water quality, particularly the slime content in water.

Direct flotation using anionic reagents was practiced on high-grade flotation feeds in some U.S. plants, and is still being used in a Mexican plant. Some research efforts have also been directed at developing an anionic flotation process for phosphate, but the interest faded due to the stringent requirement for Insol (at 4-5%) content in the phosphate rock product in the past. However, that requirement has been relaxed in recent years, with companies now accepting concentrate analyzing as high as 10% Insol, which an anionic rougher-cleaner process could achieve without sacrificing much flotation recovery.

Another driving force for an anionic flotation process is its environmental friendliness. Such a process would eliminate the use of sulfuric acid and amines for phosphate beneficiation, reduce water and energy usage, and curtail the total discharge of chemicals to the environment. An anionic flotation process also offers improved phosphate recovery.

Although the original proposal was designed to optimize an anionic rougher-cleaner flotation process developed by Jacobs Engineering under a previous funding, the project ended up testing a handful of anionic flotation options, including three flowsheets developed under a FIPR in-house program. All these processes demonstrated the potential to achieve higher recovery at lower reagent cost than the Crago process. As the report indicates, two of the FIPR flowsheets required further optimization of sizing, which was beyond the scope of this project.

## ABSTRACT

The Crago process has been used almost exclusively for about 50 years to recover flotation concentrate from Florida phosphate ores. Although the process has been able to produce high-grade concentrate from all manner of feeds, relatively high costs were incurred because of the need for anionic reagents, sulfuric acid, and cationic reagents. The character of current and future phosphate ores indicates that reagent costs per ton of recovered product will continue to increase. A program to evaluate alternative flotation processes that use only anionic reagents was proposed by Jacobs and approved by the Florida Institute of Phosphate Research.

Of the six process options considered, four were selected for pilot plant testing. Flotation feed from three different mines was used in the comparative testing. The majority of the tests were performed with tap water; however, plant process water was used to compare the best anionic process options to the Crago process.

One of the anionic processes tested met the goals set for an alternative process. These goals were, relative to the conventional Crago process, to improve BPL % recovery by 2 to 4% and reduce reagent costs by 33%. The disadvantage of the anionic process is production of lower grade concentrates.

## ACKNOWLEDGEMENTS

This research program was funded by the Florida Institute of Phosphate Research (FIPR), contract number FIPR #01-02-151. The successful completion of the pilot plant program would not have been possible without the Institute's sponsorship and continued interest. Dr. Zhang provided leadership and direction to the program. Mr. Snow and/or Mr. Yu were frequent pilot plant visitors, contributing flowsheets and reagent combinations to the test program.

Cargill Crop Nutrition, CF Industries, IMC Phosphates, and PCS Phosphate provided samples of plant prepared flotation feed and contributed personnel and resources to the plant sampling effort. The author and Jacobs' pilot plant staff sincerely appreciate the assistance provided by the management and numerous plant personnel. The operating companies also cooperated by allowing tanker trucks of plant water to be obtained and by providing samples of plant reagents.

Jacobs gratefully acknowledges Dr. Gerald Luttrell of Virginia Tech for providing the complimentary material balance software, which was used to prepare the material balance for each pilot plant run. Arizona Chemical, Custom Chemical, and US Filter contributed reagent samples for use in the test program.

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## EXECUTIVE SUMMARY

The role of phosphate flotation is expanding because the Florida ores currently exploited contain less pebble and more flotation concentrate than previously mined ores. The reduced content of pebble is even more pronounced for future ores. Consequently the efficiency and reagent costs of flotation are of increasing importance.

### SCOPE

The Board of Directors of the Florida Institute of Phosphate Research approved funding of a program (FIPR #01-02-151) to test alternatives to the conventional flotation method (Crago process) that has been practiced for 50 years by Florida producers of phosphate rock. The scope of work involved various tasks as outlined below.

#### Laboratory Testing

Samples of phosphate flotation feed were collected from four plants operated by Cargill Crop Nutrition, CF Industries, IMC Phosphates, and PCS Phosphate. More than 100 laboratory scale flotation tests were performed with these samples to compare the collectors currently used by the four plants with various anionic collectors. The test data were evaluated to select effective collectors for the pilot scale evaluation of anionic flotation process alternatives to the Crago process.

#### Pilot Testing

Five truckloads of feed (75 tons total) were obtained from three of the above-mentioned phosphate flotation plants. Plant flotation reagents and selected anionic collectors were also obtained. The proposed program intended a minimum of 12 formal tests (runs), eight with laboratory tap water and four with plant process water. However, the work went well and 39 formal runs were performed within the original budget. The majority of the testing (29 runs) was performed with tap water and some comparative tests (10 runs) were performed with plant process water. Four tanker trucks (20,000 gallons total) of plant process water were obtained from two of the plants.

Four of six anionic process options were selected for pilot scale testing, based on previous work and preliminary laboratory testing. The flotation processes evaluated in the pilot plant program are identified and briefly described in the following listing.

- **Crago process:** This three-step process uses three suites of reagents and produces two tailing streams and a final concentrate.

1. Rougher flotation with anionic reagents to reject a sand tailing and recover a low-grade concentrate (direct flotation).
  2. De-oiling of rougher concentrate by sulfuric acid scrubbing and water rinsing to remove anionic reagents.
  3. Cleaner flotation with cationic reagents to reject a sand tailing and recover a high-grade phosphate concentrate (inverse flotation).
- **Anionic process option 1:** This process has two steps and a single suite of reagents. Two tailing streams and a final product are produced.
    1. Rougher flotation with anionic reagents to reject a sand tailing and recover a low-grade concentrate (direct flotation).
    2. Cleaner flotation by re-floating the rougher concentrate to drop out a sand tailing and recover a medium-grade phosphate concentrate (direct flotation).
  - **Anionic process option 4:** This process has three steps and a single suite of reagents. Two tailing streams and a final product are produced.
    1. Rougher flotation with anionic reagents to reject a sand tailing and recover a low-grade concentrate (direct flotation).
    2. Cleaner flotation by re-floating the rougher concentrate to drop out a sand middling and recover a medium-grade phosphate concentrate (direct flotation).
    3. Middling treatment: The sand middling is size classified into fine and coarse fractions. The fine fraction is a tailing and the coarse fraction is recycled for treatment with anionic reagents and rougher flotation.
  - **Anionic process option 5:** This process also has three steps and a single reagent suite; however, two tailing streams and two products are produced.
    1. Rougher flotation with anionic reagents to reject a sand tailing and recover a low-grade concentrate (direct flotation).
    2. Rougher concentrate treatment: The rougher concentrate is size classified into fine and coarse fractions. The coarse fraction is retained as a medium-grade phosphate concentrate, and the fine fraction is sent to cleaner flotation.

3. Cleaner flotation by re-floating the fine rougher concentrate to drop out a sand tailing and recover a medium-grade phosphate concentrate (direct flotation). The fine and coarse concentrates may be combined.
- **Anionic process option 6:** This process also has three steps and produces two tailing streams and two products. Two suites of anionic reagents are used.
    1. Rougher (starvation) flotation with one suite of anionic reagents to recover a sand middling and a medium-grade concentrate of fine phosphate (direct flotation).
    2. Middling treatment: The sand middling is size classified into fine and coarse fractions. The fine fraction is a tailing and the coarse fraction is sent to scavenger flotation.
    3. Scavenger flotation of the coarse middling with a second suite of anionic reagents to reject a sand tailing and recover a medium-grade concentrate of coarse phosphate (direct flotation).

## **PROGRAM OBJECTIVE AND RESULTS**

The purpose of this program was to demonstrate an alternative to the conventional Crago process. Goals for the alternative flotation method, relative to the Crago process are listed below.

### **Goals**

#### **Increase Flotation Recovery by 2 to 4%**

This goal was achieved by anionic process option 4, which increased flotation recovery by 3 to 7% relative to the Crago process.

#### **Reduce the Cost of Flotation Reagents by 33%**

This goal was also achieved by anionic process option 4, which reduced reagent costs by 30% per ton of feed and 42% per ton of concentrate.

#### **Eliminate Usage of Sulfuric Acid and Cationic Reagents**

All anionic flotation processes tested accomplished this goal. Of those anionic processes tested, Options 4 and 5 gave the best metallurgical performance and lowest reagent costs.

## **Results**

Summarized data for each process tested are shown in Table 1. The data for the Crago process and anionic process options 4 and 5 are comparable because they were obtained from testing the same feeds. Data for Options 1 and 6 were obtained from tests of one feed only and are not directly comparable to the other process data.

Anionic process option 4 was superior to the other processes with respect to BPL % recovery and reagent consumption. The Crago process, as expected, was superior to the other processes with respect to final concentrate grade.

## **CONCLUSION**

Alternatives to the Crago process were successfully demonstrated by pilot scale tests. The results obtained with anionic process option 4 met identified goals. Additional evaluation of anionic flotation by operating companies is warranted if the production of low-Insol concentrates is not an operating requirement.

**Table 1. Summary of Pilot Plant Test Results.**

|  | <b>Crago</b>                 | <b>Anionic Flotation Process</b> |                               |                               |                               |
|--|------------------------------|----------------------------------|-------------------------------|-------------------------------|-------------------------------|
|  | <b>Process<sup>(1)</sup></b> | <b>Option 1<sup>(2)</sup></b>    | <b>Option 4<sup>(1)</sup></b> | <b>Option 5<sup>(1)</sup></b> | <b>Option 6<sup>(3)</sup></b> |
| <b>Feed grade</b>                      |                              |                                  |                               |                               |                               |
| % BPL                                  | 16.7                         | 19.7                             | 17.5                          | 17.3                          | 17.9                          |
| % Insol                                | 76.0                         | 72.1                             | 75.2                          | 75.0                          | 74.4                          |
| <b>BPL % Recovery<sup>(4)</sup></b>    |                              |                                  |                               |                               |                               |
| First tailing                          | 11.6                         | 9.5                              | 9.7                           | 11.7                          | 10.1                          |
| Second tailing                         | 6.5                          | 0.8                              | 1.7                           | 2.7                           | 9.7                           |
| Final concentrate                      | 81.9                         | 89.7                             | 88.6                          | 85.6                          | 80.2                          |
| <b>Concentrate Grade<sup>(4)</sup></b> |                              |                                  |                               |                               |                               |
| % BPL                                  | 69.3                         | 64.4                             | 62.9                          | 61.0                          | 63.7                          |
| % Insol                                | 4.1                          | 10.7                             | 12.9                          | 14.1                          | 9.1                           |
| <b>Reagent (Lbs/t)</b>                 |                              |                                  |                               |                               |                               |
| Anionic collector                      | 1.03                         | 1.46                             | 0.98                          | 1.23                          | 0.73                          |
| Fuel oil                               | 0.43                         | 0.87                             | 0.40                          | 0.37                          | 0.60                          |
| Soda ash                               | 0.57                         | 0.35                             | 0.37                          | 0.57                          | 0.78                          |
| Sodium silicate                        | 0.35                         | 0.20                             | 0.11                          | 0.27                          | 0.78                          |
| Sulfuric acid                          | 3.69                         | -                                | -                             | -                             | -                             |
| Cationic collector                     | 0.29                         | -                                | -                             | -                             | -                             |

- (1) Data are averaged for feeds from plants 1, 3, & 4.
- (2) Data obtained from plant 3 feed only
- (3) Data obtained from plant 4 feed only
- (4) Plant 1 feed was refractory, causing recovery losses and significant grade dilution in options 4 & 5.

|  |      |      |      |      |      |
|--|------|------|------|------|------|
| <b>BPL % Recovery<sup>(5)</sup></b>    |      |      |      |      |      |
| Final concentrate                      | 87.1 | 89.7 | 90.6 | 88.3 | 80.2 |
| <b>Concentrate Grade<sup>(5)</sup></b> |      |      |      |      |      |
| % BPL                                  | 69.2 | 64.4 | 65.1 | 63.4 | 63.7 |
| % Insol                                | 3.8  | 10.7 | 9.5  | 10.9 | 9.1  |

- (5) Data exclude results from tests with plant 1 feed

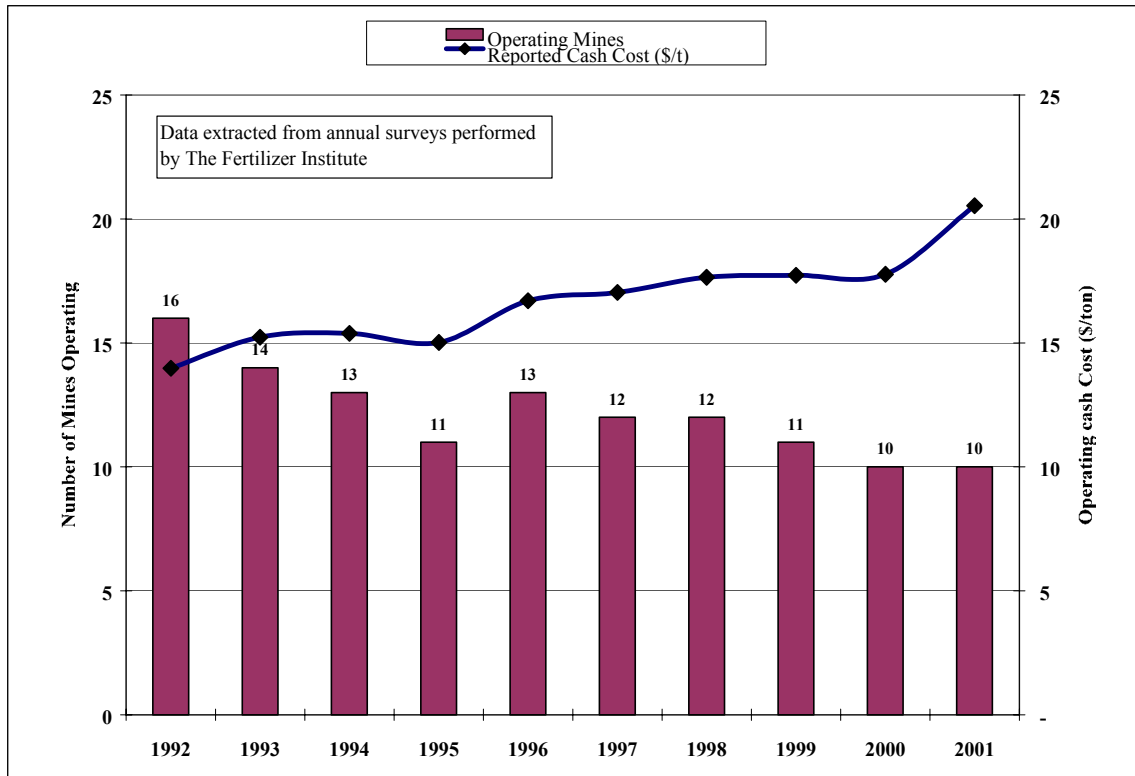


## INTRODUCTION

### PROBLEM

Phosphate rock profit margins are declining because sales prices have not kept pace with production costs. Consequently the incentive to replace depleted mines with new mines is low.

Data extracted from The Fertilizer Institute's annual surveys of American phosphate mines are summarized in Figure 1. Cash costs, which exclude charges for depreciation, depletion, and royalties, increased to about \$20 per ton of product over the last decade. During that time sales prices were flat at about \$24 per ton of product, and the number of operating mines decreased from 16 to 10.

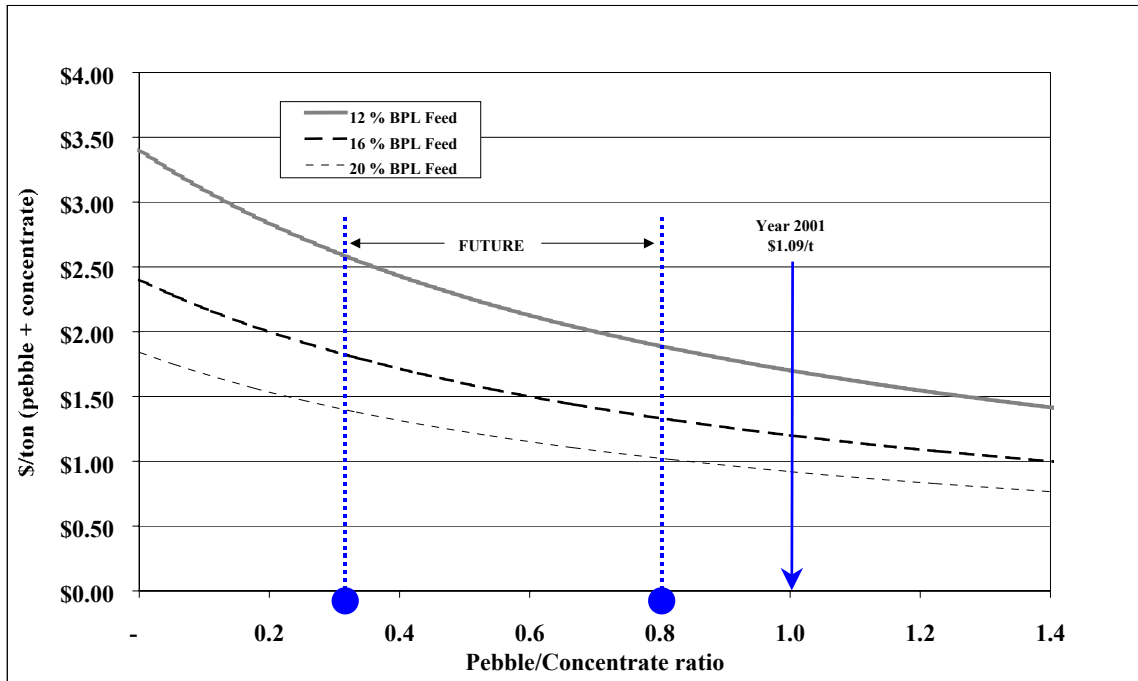


**Figure 1. Phosphate Rock Production Costs and Operating Mines.**

One factor causing material handling costs and flotation reagent costs to increase at Central Florida mines is the changing character of the phosphate ore. The yield of product is decreasing and the portion of product recovered as flotation concentrate is increasing.

The impacts of flotation on product yield and product costs are more critical to the Florida Phosphate Industry than ever before. The current ratio of pebble/concentrate is about 1. New mines will have pebble/concentrate ratios of 0.8 to 0.3, which will cause

production costs to increase. The impacts of producing less pebble and more concentrate on reagent costs are illustrated in Figure 2. As shown, the severity of the impact increases as the phosphate content of the feed decreases.



**Figure 2. Reagent Cost vs. Pebble/Concentrate Ratio.**

Reagent cost, expressed as \$/ton of total product will increase, unless compensating changes are made in the flotation process. The conventional Crago process utilizes three sets of reagents; anionic flotation reagents (fatty acids, fuel oils, and alkaline pH modifiers) for rougher flotation, sulfuric acid to remove anionic reagents from the rougher concentrate, and cationic flotation reagents (amines and possibly diesel oil) for cleaner flotation.

## HISTORICAL PERSPECTIVE

Production of phosphate rock from land pebble deposits commenced in 1890 and continues to the present day. Phosphate occurs in all particle sizes of the ore; however, only the enriched pebble fraction (>1 mm material) can be recovered as a commercial product by simple washing.

The advent of froth flotation made beneficiation of the feed fraction (1/0.1 mm material) a commercial reality also. Many flotation processes were examined, but the Crago Process, which was patented in 1942, became the standard method of recovering phosphate from the feed fraction of Florida phosphate ores. It should be noted that the Crago Process evolved in an industry where the economics of easily recovered pebble and the requirement for high-grade concentrate dominated beneficiation. This process allowed

significant quantities of phosphate resources to be reclassified as ore, and a significant period of Industry growth was initiated.

Arthur Crago's patented process was 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."

Although the process achieves Crago's objective, a re-evaluation is warranted because of the following evolutionary changes in the Phosphate Industry.

- Demand for high-grade concentrate has been significantly reduced because:
  - Phosphate is now exported in the form of high analysis fertilizers rather than as high-grade rock.
  - DAP and MAP fertilizers have displaced TSP in the market place. The former do not require high-grade rock.
- Florida phosphate deposits having a high yield of low-cost pebble are being depleted. The remaining ore reserves contain relatively more phosphate rock as concentrate than as pebble. Consequently, material handling costs and the Crago process reagent costs will continue to increase.

## **LITERATURE REVIEW**

Phosphate flotation processes with single reagent systems have the potential to lower reagent costs, relative to the process patented by Crago (1942), which utilizes three reagent systems. Zellars-Williams (1984) identified more than 10 phosphate plants that produced flotation concentrate with a single reagent system. The collectors used in these plants were mostly anionic, a few were amphoteric, and none were cationic.

Gruber (1989) demonstrated potential economic benefits for anionic rougher-cleaner flotation of Florida phosphate. Pilot plant testing of an all-cationic flotation process to remove silica from phosphate, reported by Slutskiy (1999) for the Florida Institute of Phosphate Research, did not reproduce the favorable results attained in laboratory testing.

Thom and Gisler presented a flowsheet for an early phosphate flotation operation in Florida utilizing a rougher-cleaner configuration with only anionic reagents. The rougher flotation stage of this and other early plants was essentially identical to that of present day plants; however, the rougher concentrate was re-floated to obtain a cleaner concentrate and one or more middlings that were recycled to rougher flotation. These "anionic" plants were phased out because the circulating load of middlings was unmanageable. Baderkhan (1999) reported middlings recycle problems at the Eshidiya phosphate flotation plant in Jordan.

Variations of the anionic rougher-cleaner process have been implemented to make the middlings circulating load more manageable. Denver Bulletin No. M7-F86, for flotation of western phosphates, shows the middlings being deslimed, dewatered, and conditioned with anionic reagents before they were recycled to the rougher flotation cells. Flow diagrams for western phosphates shown by Clitheroe (1967) had middlings being ground and/or conditioned with anionic reagents before being recycled to scavenger and cleaner flotation. Allen (1993) reported that the new phosphate beneficiation plant near Vernal, Utah also treats the cleaner tailing in scavenger circuits.

## PROJECT SCOPE

### Scope Development

Jacobs performed laboratory testing to compare the four options for anionic rougher-cleaner flotation illustrated in Figure 3. This preliminary test work was completed prior to submitting a proposal. Options 1 and 2 are the conventional configurations for rougher-cleaner circuits with direct flotation. Options 3 and 4 were addressed with locked cycle tests to examine the reported problems with middlings recycle. The Crago process was tested with two levels of cationic collector; one to attain a high-grade concentrate, and the second to maximize recovery. The laboratory test results are summarized in Table 2.

Comparing averaged data for Options 1 and 2 reveals a small trade off between grade (highest for Option 1) and recovery (highest for Option 2). Option 3 gave a relatively higher recovery; however, the middlings recycle was unstable and concentrate grade was still declining on cycle 4 of the locked cycle tests. Option 4 gave the best performance of the rougher-cleaner configurations and a very stable recycle of middlings. Comparing averaged data for the Crago process reveals a greater trade off between grade and recovery. With the high-grade operating mode the Crago process had higher reagent costs and lower recovery than the anionic options. With the high-recovery operating mode the Crago process had comparable recovery to Option 4, but higher reagent costs than the anionic options.

**Table 2. Jacobs Scope Definition Test Results.**

|                       | Anionic Rougher-Cleaner Option |      |       |      | Crago Process |          |
|-----------------------|--------------------------------|------|-------|------|---------------|----------|
|                       | 1                              | 2    | 3     | 4    | Grade         | Recovery |
| Concentrate % BPL     | 61.9                           | 61.5 | <54.1 | 62.0 | 68.0          | 61.2     |
| BPL % Recovery        | 90.7                           | 92.2 | 93.5  | 93.5 | 89.0          | 93.8     |
| Reagents (\$/t conc.) | 1.25                           | 1.22 | 1.28  | 1.17 | 1.78          | 1.55     |

The Florida Institute of Phosphate Research was also examining phosphate flotation with a single anionic collector. Their laboratory tests showed that rougher concentrates containing 12 to 13 percent acid insoluble material (Insol) could be

upgraded to 8 to 9 percent Insol at high recovery. Moreover, the Insol dilution in the rougher concentrate occurred in the finer particle sizes. Laboratory testing by the Florida Institute of Phosphate Research demonstrated two other options with potential to improve recovery relative to normal rougher-cleaner flotation and result in acceptable concentrate grade. Consequently, two additional process schemes, each involving sizing and identified as Options 5 and 6 in Figure 3, were added to the scope. Not shown in Figure 3 for Option 6 are two required anionic conditioning steps, the first prior to starvation flotation, and the second prior to scavenger flotation.

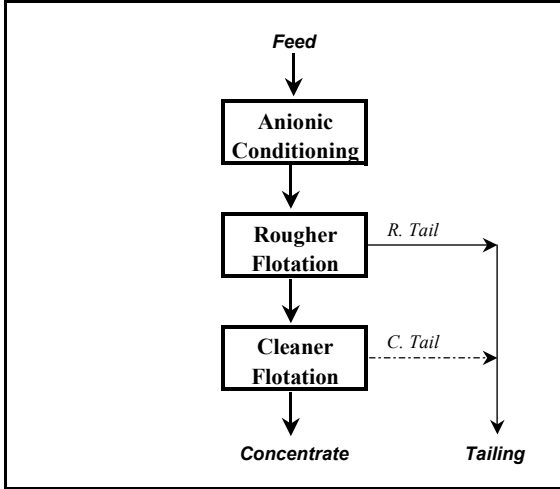
### **Contractual Scope**

Portions of the proposed scope dealing with preliminary technical and economic analysis of implementing the anionic rougher-cleaner process in existing plant and new plants, and fundamental research on selected anionic collectors were not approved. The objective of the approved scope was to demonstrate the technical feasibility of three anionic flotation options by pilot-scale and laboratory testing.

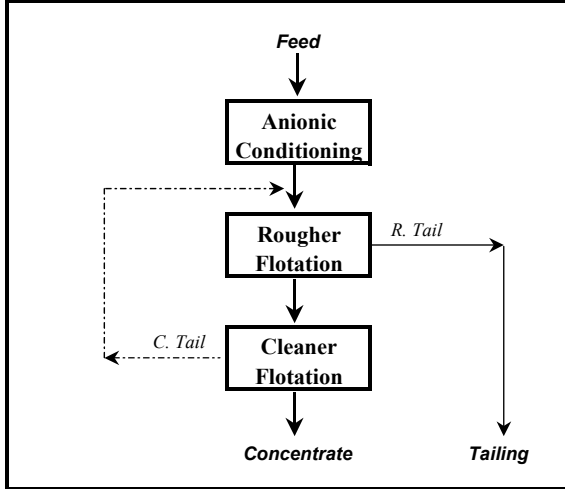
Major tasks for the approved scope are listed below.

1. Laboratory flotation tests to select the specific collectors for pilot testing.
  - a. Collect feed samples from four beneficiation plants
  - b. Obtain anionic reagent samples (nominally 6 per feed sample)
  - c. Perform comparative laboratory tests and establish reagent levels.
2. Beneficiation pilot plant testing of flotation feed samples.
  - a. Collect bulk samples of plant flotation feed
  - b. Obtain samples of plant reagents and selected anionic reagents
  - c. Perform comparative pilot-scale tests of the Crago process and anionic flotation options 4, 5 and 6.
3. Preparation of the project report.

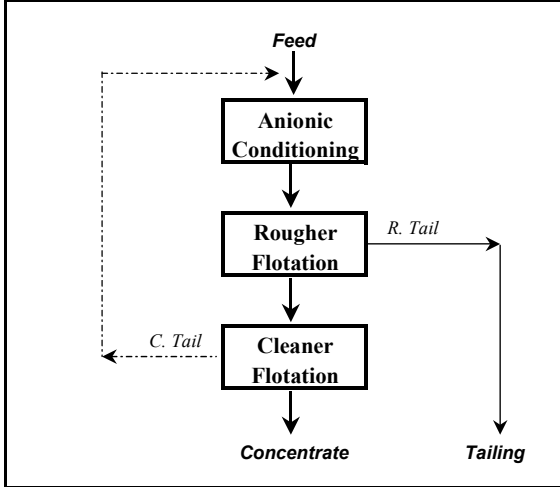
**Option 1**



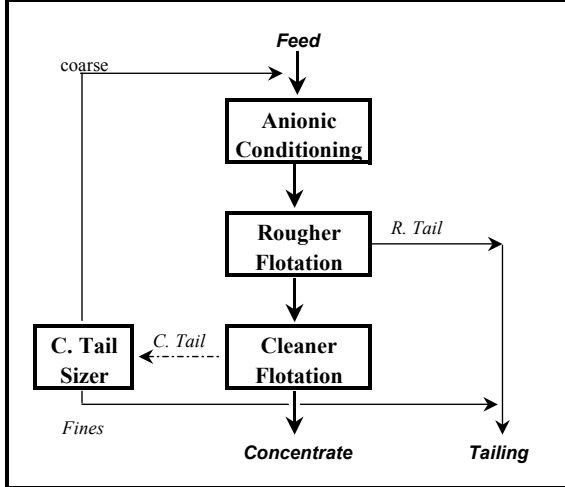
**Option 2**



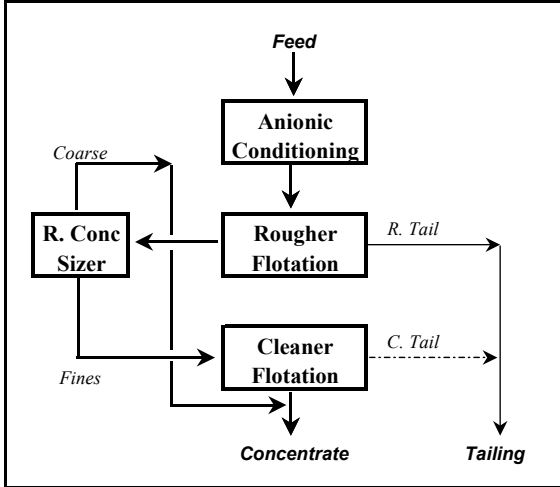
**Option 3**



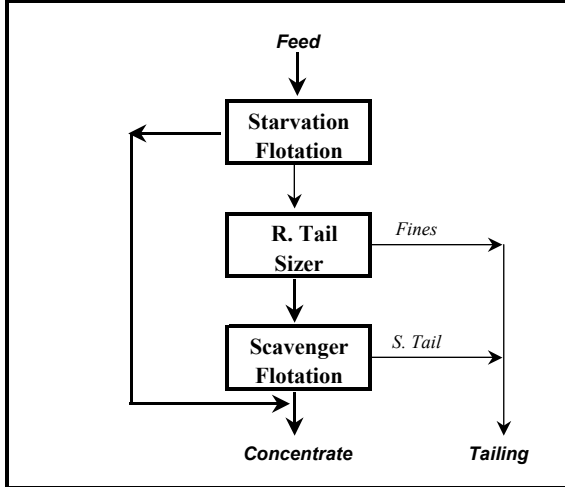
**Option 4**



**Option 5**



**Option 6**



**Figure 3. Process Options for Anionic Flotation.**

The initial proposal intended 16 formal runs, including six for comparing mechanical and column cells for anionic flotation. The approved scope excluded the comparisons of mechanical and column cells, but added testing of Option 6. The experimental design of the pilot-scale tests, which includes four formal runs with plant process water, is indicated in Table 3 below.

**Table 3. Experimental Design of Pilot-Scale Tests.**

| <b>Feed</b> | <b>Run</b> | <b>Water</b> | <b>Collector</b> | <b>Test Description</b>       |
|-------------|------------|--------------|------------------|-------------------------------|
| 1           | 1          | tap          | Plant 1          | Crago Process                 |
|             | 2          | tap          | C 1              | Anionic flotation option      |
|             | 3          | tap          | C 1              | Anionic flotation option      |
|             | 4          | tap          | C 1              | Anionic flotation option      |
|             | 5          | Plant        | Plant 1          | Crago Process                 |
|             | 6          | Plant        | C 1              | Best anionic flotation option |
| 2           | 7          | tap          | Plant 2          | Crago Process                 |
|             | 8          | tap          | C 2              | Anionic flotation option      |
|             | 9          | tap          | C 2              | Anionic flotation option      |
|             | 10         | tap          | C 2              | Anionic flotation option      |
|             | 11         | Plant        | Plant 2          | Crago Process                 |
|             | 12         | Plant        | C 2              | Best anionic flotation option |





## METHODOLOGY

### LABORATORY TESTING

#### Collection of Feed Samples

Samples of plant prepared flotation feed were collected from four phosphate plants operating in Florida. The samples, ranging from 40 to 60 kg, were typically collected from the plant conditioner feed streams, placed in sealed containers, and transported to Jacobs' laboratory in Lakeland, Florida.

In the laboratory, after the free water was drained from the samples, each sample was blended by hand and duplicate moisture determinations were made. Based on the moisture content, representative samples were weighed out to provide 1 kg (dry basis) charges for flotation tests. Sieve and chemical analyses of each moisture sample were performed. The averaged sieve and chemical data for each feed sample are presented later in the report.

Cargill Crop Nutrition, CF Industries, IMC Phosphates, and PCS Phosphate operated the plants; however, the sample identification below is based on collection sequence.

- Plant 1
- Plant 2
- Plant 3
- Plant 4

#### Reagent Samples

Plant reagents were collected and used for Crago Process flotation tests. Additional anionic collectors were obtained for testing each feed, based on suggestions from plant metallurgists, reagent vendors, and the experiences of the Florida Institute of Phosphate Research and Jacobs.

Soda ash was used as the pH modifier for three feed samples and aqueous ammonia was used for the fourth feed. Sodium silicate was used as a depressant for one feed sample.

#### Flotation Tests

Anionic conditioning was performed at nominally 72% solids in a 2000-ml. stainless steel beaker, with agitation by a cruciform impeller operated at 350 rpm by a

Labmaster LIU08 mixer. Anionic reagents (fatty acid and fuel oil blend) were added to the 1 kg (dry basis) flotation charge by micro-burette. The pH modifier was added with a 10 ml pipette and the pH was monitored with a digital pH meter. Sodium silicate, when used, was added with a micro-burette.

The conditioned feed was transferred into a 5000-ml. cell and diluted with tap water. Rougher flotation was performed with using a DECO Model D-12 laboratory flotation machine operated at 1500 rpm. The entire froth product was collected in a pan and transferred to the next process step. The rougher tails were dewatered, dried, weighed, and analyzed for BPL and acid insoluble material (Insol), using analytical procedures approved by the Association of Florida Phosphate Chemists.

For the Crago process tests, the rougher concentrates were dewatered using a 325-mesh screen, and then scrubbed at nominally 70% solids and pH 3 (pH adjusted with sulfuric acid). After scrubbing, the concentrate was rinsed on a 325-mesh screen with tap water to remove spent reagents and acidic water. Cationic cleaner flotation was performed with the same model of flotation machine, operated at 1200 rpm with a 3000-ml. cell. Amine reagent was added directly to the cell prior to opening the air valve. The sand froth and phosphate cell products were dewatered, dried, weighed, and analyzed for BPL and Insol.

For anionic cleaner flotation tests, the rougher concentrate was transferred into a 5000-ml. cell and diluted with tap water as required for level control. Cleaner flotation was performed with using a DECO Model D-12 laboratory flotation machine operated at 1500 rpm. The froth product was collected in a pan, dewatered, and dried. The cleaner tails were dewatered, dried, and weighed. Both the cleaner concentrate and tails were analyzed for BPL and Insol.

Test data, including product weights, analyses, and reagent usages were input to an Excel file that computed material balances and reagent consumptions. The laboratory flotation data is presented later in this report.

## **PILOT PLANT TESTING**

### **Collection of Feed Samples**

Bulk samples of plant feed were collected from three phosphate flotation plants operating in Florida. Feed slurry was extracted from pipeline sample taps, dewatered, and transported by a 15-ton haul truck to Jacobs' pilot plant in Lakeland, Florida. Slurry was collected and dewatered in the truck body at two plants. At the third plant the slurry was dewatered on an adjacent paved surface and then loaded onto the truck by a front-end loader. The quantity of sample from each plant follows.

- Plant 1: 2 loads
- Plant 3: 1 loads
- Plant 4: 2 loads

The trucks dumped feed onto a clean paved area near the pilot plant. A Bobcat was used for blending and reclaiming the feed.

## **Reagent Samples**

Plant reagents were used for Crago process flotation tests. Anionic collectors, selected on the basis of test results, were obtained for rougher-cleaner flotation testing. Soda ash was used as the pH modifier for the three feed samples. Sodium silicate was used for some tests as a depressant.

## **PILOT PLANT OPERATION**

### **Flow Diagrams and Process Equipment**

A listing of equipment used in the pilot scale testing of the anionic process options follows. The equipment numbers below are also shown on the flow diagrams corresponding to Option 4 (Figure 4), Option 5 (Figure 5), and Option 6 (Figure 6).

Item 1: Variable speed screw feeder (fabricated item - 500 to 1500 pounds/hr.)

Item 2: Feed transfer pump, (H-Q model P-003)

Item 3: Feed dewatering screw (fabricated item)

Item 4: Anionic conditioner (2 tanks @ 10-inch diameter x 10 inches high)

Item 5: Rougher flotation machine (Denver No.8, two cells)

Item 6: Cleaner flotation machine (Wemco model 18, 4 cells)

Item 7: Cleaner tails transfer pump, (HR 1-inch sand pump)

Item 8: Cleaner tails sizer, (Derrick model J24-36ms-1, with DF66 cloth)

Item 9: Coarse cleaner tails recycle pump, (H-Q model P-003)

Item 10: Rougher concentrate transfer pump (HR 1-inch sand pump)

Item 11: Rougher concentrate sizer (11a: Derrick model J24-36ms-1, with DF66 cloth), (11b: CFS Density Separator model 8x8)

Item 12: Rougher tails transfer pump, (H-Q model P-003)

Item 13: Rougher tails sizer, (Derrick model J24-36ms-1, with DF66 cloth)

Item 14: Scavenger feed transfer pump, (H-Q model P-003)

Item 15: Scavenger feed dewatering screw (fabricated item)

Item 16: Anionic conditioner (2 tanks @ 8-inch diameter x 8 inches high)

Item 17: Scavenger flotation machine (Wemco model 18, 4 cells)

The above equipment items were also used for testing the Crago process, except that item 15 was reconfigured to dewater rougher concentrate prior to de-oiling and item 16 was reconfigured as an acid scrubber. Two items not listed above were used for acid washing (Krebs cyclone model U2, and a fabricated acid wash tank).

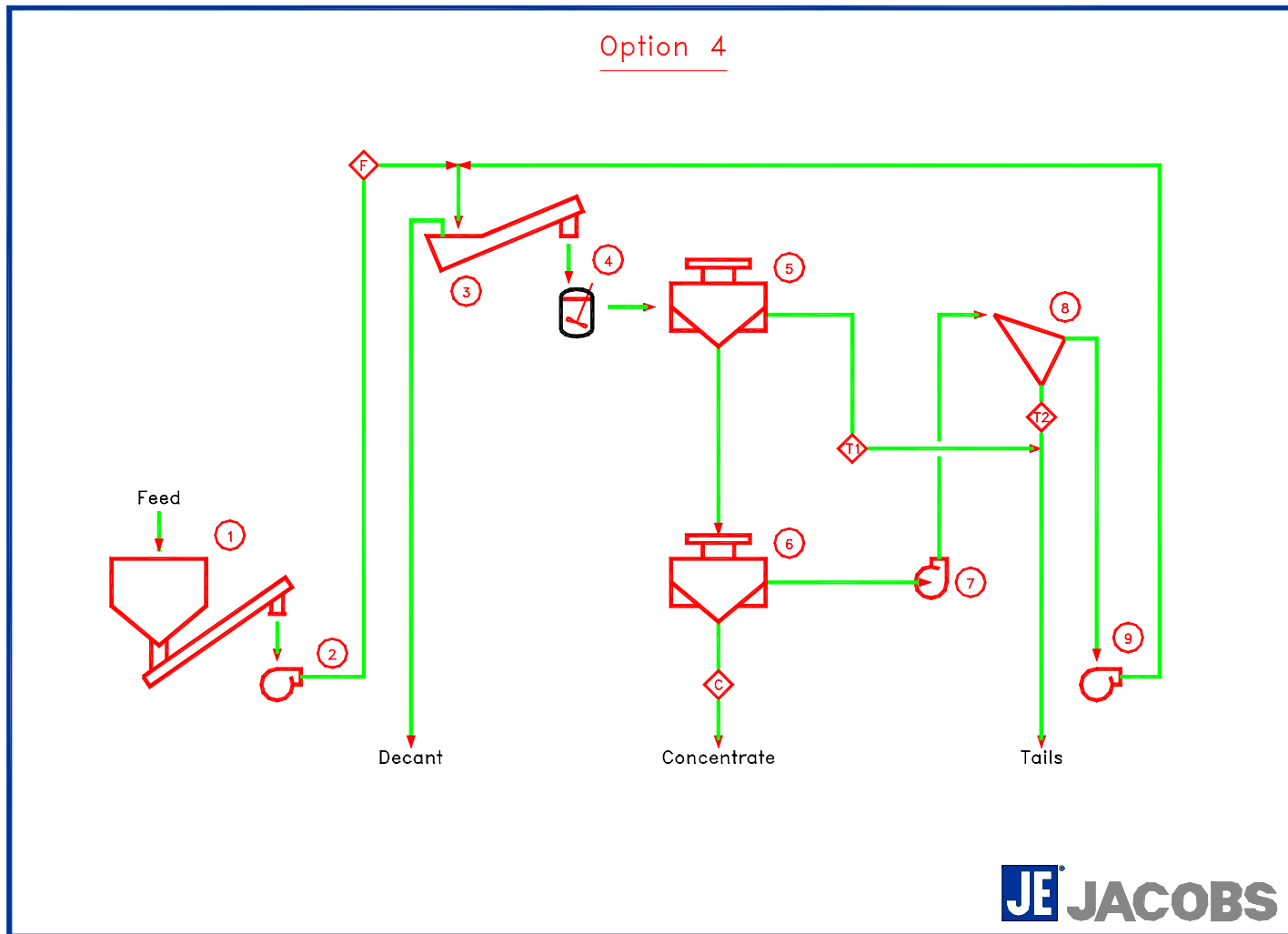


Figure 4. Anionic Process Option 4.

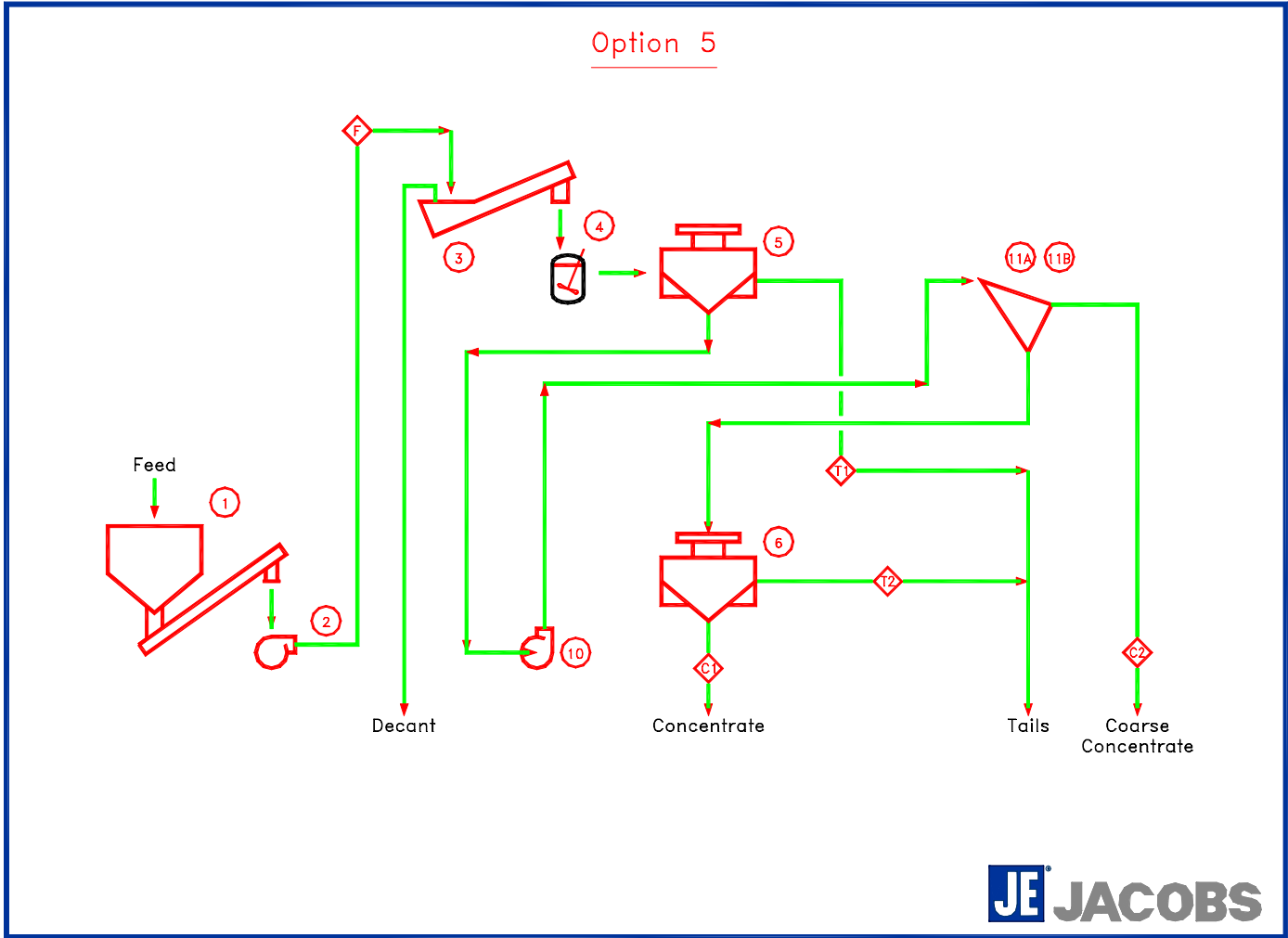


Figure 5. Anionic Process Option 5.

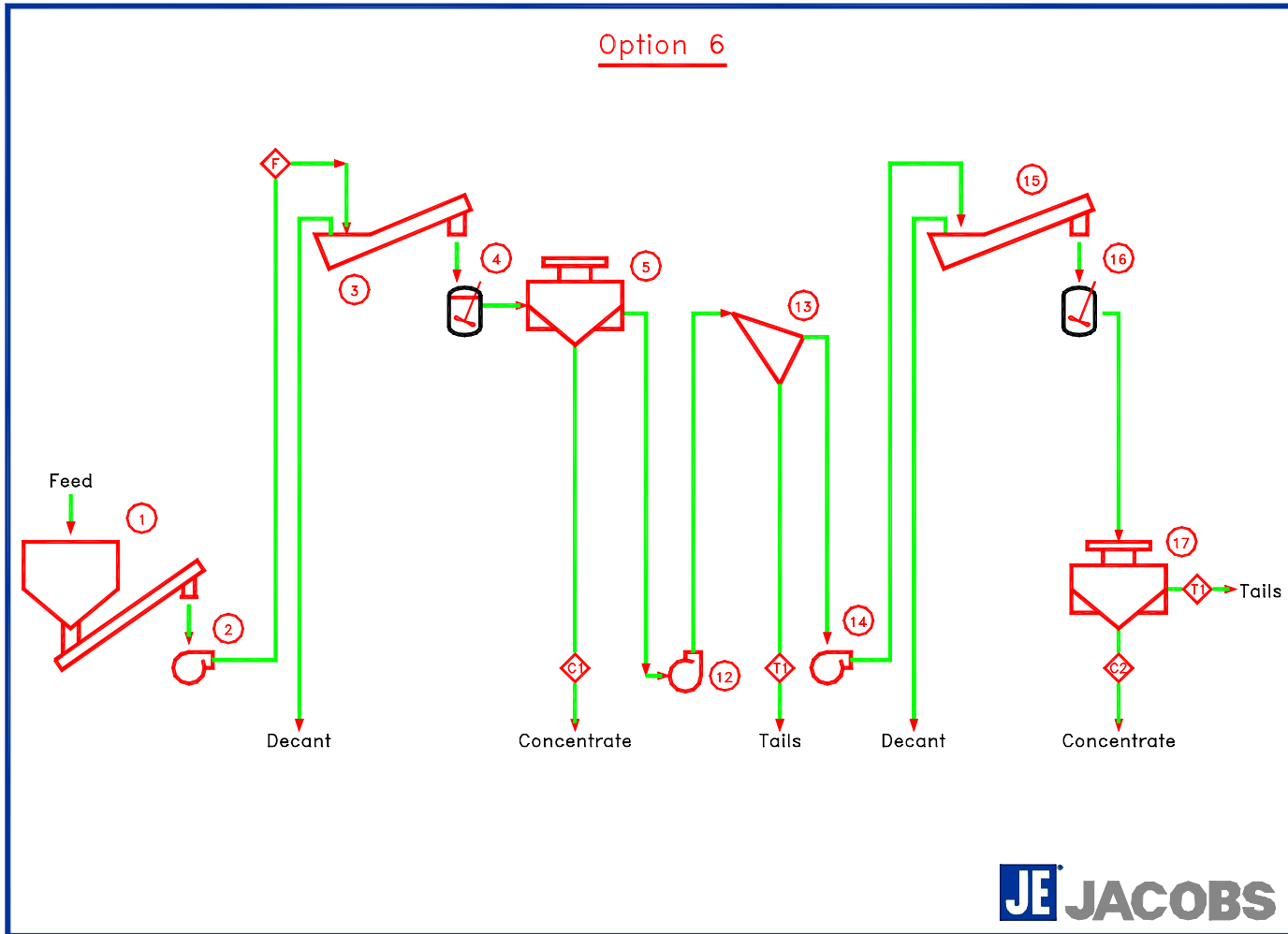


Figure 6. Anionic Process Option 6.

## Pilot Plant Sampling and Operation

Sample stations for the major streams for each process are shown as diamonds in Figures 4, 5, and 6. These slurry-sampling stations were configured to allow the entire stream to be diverted into a 5-gallon bucket. The interval between rounds of samples was normally 20 minutes. The duration of sampling and weight of collected sample for each major stream was measured and recorded to determine flow rates. Additionally, samples of critical internal streams, such as rougher concentrates, were taken during each round of samples.

Composite samples for each test were dried, weighed, and analyzed. The analyzed data for major streams are identified in Table 4. The sampled internal streams were analyzed for BPL, Insol, and % weight >48 mesh. The analyzed data for each test were input to an Excel program template provided by Luttrell (2001) to determine the best-fit material balance.

**Table 4. Major Stream Data.**

|          | Head Sample | Tyler Mesh Fractions |       |       |       |        |         |      |
|----------|-------------|----------------------|-------|-------|-------|--------|---------|------|
|          |             | >28                  | 28/35 | 35/48 | 48/65 | 65/100 | 100/150 | <150 |
| Lbs/hr   | yes         | no                   | no    | no    | no    | no     | no      | no   |
| % weight | no          | yes                  | yes   | yes   | yes   | yes    | yes     | yes  |
| % BPL    | yes         | yes                  | yes   | yes   | yes   | yes    | yes     | yes  |
| % Insol  | yes         | yes                  | yes   | yes   | yes   | yes    | yes     | yes  |

The normal routine for the pilot plant crew comprised four 10-hour days per week, as shown in Figure 7.

|                               | Day 1 | Day 2 | Day 3 | Day 4 |
|-------------------------------|-------|-------|-------|-------|
| Pilot Plant Formal Testing    |       |       |       |       |
| Sample preparation & analysis |       |       |       |       |
| Pilot Plant Formal Testing    |       |       |       |       |
| Sample preparation & analysis |       |       |       |       |

**Figure 7. Normal Pilot Plant Routine.**



## RESULTS

### LABORATORY FEED SAMPLES

Sieve and chemical analyses of the plant feed samples collected for reagent testing are presented in Table 5. Plant 3 was sampled twice, and both feed samples are shown. The BPL content of the plant flotation feed samples range from 13 to 20%. The cumulative distribution of BPL in the >35 mesh fractions of the feeds was fairly uniform at 21 to 25%, except for Plant 3 feed(b), which had 69% of its BPL in the >35 mesh fractions.

### LABORATORY TESTING

Rougher flotation tests were performed with selected anionic collectors on feeds from the four flotation plants. The purpose of these tests was to compare flotation results and identify anionic collectors more selective than the plant collector for subsequent pilot testing of the anionic process options. The coefficient of separation (*Coefficient of Separation = % BPL recovery - % Insol recovery*) was plotted against collector usage to compare reagent selectivity. Collector usages, expressed as pound of collector per ton of new feed to flotation (Lbs/t), are compared over a range of 0.2 to 1.2 Lbs/t. The usages shown are exclusive of fuel oil. Results from selected tests of feeds from plants 1, 2, 3, and 4 are discussed below.

#### Plant 1 Feed

From Figure 8 it is apparent that Sylfat FA12 was more selective than the plant collector and the other collectors tested on feed from Plant 1. Selectivity was highest for Sylfat FA12 and remained high over a broader range of reagent usage than for the other collectors.

The plant collector was added as a blend of 90% collector and 10% fuel oil. MO-5 and FA12 were added neat. The MO-5 blend and FA12 blend each contained 37.5% Ligo GA and did not give satisfactory performance. Tests of Ligo GA as the only collector confirmed that over 2 Lbs/t were required to attain acceptable recovery, consequently use of that sample of Ligo GA was discontinued.

Additional details of the 25 tests are presented in Table 6. Further testing of this feed showed that sodium silicate use increased the concentrate grade by 6 to 20% BPL and reduced BPL recovery by 2 to 4% for rougher flotation. Testing this feed also revealed that flotation results with fresh feed were more reproducible than with aged feed.

## **Plant 2 Feed**

The results from testing Plant 2 feed are illustrated in Figure 9 and summarized in Table 7. The plant collector was added as a blend of 80% collector and 20% fuel oil, while the other collectors were each added as a blend of 70% collector and 30% fuel oil. CENTURY MO-5 was the most selective of the collectors tested for Plant 2 feed.

## **Plant 3 Feed(a)**

Figure 10 indicates little difference in selectivity between the reagents tested on flotation feed from Plant 3. The plant collector was added as a blend of 60% collector and 40% fuel oil. MO-5, FA12, No. 151, and No. 225 were each added as a blend of 70% collector and 30% fuel oil. The least selective collector was No. 151.

Additional details for the 25 tests of feed(a) from Plant 3 are presented in Table 8.

## **Plant 3 Feed(b)**

The 40-kg feed sample collected from Plant 3 to establish collector dosage prior to pilot plant work contained about 12% BPL and 80% Insol. The bulk sample collected a few days later for pilot testing was significantly coarser and contained more phosphate. Data from the latter sample is presented for feed(b) in Table 3.

Laboratory tests on the 40-kg sample confirmed the finding from feed(a), that Sylfat FA12, CENTURY MO-5, and the plant collector had similar selectivity. At the same nominal dosage, these three collectors gave rougher flotation performances that were essentially identical (BPL recovery: 93 to 94.5%, concentrate % BPL: 58.4 to 59.4, and concentrate % Insol: 15 to 15.8).

## **Plant 4 Feed**

From Figure 11 it is apparent that CENTURY MO-5 was more selective than the collector used by Plant 4. MO-5 was also more selective than the other reagents tested, except for Sylfat FA12 at higher reagent usage.

The plant collector was added as a blend of 80% collector and 20% fuel oil. The other four collectors were each added as a blend of 70% collector and 30% fuel oil.

Additional details for the 25 tests on Plant 4 feed are presented in Table 9.

**Table 5. Sieve and Chemical Analyses of Plant Feed Samples for Laboratory Testing.**

| Tyler<br>Mesh | Plant 1 Feed |         |                |       |       | Plant 2 Feed |         |                |       |       | Plant 4 Feed |         |                |       |       |
|---------------|--------------|---------|----------------|-------|-------|--------------|---------|----------------|-------|-------|--------------|---------|----------------|-------|-------|
|               | Grade        |         | % Distribution |       |       | Grade        |         | % Distribution |       |       | Grade        |         | % Distribution |       |       |
|               | % BPL        | % Insol | Wt.            | BPL   | Insol | % BPL        | % Insol | Wt.            | BPL   | Insol | % BPL        | % Insol | Wt.            | BPL   | Insol |
| 20            | 54.9         | 21.8    | 0.4            | 1.6   | 0.1   | 35.8         | 48.8    | 1.9            | 3.4   | 1.2   | 58.8         | 14.6    | 0.6            | 2.7   | 0.1   |
| 28            | 40.5         | 43.3    | 1.9            | 5.5   | 1.0   | 23.2         | 67.2    | 4.2            | 5.0   | 3.9   | 21.8         | 68.7    | 4.3            | 7.4   | 3.6   |
| 35            | 24.5         | 65.2    | 7.9            | 14.2  | 6.4   | 21.2         | 69.9    | 11.0           | 12.1  | 10.5  | 9.5          | 86.3    | 19.6           | 14.5  | 20.7  |
| 48            | 15.1         | 78.2    | 25.2           | 27.9  | 24.5  | 22.7         | 68.1    | 22.3           | 26.4  | 20.9  | 10.9         | 84.0    | 22.3           | 19.0  | 23.0  |
| 65            | 11.1         | 83.7    | 41.4           | 33.9  | 43.2  | 18.7         | 73.2    | 33.3           | 32.5  | 33.5  | 13.9         | 79.8    | 25.4           | 27.6  | 24.9  |
| 200           | 9.7          | 85.6    | 23.0           | 16.4  | 24.5  | 14.2         | 80.1    | 26.6           | 19.6  | 29.2  | 13.4         | 80.8    | 26.8           | 27.9  | 26.6  |
| pan           | 17.6         | 71.3    | 0.3            | 0.4   | 0.3   | 22.7         | 66.3    | 0.8            | 1.0   | 0.7   | 9.8          | 82.7    | 1.0            | 0.8   | 1.0   |
| Total         | 13.6         | 80.3    | 100.0          | 100.0 | 100.0 | 19.2         | 72.8    | 100.0          | 100.0 | 100.0 | 12.8         | 81.4    | 100.0          | 100.0 | 100.0 |
| Head          | 13.1         | 81.2    |                |       |       | 19.3         | 72.8    |                |       |       | 12.8         | 81.5    |                |       |       |

| Tyler<br>Mesh | Plant 3 Feed(a) |         |                |       |       | Plant 3 Feed(b) |         |                |       |       |
|---------------|-----------------|---------|----------------|-------|-------|-----------------|---------|----------------|-------|-------|
|               | Grade           |         | % Distribution |       |       | Grade           |         | % Distribution |       |       |
|               | % BPL           | % Insol | Wt.            | BPL   | Insol | % BPL           | % Insol | Wt.            | BPL   | Insol |
| 20            | 47.9            | 24.1    | 1.1            | 3.8   | 0.3   | 49.8            | 28.4    | 11.2           | 27.8  | 4.5   |
| 28            | 39.8            | 40.1    | 2.6            | 7.2   | 1.3   | 27.6            | 60.9    | 15.0           | 20.5  | 12.8  |
| 35            | 27.7            | 59.3    | 7.3            | 14.1  | 5.4   | 19.0            | 72.8    | 22.3           | 21.0  | 22.9  |
| 48            | 19.0            | 72.4    | 15.4           | 20.7  | 14.1  | 13.1            | 80.9    | 26.5           | 17.2  | 30.2  |
| 65            | 12.9            | 81.7    | 28.4           | 25.8  | 29.3  | 11.7            | 83.0    | 17.7           | 10.3  | 20.7  |
| 200           | 8.9             | 87.2    | 44.2           | 27.8  | 48.6  | 8.8             | 86.8    | 6.4            | 2.8   | 7.8   |
| pan           | 8.0             | 69.7    | 1.1            | 0.6   | 0.9   | 8.4             | 87.0    | 0.9            | 0.4   | 1.1   |
| Total         | 14.2            | 79.2    | 100.0          | 100.0 | 100.0 | 20.1            | 71.0    | 100.0          | 100.0 | 100.0 |
| Head          | 14.1            | 79.4    |                |       |       | 19.9            | 71.5    |                |       |       |

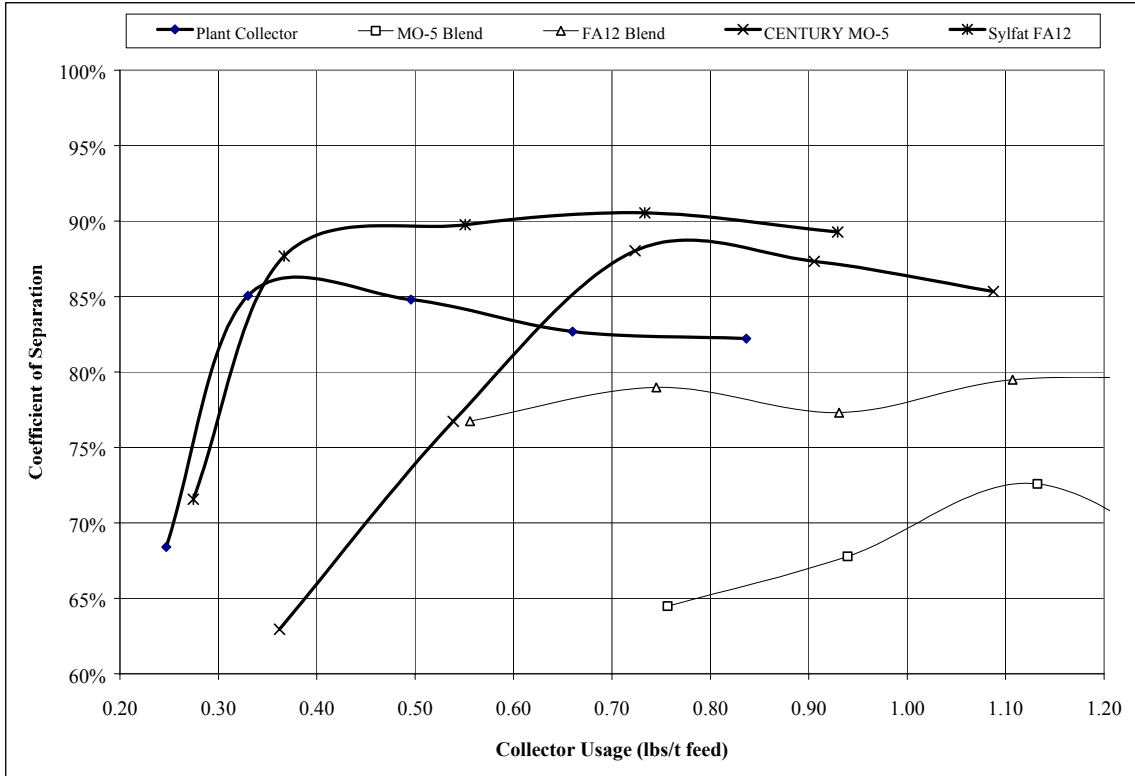


Figure 8. Collector Selectivity – Plant 1 Feed.

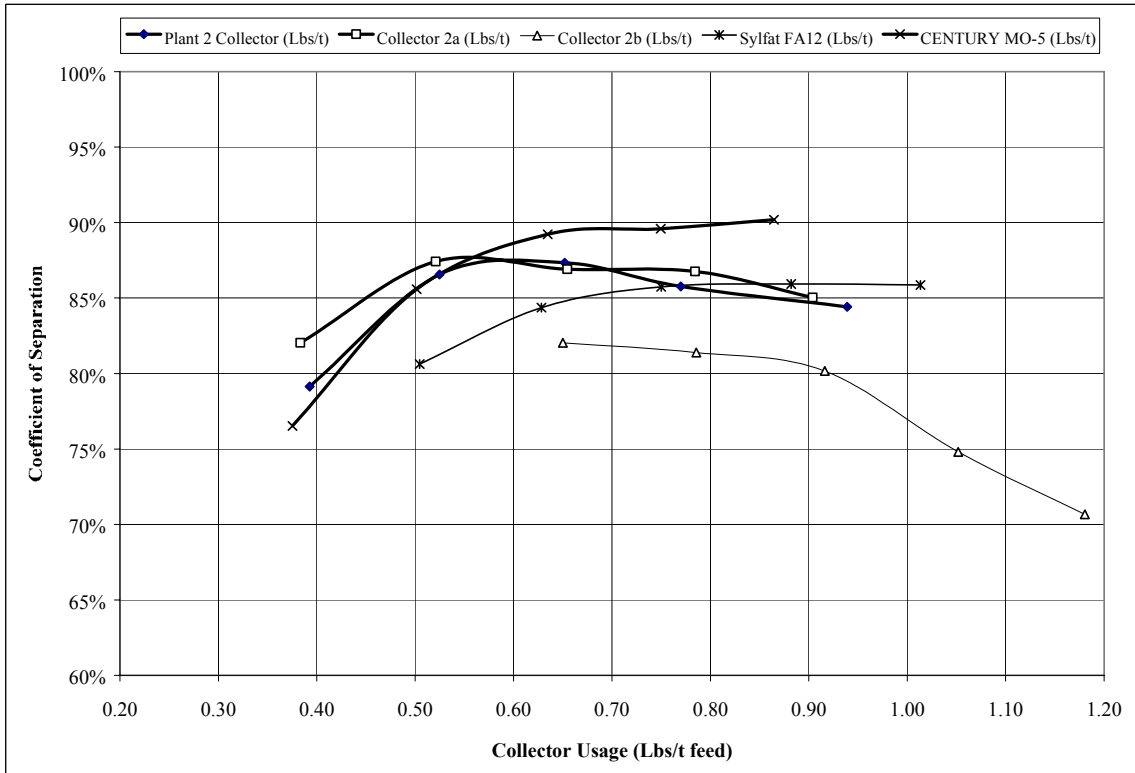


Figure 9. Collector Selectivity – Plant 2 Feed.

**Table 6. Laboratory Testing of Collectors on Plant 1 Feed.**

| <b>Plant 1 Collector (Lbs/t)</b> | <b>0.25</b> | <b>0.33</b> | <b>0.50</b> | <b>0.66</b> | <b>0.84</b> |
|----------------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery                | 14.6        | 21.1        | 23.3        | 26.3        | 28.9        |
| Concentrate % BPL                | 64.4        | 57.5        | 52.9        | 48.5        | 44.7        |
| Concentrate % Insol              | 10.1        | 19.7        | 26.0        | 33.0        | 38.0        |
| BPL % Recovery                   | 70.2        | 90.2        | 92.3        | 93.4        | 95.8        |
| Insol % Recovery                 | 1.8         | 5.1         | 7.5         | 10.7        | 13.6        |
| Separation Coefficient           | 68.4        | 85.1        | 84.8        | 82.7        | 82.2        |

| <b>MO-5 Blend (Lbs/t)</b> | <b>0.76</b> | <b>0.94</b> | <b>1.13</b> | <b>1.33</b> | <b>1.49</b> |
|---------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery         | 16.0        | 20.7        | 34.0        | 41.4        | 50.7        |
| Concentrate % BPL         | 56.4        | 48.4        | 36.3        | 30.3        | 24.6        |
| Concentrate % Insol       | 20.5        | 31.6        | 48.5        | 57.7        | 65.2        |
| BPL % Recovery            | 68.5        | 75.9        | 93.0        | 94.3        | 94.8        |
| Insol % Recovery          | 4.0         | 8.1         | 20.4        | 29.5        | 40.8        |
| Separation Coefficient    | 64.5        | 67.8        | 72.6        | 64.9        | 54.1        |

| <b>FA12 Blend (Lbs/t)</b> | <b>0.56</b> | <b>0.74</b> | <b>0.93</b> | <b>1.11</b> | <b>1.32</b> |
|---------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery         | 19.4        | 28.6        | 32.1        | 31.6        | 32.5        |
| Concentrate % BPL         | 57.1        | 42.6        | 39.5        | 40.5        | 39.5        |
| Concentrate % Insol       | 20.0        | 40.2        | 44.8        | 42.9        | 44.1        |
| BPL % Recovery            | 81.6        | 93.1        | 95.1        | 96.2        | 97.2        |
| Insol % Recovery          | 4.8         | 14.1        | 17.8        | 16.8        | 17.7        |
| Separation Coefficient    | 76.7        | 79.0        | 77.3        | 79.5        | 79.5        |

| <b>CENTURY MO-5 (Lbs/t)</b> | <b>0.36</b> | <b>0.54</b> | <b>0.72</b> | <b>0.91</b> | <b>1.09</b> |
|-----------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery           | 12.9        | 16.6        | 21.1        | 25.1        | 26.8        |
| Concentrate % BPL           | 64.7        | 63.4        | 58.4        | 50.8        | 47.6        |
| Concentrate % Insol         | 8.2         | 10.4        | 17.7        | 28.2        | 32.6        |
| BPL % Recovery              | 64.3        | 78.9        | 92.6        | 96.0        | 96.1        |
| Insol % Recovery            | 1.3         | 2.1         | 4.6         | 8.7         | 10.8        |
| Separation Coefficient      | 63.0        | 76.7        | 88.0        | 87.3        | 85.3        |

| <b>Sylfat FA12 (Lbs/t)</b> | <b>0.27</b> | <b>0.37</b> | <b>0.55</b> | <b>0.73</b> | <b>0.93</b> |
|----------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery          | 14.9        | 19.6        | 20.5        | 21.4        | 24.6        |
| Concentrate % BPL          | 65.1        | 62.7        | 60.6        | 59.1        | 53.3        |
| Concentrate % Insol        | 7.8         | 12.1        | 14.3        | 16.8        | 25.1        |
| BPL % Recovery             | 73.0        | 90.6        | 93.4        | 95.0        | 96.9        |
| Insol % Recovery           | 1.4         | 2.9         | 3.6         | 4.4         | 7.7         |
| Separation Coefficient     | 71.6        | 87.7        | 89.8        | 90.5        | 89.3        |

**Table 7. Laboratory Testing of Collectors on Plant 2 Feed.**

| <b>Plant 2 Collector (Lbs/t)</b> | <b>0.39</b> | <b>0.52</b> | <b>0.65</b> | <b>0.77</b> | <b>0.94</b> |
|----------------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery                | 23.5        | 27.2        | 29.4        | 33.2        | 32.6        |
| Concentrate % BPL                | 65.8        | 62.1        | 61.0        | 56.3        | 53.2        |
| Concentrate % Insol              | 9.2         | 12.7        | 16.0        | 21.6        | 25.7        |
| % BPL Recovery                   | 82.1        | 91.2        | 93.8        | 95.7        | 95.7        |
| % A.I. Recovery                  | 3.0         | 4.7         | 6.4         | 9.9         | 11.3        |
| Separation Coefficient           | 79.1        | 86.6        | 87.3        | 85.8        | 84.4        |

| <b>No. 151 (Lbs/t)</b> | <b>0.38</b> | <b>0.52</b> | <b>0.65</b> | <b>0.78</b> | <b>0.90</b> |
|------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery      | 24.0        | 28.1        | 31.3        | 32.5        | 34.2        |
| Concentrate % BPL      | 66.1        | 62.4        | 57.6        | 56.3        | 53.9        |
| Concentrate % Insol    | 7.8         | 14.0        | 20.3        | 21.5        | 25.3        |
| % BPL Recovery         | 84.6        | 92.8        | 95.6        | 96.3        | 96.8        |
| % A.I. Recovery        | 2.5         | 5.4         | 8.7         | 9.6         | 11.8        |
| Separation Coefficient | 82.0        | 87.4        | 86.9        | 86.8        | 85.0        |

| <b>No. 225 (Lbs/t)</b> | <b>0.65</b> | <b>0.79</b> | <b>0.92</b> | <b>1.05</b> | <b>1.18</b> |
|------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery      | 28.5        | 33.1        | 36.3        | 40.9        | 44.6        |
| Concentrate % BPL      | 58.3        | 53.1        | 50.2        | 43.9        | 40.6        |
| Concentrate % Insol    | 18.2        | 25.6        | 30.8        | 38.8        | 42.9        |
| % BPL Recovery         | 89.1        | 93.0        | 95.5        | 96.5        | 96.8        |
| % A.I. Recovery        | 7.1         | 11.6        | 15.3        | 21.6        | 26.2        |
| Separation Coefficient | 82.0        | 81.4        | 80.2        | 74.8        | 70.7        |

| <b>Sylfat FA12 (Lbs/t)</b> | <b>0.50</b> | <b>0.63</b> | <b>0.75</b> | <b>0.88</b> | <b>1.01</b> |
|----------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery          | 24.6        | 26.9        | 28.5        | 31.7        | 31.8        |
| Concentrate % BPL          | 64.4        | 62.4        | 60.4        | 57.2        | 57.0        |
| Concentrate % Insol        | 10.2        | 13.0        | 15.9        | 20.0        | 20.3        |
| % BPL Recovery             | 84.1        | 89.2        | 91.9        | 94.6        | 94.7        |
| % A.I. Recovery            | 3.4         | 4.8         | 6.2         | 8.7         | 8.9         |
| Separation Coefficient     | 80.6        | 84.4        | 85.7        | 85.9        | 85.9        |

| <b>CENTURY MO-5 (Lbs/t)</b> | <b>0.38</b> | <b>0.50</b> | <b>0.63</b> | <b>0.75</b> | <b>0.86</b> |
|-----------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery           | 21.9        | 25.5        | 28.0        | 29.1        | 30.0        |
| Concentrate % BPL           | 67.0        | 64.8        | 63.2        | 62.4        | 60.6        |
| Concentrate % Insol         | 7.5         | 9.5         | 12.3        | 13.8        | 15.4        |
| % BPL Recovery              | 78.7        | 88.9        | 93.9        | 95.1        | 96.5        |
| % A.I. Recovery             | 2.2         | 3.3         | 4.7         | 5.5         | 6.3         |
| Separation Coefficient      | 76.5        | 85.6        | 89.2        | 89.6        | 90.2        |

**Table 8. Laboratory Testing of Collectors on Plant 3 Feed.**

| <b>Plant 3 Collector (Lbs/t)</b> | <b>0.43</b> | <b>0.54</b> | <b>0.65</b> | <b>0.75</b> | <b>0.86</b> |
|----------------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery                | 22.4        | 23.9        | 24.1        | 25.9        | 26.4        |
| Concentrate % BPL                | 65.7        | 63.3        | 61.7        | 61.5        | 61.1        |
| Concentrate % Insol              | 9.0         | 11.9        | 14.2        | 14.5        | 14.7        |
| % BPL Recovery                   | 84.2        | 91.9        | 93.7        | 94.2        | 94.6        |
| % A.I. Recovery                  | 2.0         | 3.1         | 3.8         | 3.9         | 4.0         |
| Separation Coefficient           | 82.2        | 88.9        | 89.9        | 90.3        | 90.6        |

| <b>CENTURY MO-5 (Lbs/t)</b> | <b>0.37</b> | <b>0.49</b> | <b>0.62</b> | <b>0.74</b> | <b>0.86</b> |
|-----------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery           | 16.3        | 19.8        | 21.4        | 22.0        | 22.8        |
| Concentrate % BPL           | 65.4        | 63.0        | 61.4        | 60.3        | 59.2        |
| Concentrate % Insol         | 8.1         | 11.5        | 13.4        | 15.1        | 16.8        |
| % BPL Recovery              | 76.7        | 89.2        | 93.6        | 94.4        | 95.6        |
| % A.I. Recovery             | 1.7         | 2.9         | 3.6         | 4.2         | 4.8         |
| Separation Coefficient      | 75.1        | 86.4        | 89.9        | 90.3        | 90.8        |

| <b>Sylfat FA12 (Lbs/t)</b> | <b>0.37</b> | <b>0.50</b> | <b>0.62</b> | <b>0.74</b> | <b>0.86</b> |
|----------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery          | 15.5        | 19.8        | 20.4        | 21.8        | 22.7        |
| Concentrate % BPL          | 65.2        | 62.5        | 61.5        | 60.2        | 58.8        |
| Concentrate % Insol        | 8.6         | 12.2        | 13.1        | 15.3        | 17.5        |
| % BPL Recovery             | 72.4        | 89.4        | 90.5        | 94.4        | 95.3        |
| % A.I. Recovery            | 1.7         | 3.0         | 3.3         | 4.2         | 5.0         |
| Separation Coefficient     | 70.7        | 86.4        | 87.1        | 90.2        | 90.3        |

| <b>No. 151 (Lbs/t)</b> | <b>0.39</b> | <b>0.52</b> | <b>0.64</b> | <b>0.77</b> | <b>0.90</b> |
|------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery      | 16.1        | 19.8        | 21.3        | 22.4        | 23.1        |
| Concentrate % BPL      | 64.1        | 62.1        | 59.4        | 58.6        | 56.4        |
| Concentrate % Insol    | 9.3         | 12.0        | 14.8        | 17.3        | 18.3        |
| % BPL Recovery         | 76.9        | 89.2        | 92.1        | 94.1        | 95.1        |
| % A.I. Recovery        | 1.9         | 3.0         | 4.0         | 4.9         | 5.3         |
| Separation Coefficient | 75.0        | 86.2        | 88.1        | 89.3        | 89.8        |

| <b>No. 225 (Lbs/t)</b> | <b>0.38</b> | <b>0.51</b> | <b>0.63</b> | <b>0.76</b> | <b>0.90</b> |
|------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery      | 16.0        | 20.1        | 21.1        | 22.2        | 22.9        |
| Concentrate % BPL      | 64.6        | 61.7        | 60.8        | 59.0        | 57.8        |
| Concentrate % Insol    | 8.3         | 12.0        | 13.5        | 16.2        | 17.3        |
| % BPL Recovery         | 75.3        | 90.4        | 93.1        | 94.4        | 95.1        |
| % A.I. Recovery        | 1.7         | 3.0         | 3.6         | 4.5         | 5.0         |
| Separation Coefficient | 73.7        | 87.4        | 89.5        | 89.9        | 90.1        |

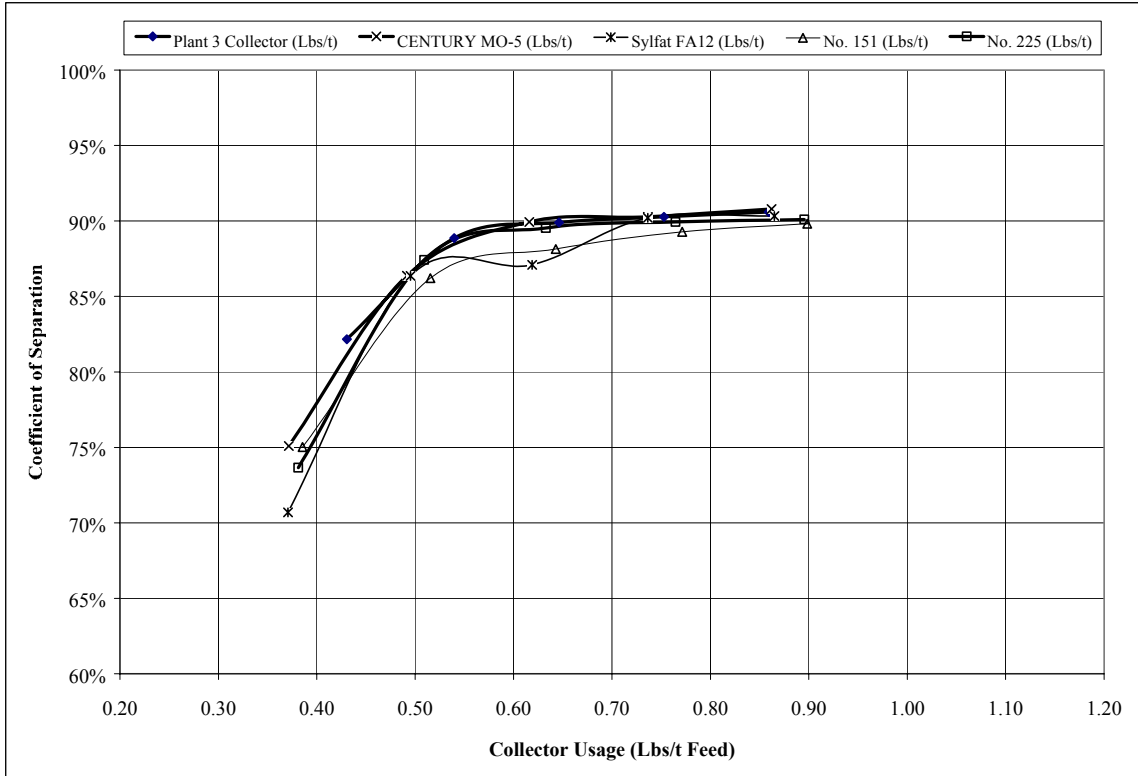


Figure 10. Collector Selectivity – Plant 3 Feed.

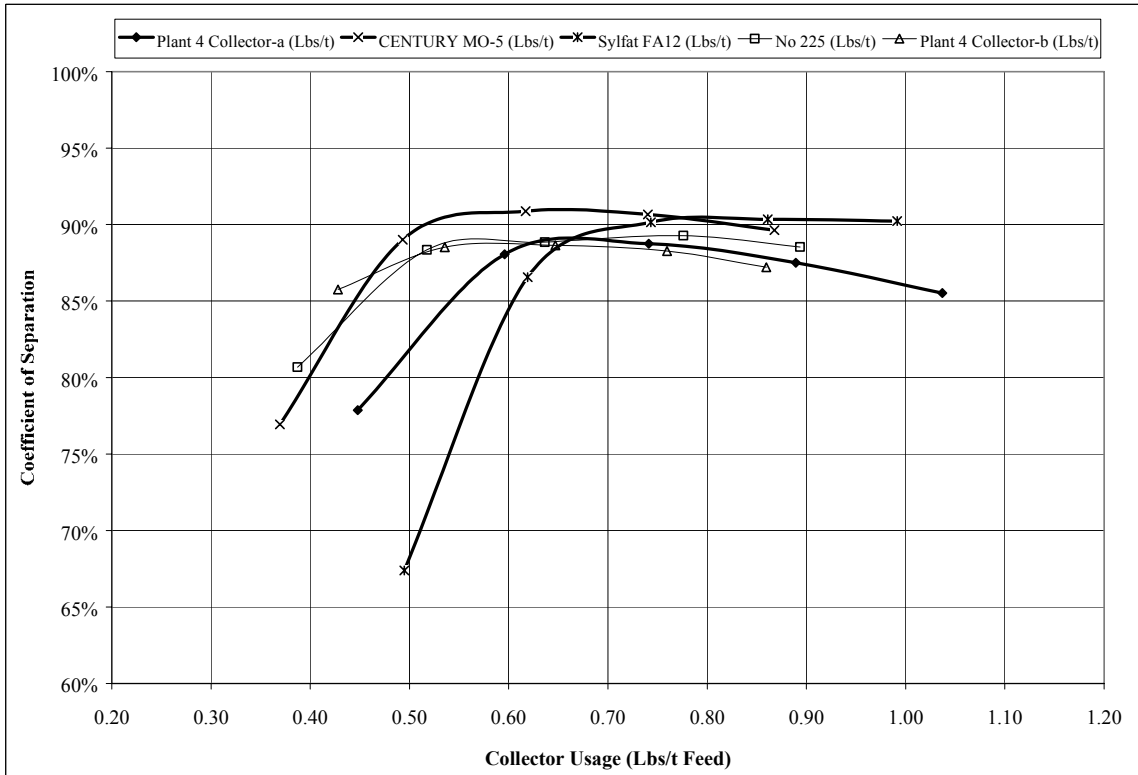


Figure 11. Collector Selectivity – Plant 4 Feed.



**Table 9. Laboratory Testing of Collectors on Plant 4 Feed.**

| <b>Plant 4 Collector (Lbs/t)</b> | <b>0.45</b> | <b>0.60</b> | <b>0.74</b> | <b>0.89</b> | <b>1.04</b> |
|----------------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery                | 16.0        | 19.6        | 22.0        | 24.2        | 26.6        |
| Concentrate % BPL                | 62.8        | 58.9        | 54.4        | 50.9        | 46.3        |
| Concentrate % Insol              | 11.4        | 16.9        | 22.9        | 28.7        | 34.6        |
| % BPL Recovery                   | 80.1        | 92.1        | 94.9        | 96.0        | 96.8        |
| % A.I. Recovery                  | 2.2         | 4.0         | 6.2         | 8.5         | 11.3        |
| Separation Coefficient           | 77.9        | 88.1        | 88.7        | 87.5        | 85.5        |

| <b>CENTURY MO-5 (Lbs/t)</b> | <b>0.37</b> | <b>0.49</b> | <b>0.62</b> | <b>0.74</b> | <b>0.87</b> |
|-----------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery           | 15.1        | 18.9        | 20.0        | 21.8        | 23.0        |
| Concentrate % BPL           | 65.0        | 61.9        | 59.6        | 56.0        | 53.2        |
| Concentrate % Insol         | 8.5         | 13.1        | 15.5        | 20.6        | 25.0        |
| % BPL Recovery              | 78.5        | 92.0        | 94.7        | 96.2        | 96.7        |
| % A.I. Recovery             | 1.6         | 3.0         | 3.8         | 5.5         | 7.0         |
| Separation Coefficient      | 76.9        | 89.0        | 90.9        | 90.7        | 89.6        |

| <b>Sylfat FA12 (Lbs/t)</b> | <b>0.49</b> | <b>0.62</b> | <b>0.74</b> | <b>0.86</b> | <b>0.99</b> |
|----------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery          | 13.3        | 17.9        | 19.8        | 20.9        | 22.2        |
| Concentrate % BPL          | 64.1        | 62.3        | 59.5        | 57.7        | 55.0        |
| Concentrate % Insol        | 8.2         | 12.6        | 17.3        | 24.0        | 29.4        |
| % BPL Recovery             | 68.7        | 89.2        | 94.0        | 95.2        | 96.3        |
| % A.I. Recovery            | 1.4         | 2.6         | 3.8         | 4.8         | 6.1         |
| Separation Coefficient     | 67.4        | 86.6        | 90.1        | 90.3        | 90.2        |

| <b>No 225 (Lbs/t)</b>  | <b>0.39</b> | <b>0.52</b> | <b>0.64</b> | <b>0.78</b> | <b>0.89</b> |
|------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery      | 16.4        | 19.4        | 19.2        | 21.2        | 23.3        |
| Concentrate % BPL      | 62.5        | 59.5        | 60.3        | 56.6        | 52.0        |
| Concentrate % Insol    | 9.2         | 14.9        | 13.0        | 19.6        | 25.3        |
| % BPL Recovery         | 82.5        | 91.9        | 91.9        | 94.4        | 95.7        |
| % A.I. Recovery        | 1.8         | 3.5         | 3.1         | 5.1         | 7.2         |
| Separation Coefficient | 80.7        | 88.3        | 88.9        | 89.3        | 88.5        |

| <b>Plant 4 Collector-b (Lbs/t)</b> | <b>0.43</b> | <b>0.54</b> | <b>0.65</b> | <b>0.76</b> | <b>0.86</b> |
|------------------------------------|-------------|-------------|-------------|-------------|-------------|
| % Weight Recovery                  | 18.4        | 20.3        | 22.1        | 23.2        | 24.9        |
| Concentrate % BPL                  | 62.1        | 58.1        | 55.7        | 53.4        | 50.2        |
| Concentrate % Insol                | 13.1        | 18.3        | 22.5        | 25.6        | 30.1        |
| % BPL Recovery                     | 88.7        | 93.1        | 94.8        | 95.5        | 96.4        |
| % A.I. Recovery                    | 2.9         | 4.6         | 6.1         | 7.3         | 9.2         |
| Separation Coefficient             | 85.8        | 88.5        | 88.6        | 88.3        | 87.2        |

## PILOT PLANT TESTING

According to the contractual scope of work, feed samples from two plants would be tested in the pilot plant. Plant 1 and Plant 4 were selected. Expenditures for collecting and testing these two samples were below budget because of excellent cooperation from the operating companies and fewer problems with pilot plant start up and operation than envisioned. Jacobs subsequently requested permission to test a third sample within the original budget. Approval was granted, and Plant 3 was selected.

### Plant 1 Feed

One informal run and 10 formal runs were performed on this feed. The Crago process was tested in four runs, two with laboratory water and two with plant process water. Anionic process option 4 was examined in three runs, two with laboratory water and one with plant process water. Anionic process option 5 was also examined in three runs, two with laboratory water and one with plant process water. The material balances for these 10 formal pilot plant runs are included in Appendix A and a summary of the runs is presented in Table 10. Runs 1 through 6 were performed with the first load of feed and runs 7 through 11 were performed with the second load.

We were unable to duplicate the laboratory flotation performance achieved with the initial sample from this plant; however, based on informal discussions, our pilot plant results were in line with plant performance data around the time the bulk sample was obtained. Also, the flocculant content of plant water reportedly was higher than normal around the time of sampling, which would have affected the plant water sample and may have influenced the surface chemistry of the feed samples. The four test runs performed with plant process water required more collector to maintain recovery and yielded lower grade concentrates than comparable tests with laboratory water. The grade problem was particularly evident with the anionic process options.

A 4-test average material balance for the Crago process is shown of Figure 12. The Crago process recovered 72% of the BPL in a concentrate analyzing 70% BPL and 5% Insol. Phosphate losses to the rougher and cleaner tails averaged 20% and 8%, respectively, for this refractory sample.

A 3-test average material balance for anionic process option 4 is illustrated in Figure 13. Anionic option 4 recovered 85% of the BPL in a concentrate containing 59% BPL and 20% Insol. BPL recovery losses to the rougher tail and discarded fraction of the cleaner tail averaged 13% and 2%, respectively, for Option 4.

Figure 14 gives the 3-test average material balance for anionic process option 5. As shown, 80% of the BPL was recovered in a concentrate analyzing 56% BPL and 21% Insol. Phosphate losses to the rougher and cleaner tails averaged 17% and 3%, respectively, for Option 5.

**Table 10. Summary of Plant 1 Feed Pilot Tests.**

| Run Number | Process Tested | Water Used | BPL % Recovery | Concentrate |         | AC Used | Reagent Usage (Lbs/t feed) |      |      |      |      |      |    |
|------------|----------------|------------|----------------|-------------|---------|---------|----------------------------|------|------|------|------|------|----|
|            |                |            |                | % BPL       | % Insol |         | AC                         | FO   | PM   | SD   | SA   | CC   |    |
| 1          | na             | na         | na             | na          | na      | na      | na                         | na   | na   | na   | na   | na   | na |
| 2          | Crago          | Tap        | 70.4           | 69.8        | 3.2     | plant   | 0.63                       | 0.07 | 0.57 | 0.09 | 1.34 | 0.21 |    |
| 3          | Opt. 4         | Tap        | 88.4           | 57.8        | 20.1    | FA12    | 0.90                       | 0.10 | 0.38 | -    | -    | -    |    |
| 4          | Opt. 4         | Tap        | 80.6           | 63.1        | 12.4    | FA12    | 0.62                       | 0.07 | 0.29 | 0.07 | -    | -    |    |
| 5          | Opt. 5         | Tap        | 82.1           | 54.7        | 19.4    | MO-5    | 0.82                       | 0.09 | 0.36 | -    | -    | -    |    |
| 6          | Opt. 5         | Tap        | 78.0           | 63.2        | 11.7    | MO-5    | 0.60                       | 0.07 | 0.33 | -    | -    | -    |    |
| 7          | Crago          | Tap        | 67.0           | 68.0        | 5.7     | plant   | 0.57                       | 0.06 | 0.32 | 0.16 | 1.52 | 0.14 |    |
| 8          | Crago          | Plant      | 72.9           | 70.5        | 4.0     | plant   | 0.87                       | 0.10 | 0.29 | 0.16 | 2.60 | 0.20 |    |
| 9          | Crago          | Plant      | 77.3           | 70.4        | 5.8     | plant   | 0.88                       | 0.10 | 0.29 | 0.16 | 2.35 | 0.18 |    |
| 10         | Opt. 4         | Plant      | 85.5           | 54.6        | 26.7    | FA12    | 0.76                       | 0.08 | 0.29 | 0.14 | -    | -    |    |
| 11         | Opt. 5         | Plant      | 80.8           | 51.3        | 31.1    | MO-5    | 0.93                       | 0.10 | 0.33 | 0.16 | -    | -    |    |

Notes: AC = anionic collector, FO = fuel oil, PM = pH modifier (soda ash), SD = silica depressant (sodium silicate)  
 SA = sulfuric acid, CC = cationic collector (plant amine)

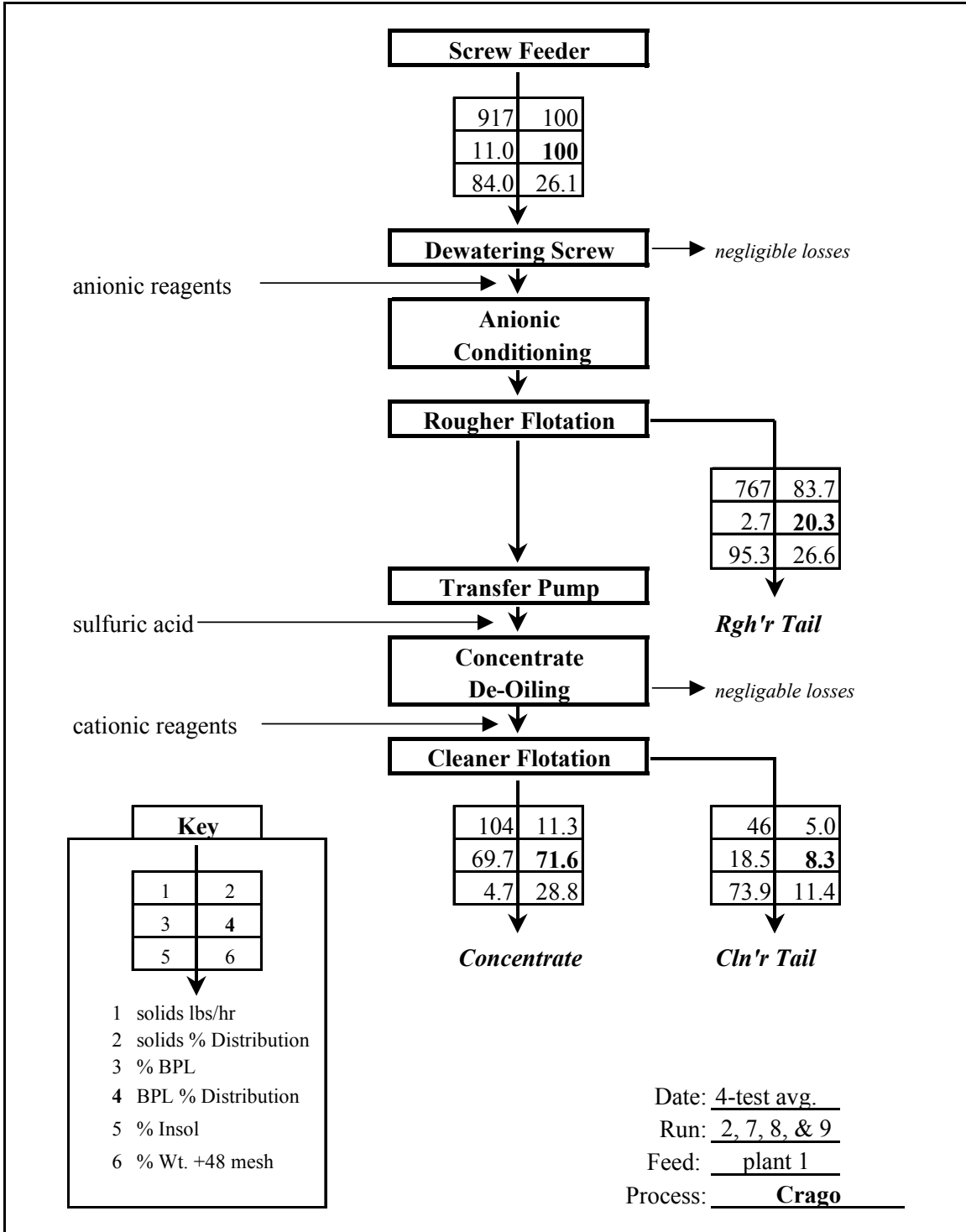


Figure 12. Crago Process Results (Plant 1 Feed).

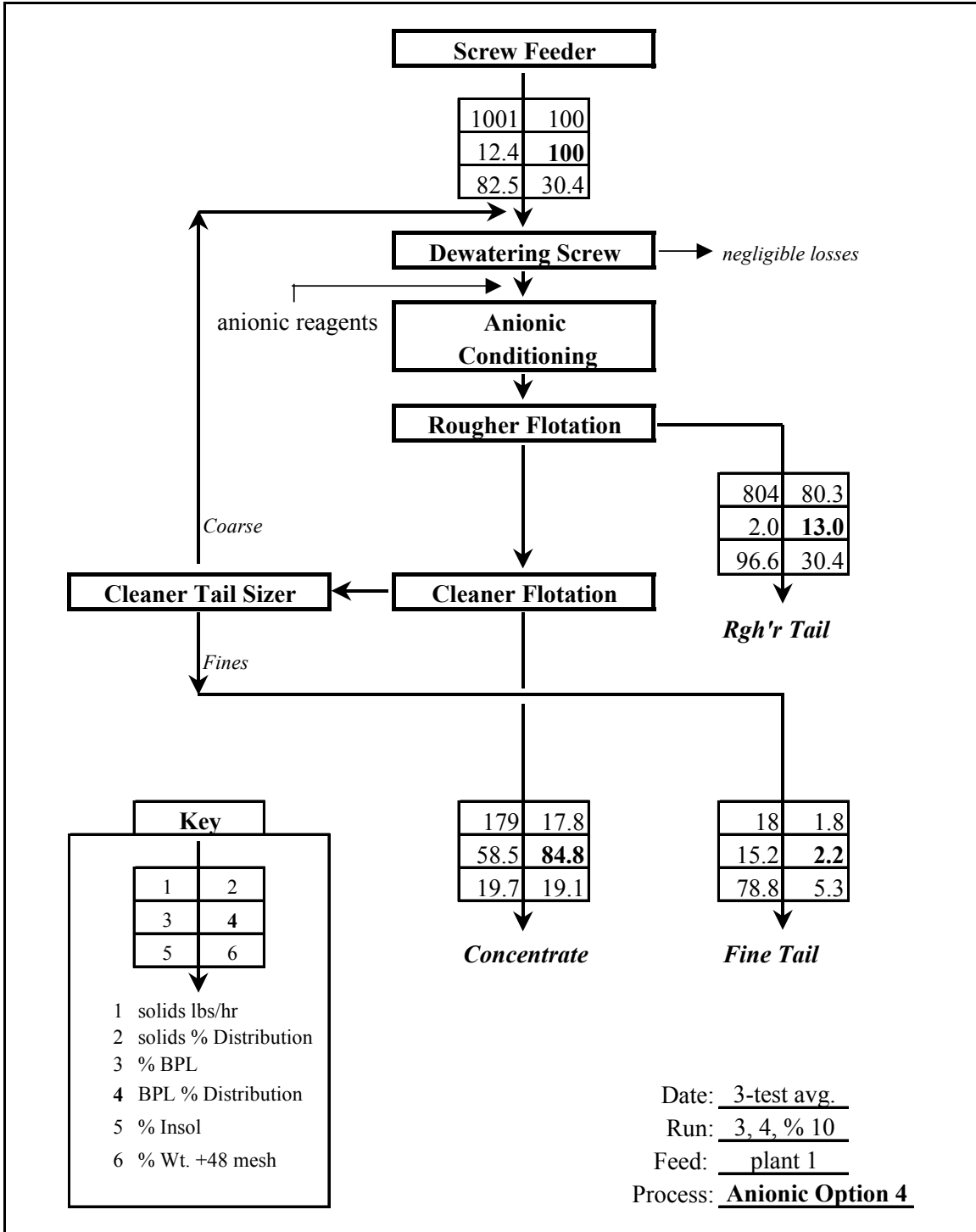


Figure 13. Anionic Process Option 4 Results (Plant 1 Feed).

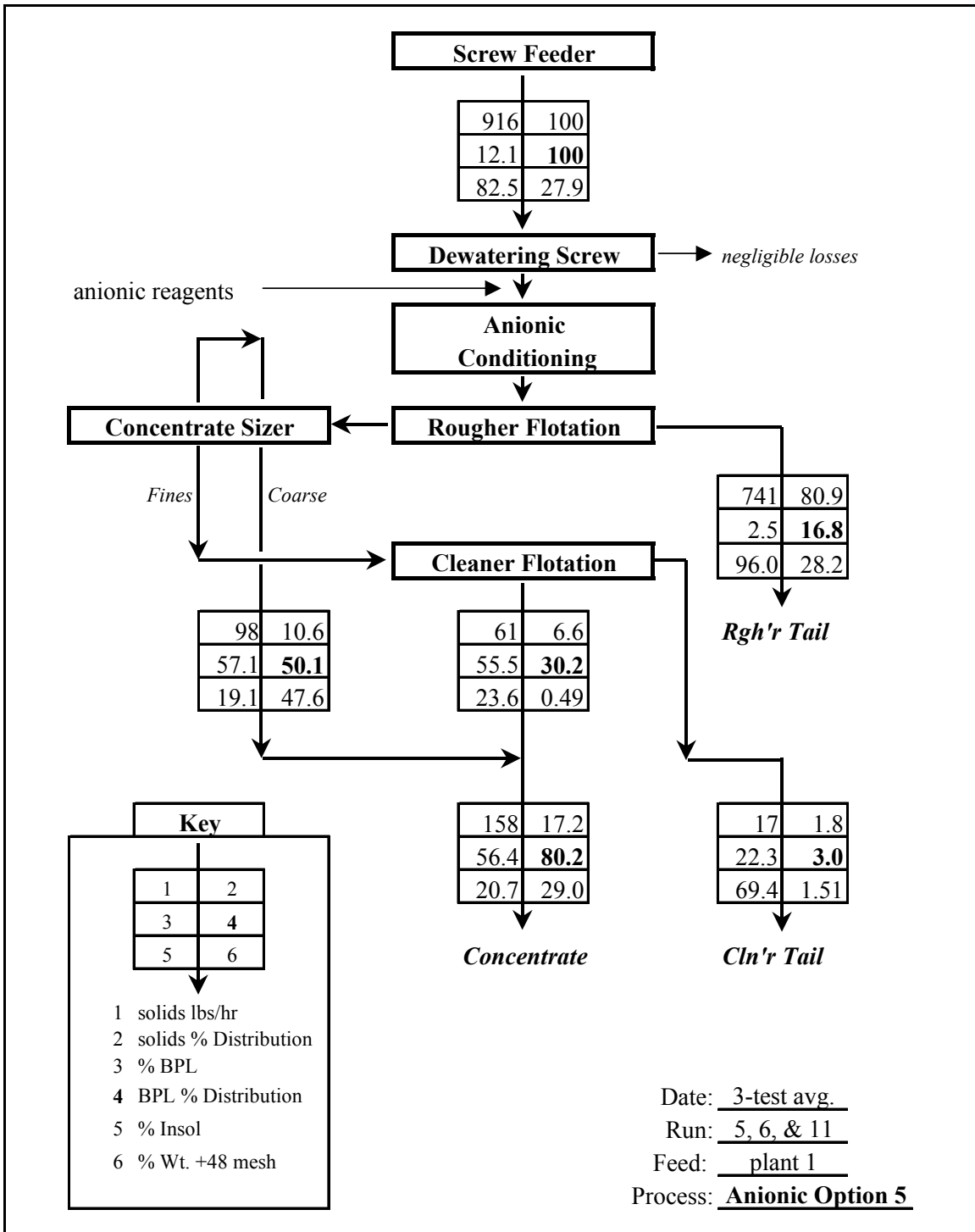


Figure 14. Anionic Process Option 5 Results (Plant 1 Feed).

The anionic process options were able to recover 8 to 13% more phosphate from Plant 1 feed than the Crago process; however, their concentrates were significantly more diluted by Insol. Option 4 gave higher recovery and slightly better concentrate grade than Option 5. The lowest recovery obtained with Option 4 was 81%, with a concentrate containing 63% BPL and 12% Insol. A pilot scale Derrick screen was used for sizing cleaner tails (Option 4) and rougher concentrate (Option 5).

### **Plant 3 Feed**

This sample was collected and tested within the approved budget but after the contractual scope of test work had been completed. Sixteen formal runs were performed, all with the same feed sample, using laboratory water only. The Crago process was tested in two runs to establish a basis of comparison. Anionic process options 4 and 5 were each examined in five runs. At the request of the Florida Institute of Phosphate Research anionic process option 1 was also tested, with one run for fine feed and three runs for coarse feed. The material balances for these 16 formal pilot plant runs are included in Appendix B and a summary of the runs is presented in Table 11.

A 2-test average material balance for the Crago process is shown of Figure 15. The Crago process recovered 86% of the BPL in a concentrate analyzing 70% BPL and 4% Insol. BPL recovery losses to the rougher and cleaner tails averaged 6% and 9%, respectively, for Plant 3 feed.

Figure 16 illustrates the average material balance for anionic process option 4, which recovered 90% of the BPL in a concentrate containing 66% BPL and 9% Insol. Phosphate losses to the rougher tail averaged less than 10%, while losses to the discarded fraction of the cleaner tail averaged less than 1% for Option 4. A derrick screen was used to size the cleaner tails.

Figure 17 gives the 3-test average material balance for anionic process option 5. As shown, 87% of the BPL was recovered in a concentrate analyzing 63% BPL and 10% Insol. BPL recovery losses to the rougher and cleaner tails averaged 12% and 1%, respectively, for Option 5. A density separator was used to size the rougher concentrate.

Anionic process options 4 and 5 were able to recover 1 to 4% more phosphate from Plant 3 feed than the Crago process. The concentrates from these two processes contained from 6.5 to 11.4% Insol. Option 4 gave 3% higher recovery and slightly better concentrate grade than Option 5. Four of the runs testing Option 4 used Plant 1 collector.

**Table 11. Summary of Plant 3 Feed Pilot Tests.**

| Run Number | Process Tested | Water Used | BPL % Recovery | Concentrate |         | AC Used | Reagent Usage (Lbs/t feed) |      |      |      |      |      |
|------------|----------------|------------|----------------|-------------|---------|---------|----------------------------|------|------|------|------|------|
|            |                |            |                | % BPL       | % Insol |         | AC                         | FO   | PM   | SD   | SA   | CC   |
| 101A       | Crago          | Tap        | 87.3           | 69.6        | 3.8     | Plant 3 | 1.30                       | 0.86 | 0.66 | -    | 2.37 | 0.51 |
| 101B       | Crago          | Tap        | 84.3           | 69.6        | 3.4     | Plant 3 | 1.63                       | 1.09 | 0.74 | -    | 2.52 | 0.50 |
| 102A       | Opt. 4         | Tap        | 91.1           | 64.6        | 10.9    | Plant 1 | 1.53                       | 1.02 | 0.29 | -    | -    | -    |
| 102B       | Opt. 4         | Tap        | 90.2           | 65.7        | 9.0     | Plant 1 | 1.29                       | 0.86 | 0.25 | -    | -    | -    |
| 102C       | Opt. 4         | Tap        | 90.8           | 65.0        | 9.8     | Plant 1 | 1.02                       | 0.68 | 0.19 | -    | -    | -    |
| 102D       | Opt. 4         | Tap        | 91.2           | 66.2        | 8.6     | Plant 1 | 1.06                       | 0.71 | 0.20 | -    | -    | -    |
| 102E       | Opt. 4         | Tap        | 88.2           | 67.3        | 6.5     | FA12    | 0.98                       | 0.59 | 0.18 | -    | -    | -    |
| 103A       | Opt. 5         | Tap        | 83.1           | 63.3        | 9.8     | Blend A | 2.40                       | -    | 0.71 | 0.25 | -    | -    |
| 103B       | Opt. 5         | Tap        | 84.9           | 62.5        | 11.4    | Blend A | 2.24                       | -    | 0.68 | 0.24 | -    | -    |
| 103C       | Opt. 5         | Tap        | 88.8           | 63.4        | 10.1    | Blend A | 1.34                       | 0.81 | 0.68 | 0.24 | -    | -    |
| 103D       | Opt. 5         | Tap        | 88.5           | 63.4        | 11.1    | Blend A | 1.36                       | 0.81 | 0.57 | 0.32 | -    | -    |
| 103E       | Opt. 5         | Tap        | 89.4           | 64.1        | 10.0    | FA12    | 1.46                       | 0.88 | 0.61 | 0.25 | -    | -    |
| 104A       | Opt. 1,F       | Tap        | 93.0           | 57.4        | 19.3    | FA12    | 1.65                       | 0.99 | 0.30 | 0.51 | -    | -    |
| 104B       | Opt. 1,C       | Tap        | 88.7           | 61.7        | 13.8    | FA12    | 1.81                       | 1.09 | 0.33 | -    | -    | -    |
| 104C       | Opt. 1,C       | Tap        | 89.0           | 66.3        | 8.3     | FA12    | 1.34                       | 0.80 | 0.25 | -    | -    | -    |
| 104D       | Opt. 1,C       | Tap        | 91.0           | 66.1        | 8.3     | FA12    | 1.15                       | 0.69 | 0.21 | -    | -    | -    |

Notes: AC = anionic collector, FO = fuel oil, PM = pH modifier (soda ash), SD = silica depressant (sodium silicate)

SA = sulfuric acid, CC = cationic collector (plant amine)

Blend A is a 1:1 mixture of MO-5 & Ligro GA

Option 1 is open circuit rougher-cleaner flotation. (F = fine feed, C = coarse feed)



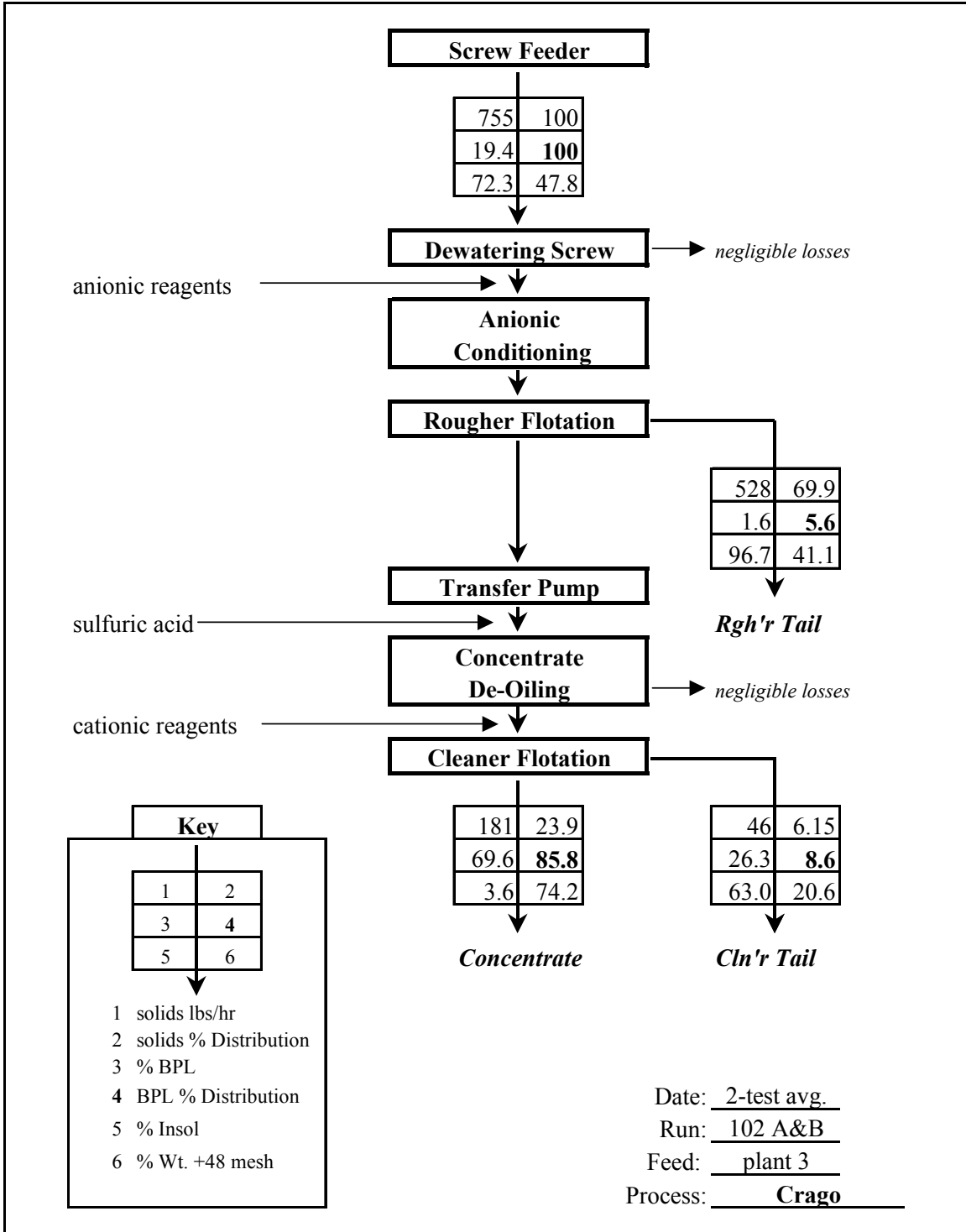


Figure 15. Crago Process Results (Plant 3 Feed).

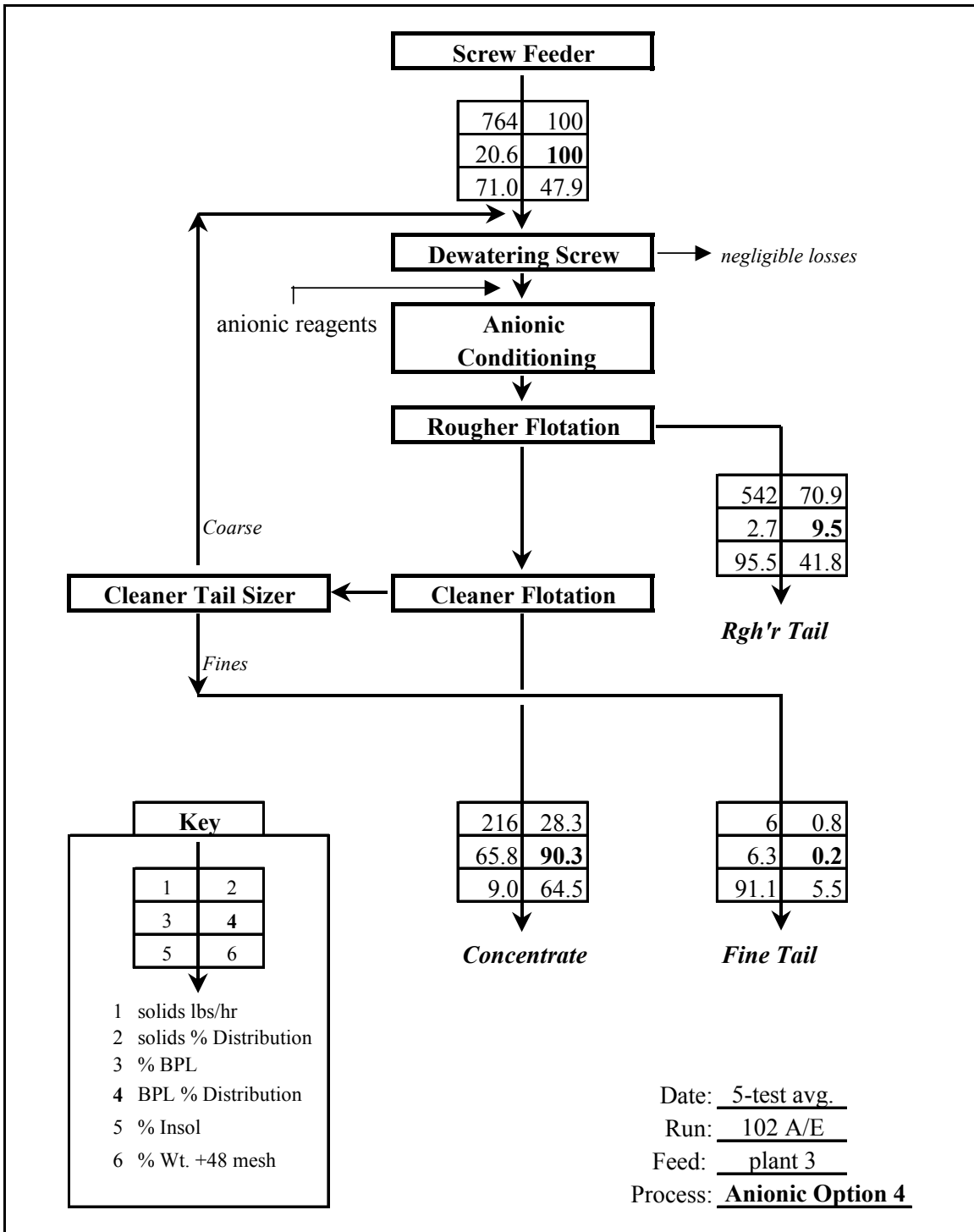


Figure 16. Anionic Process Option 4 Results (Plant 3 Feed).

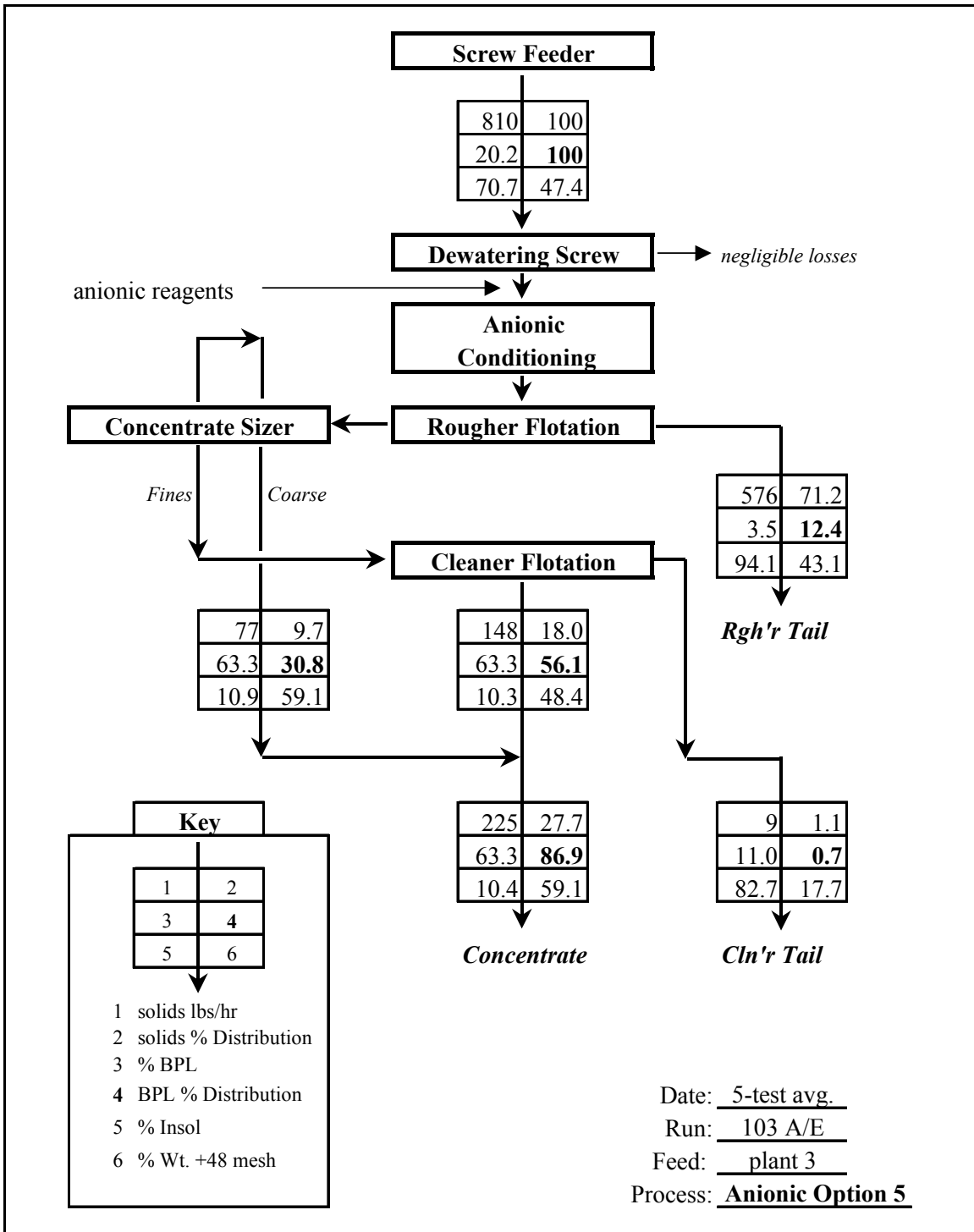


Figure 17. Anionic Process Option 5 Results (Plant 3 Feed).

Test work on anionic process option 1 involved sizing Plant 3 feed in a density separator and then performing open circuit rougher-cleaner flotation on the coarse and fine feeds. The composite performance for anionic process option 1, as shown below in Table 12, has equivalent recovery to Option 4 and equivalent grade to Option 5. Feed size distribution and the 48 mesh cut point resulted in a relatively large proportion of coarse feed. The data show that grade dilution results from Insol (silica) in the fine concentrate.

**Table 12. Composite Performance for Anionic Process Option 1.**

| Option 1 Feeds |        |        |       | BPL %<br>Recovery | Option 1 Concentrates |       |         |
|----------------|--------|--------|-------|-------------------|-----------------------|-------|---------|
| Fraction       | % >48M | Weight | % BPL |                   | Weight                | % BPL | % Insol |
| Fine           | 3.4    | 38.9   | 9.19  | 93.0              | 5.8                   | 57.37 | 19.28   |
| Coarse         | 74.1   | 61.1   | 26.32 | 89.0              | 21.6                  | 66.33 | 8.33    |
| Composite      | 46.6   | 100.0  | 19.66 | 89.7              | 27.4                  | 64.44 | 10.65   |

#### **Plant 4 Feed**

Thirteen formal runs were performed on Plant 4 feed. Runs 12 through 17 used the first truckload of feed, while runs 18 through 24 used the second truckload of feed. The Crago process was tested in two runs, one with laboratory water and one with plant process water. Anionic process option 4 was examined in seven runs, four with laboratory water and three with plant process water. Anionic process options 5 and 6 were each examined in two runs, one with laboratory water and the other with plant process water. The material balances for these 13 formal pilot plant runs are included in Appendix C and a summary of the runs is presented in Table 13.

The Crago process average material balance is shown in Figure 18. The Crago process recovered 88% of the BPL in a concentrate analyzing 69% BPL and 4% Insol. BPL recovery losses to the rougher and cleaner tails averaged 9% and 3%, respectively, for Plant 4 feed.

Figure 19 illustrates the average material balance for six anionic process option 4 tests. This process recovered 91% of the BPL in a concentrate containing 64% BPL and 10% Insol. Phosphate losses to the rougher tail averaged 7%, while losses to the discarded fraction of the cleaner tail averaged less than 3% for Option 4. For purposes of pilot plant testing, the cleaner flotation tails were sized on a Derrick screen.

Figure 20 gives the 2-test average material balance for anionic process option 5. As shown, 90% of the BPL was recovered in a concentrate analyzing 63% BPL and 11% Insol. Phosphate recovery losses to the rougher and cleaner tails averaged 6% and 4%, respectively, for Option 5. For purposes of pilot plant testing, the rougher flotation concentrate was also sized on a Derrick screen.

**Table 13. Summary of Plant 4 Feed Pilot Tests.**

| Run Number | Process Tested | Water Used | BPL % Recovery | Concentrate |         | AC Used | Reagent Usage (Lbs/t feed) |      |      |      |      |      |
|------------|----------------|------------|----------------|-------------|---------|---------|----------------------------|------|------|------|------|------|
|            |                |            |                | % BPL       | % Insol |         | AC                         | FO   | PM   | SD   | SA   | CC   |
| 12         | Crago          | Tap        | 87.9           | 69.8        | 4.1     | plant   | 0.83                       | 0.21 | 0.38 | 0.35 | 2.64 | 0.16 |
| 13         | Opt. 4         | Tap        | 91.2           | 64.2        | 11.2    | plant   | 0.94                       | 0.24 | 0.45 | 0.36 | -    | -    |
| 14         | Opt. 5         | Tap        | 88.1           | 64.2        | 11.5    | FA12    | 1.02                       | 0.26 | 0.48 | 0.39 | -    | -    |
| 15         | Opt. 4         | Tap        | 90.2           | 66.4        | 9.0     | FA12    | 1.04                       | 1.04 | 0.44 | -    | -    | -    |
| 16         | Opt. 4         | Tap        | 90.0           | 65.9        | 9.7     | plant   | 1.00                       | 0.11 | 0.34 | -    | -    | -    |
| 17         | Opt. 4         | Tap        | 82.6           | 67.5        | 8.3     | plant   | 0.89                       | 0.10 | 0.32 | -    | -    | -    |
| 18         | Crago          | Plant      | 89.4           | 67.6        | 3.6     | plant   | 0.94                       | 0.23 | 0.41 | 0.39 | 2.82 | 0.20 |
| 19         | Opt. 4         | Plant      | 91.4           | 61.7        | 10.9    | FA12    | 1.11                       | 0.28 | 0.55 | -    | -    | -    |
| 20         | Opt. 4         | Plant      | 91.5           | 64.8        | 8.4     | FA12    | 1.04                       | 0.12 | 0.49 | -    | -    | -    |
| 21         | Opt. 4         | Plant      | 90.3           | 62.8        | 10.0    | FA12    | 0.96                       | 0.30 | 0.51 | -    | -    | -    |
| 22         | Opt. 6         | Plant      | 73.1           | 64.0        | 8.2     | Blend B | 0.61                       | 0.50 | 0.80 | 0.64 | -    | -    |
| 23         | Opt. 5         | Plant      | 91.6           | 62.5        | 11.2    | FA12    | 1.28                       | 0.14 | 0.57 | 0.44 | -    | -    |
| 24         | Opt. 6         | Tap        | 87.3           | 63.4        | 10.1    | Blend B | 1.43                       | 0.89 | 0.76 | 0.93 | -    | -    |

Notes: AC = anionic collector, FO = fuel oil, PM = pH modifier (soda ash), SD = silica depressant (sodium silicate)  
 SA = sulfuric acid, CC = cationic collector (plant amine)  
 Blend B is a 1:1 mixture of FA12 & CC41601

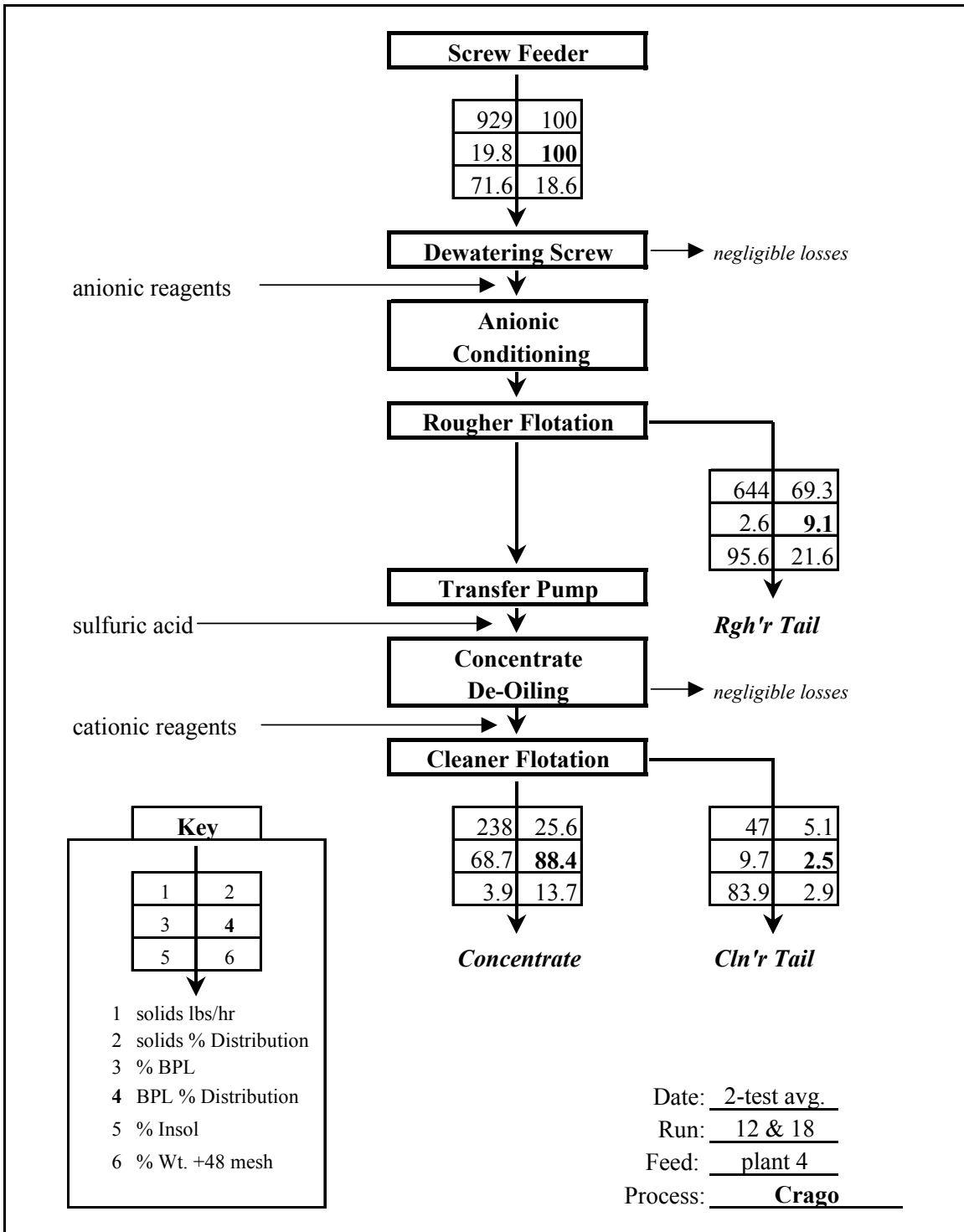


Figure 18. Crago Process Results (Plant 4 Feed).

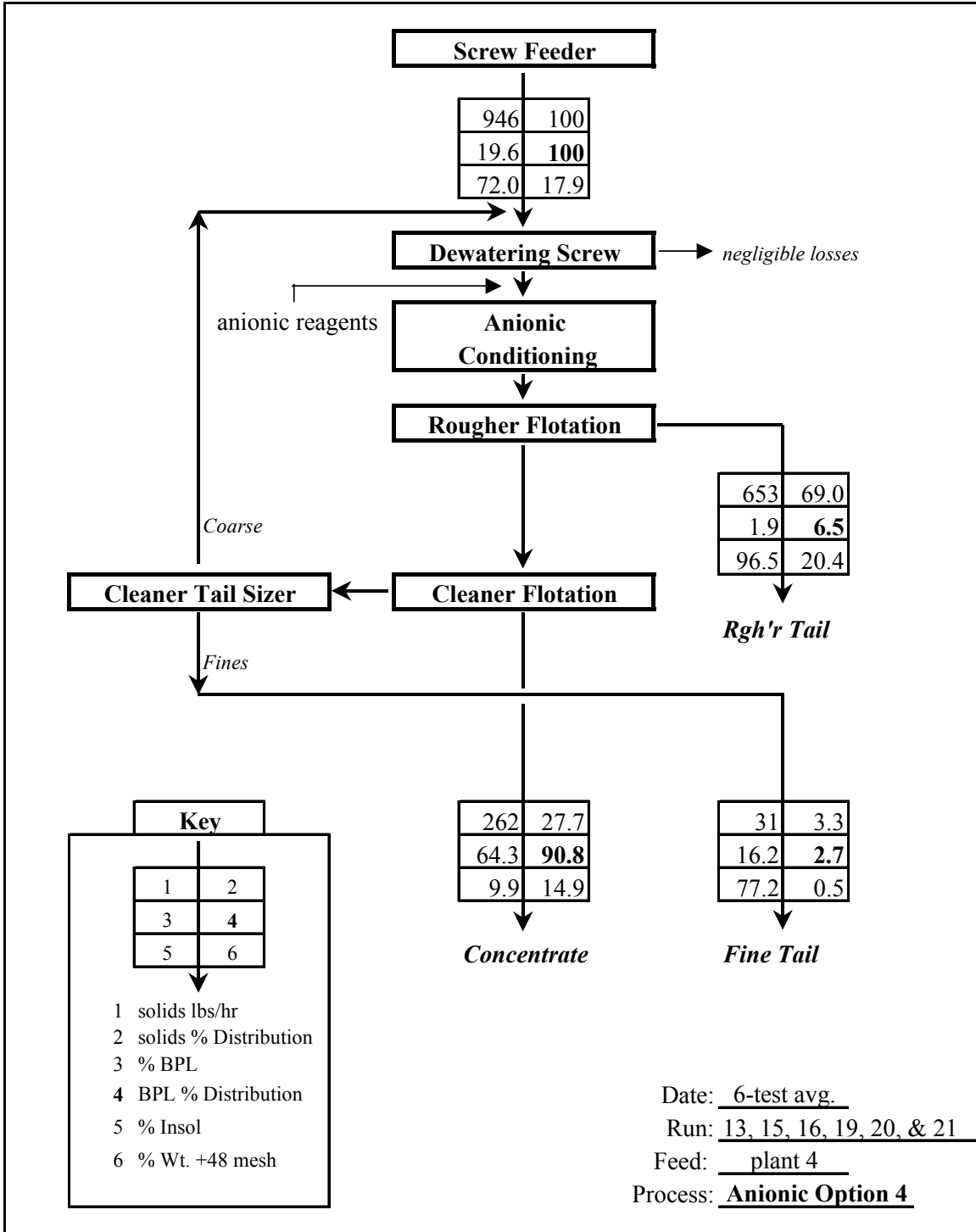


Figure 19. Anionic Process Option 4 Results (Plant 4 Feed).

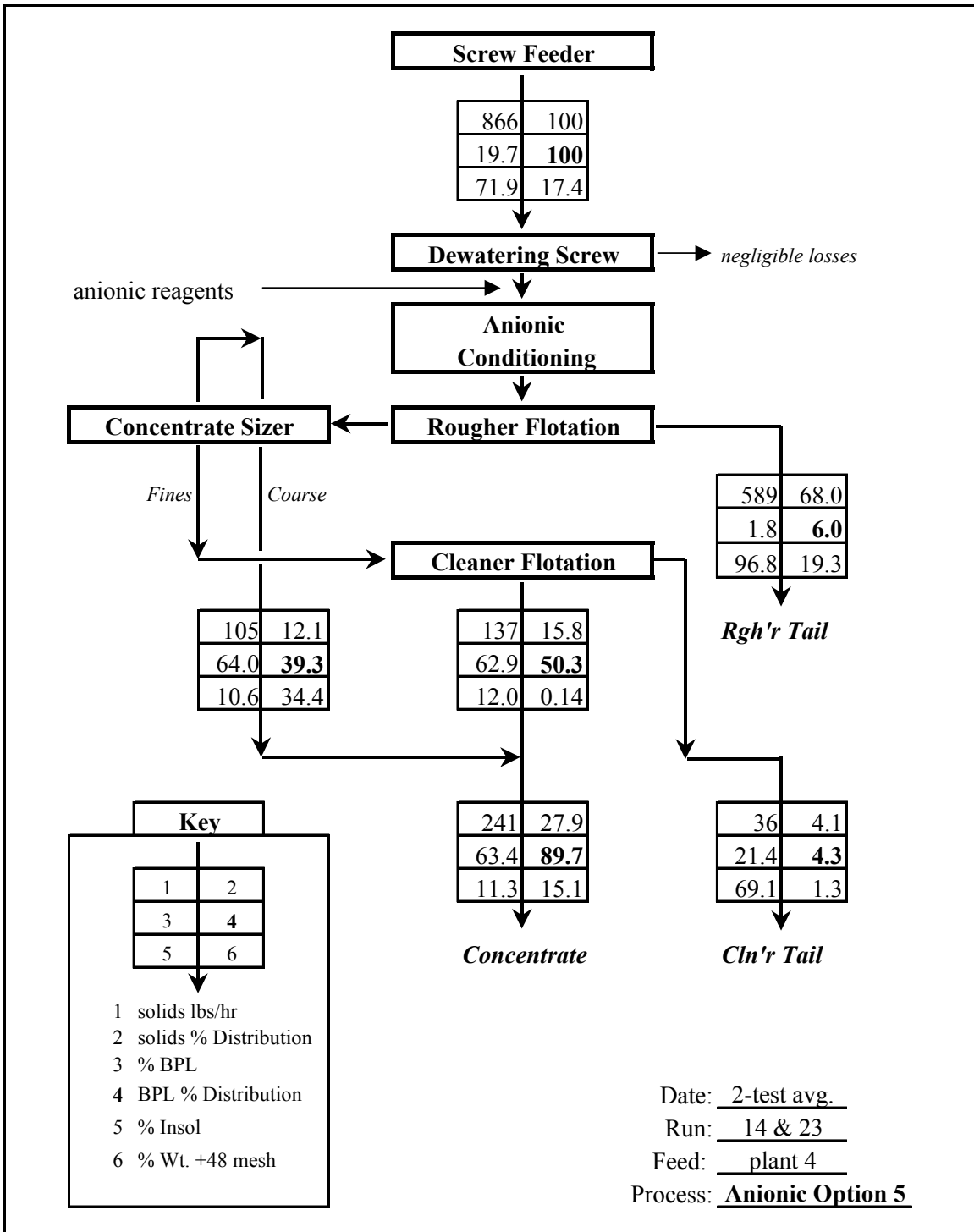


Figure 20. Anionic Process Option 5 Results (Plant 4 Feed).



The 2-test average material balance for anionic process option 6 is illustrated in Figure 21. From the viewpoint of the flotation operator, this process was the most difficult to operate. An overall BPL recovery of 80% was attained, on average, with a concentrate containing 64% BPL and 9% Insol. For purposes of pilot plant testing, the rougher (starvation) flotation tails were sized on a Derrick screen.

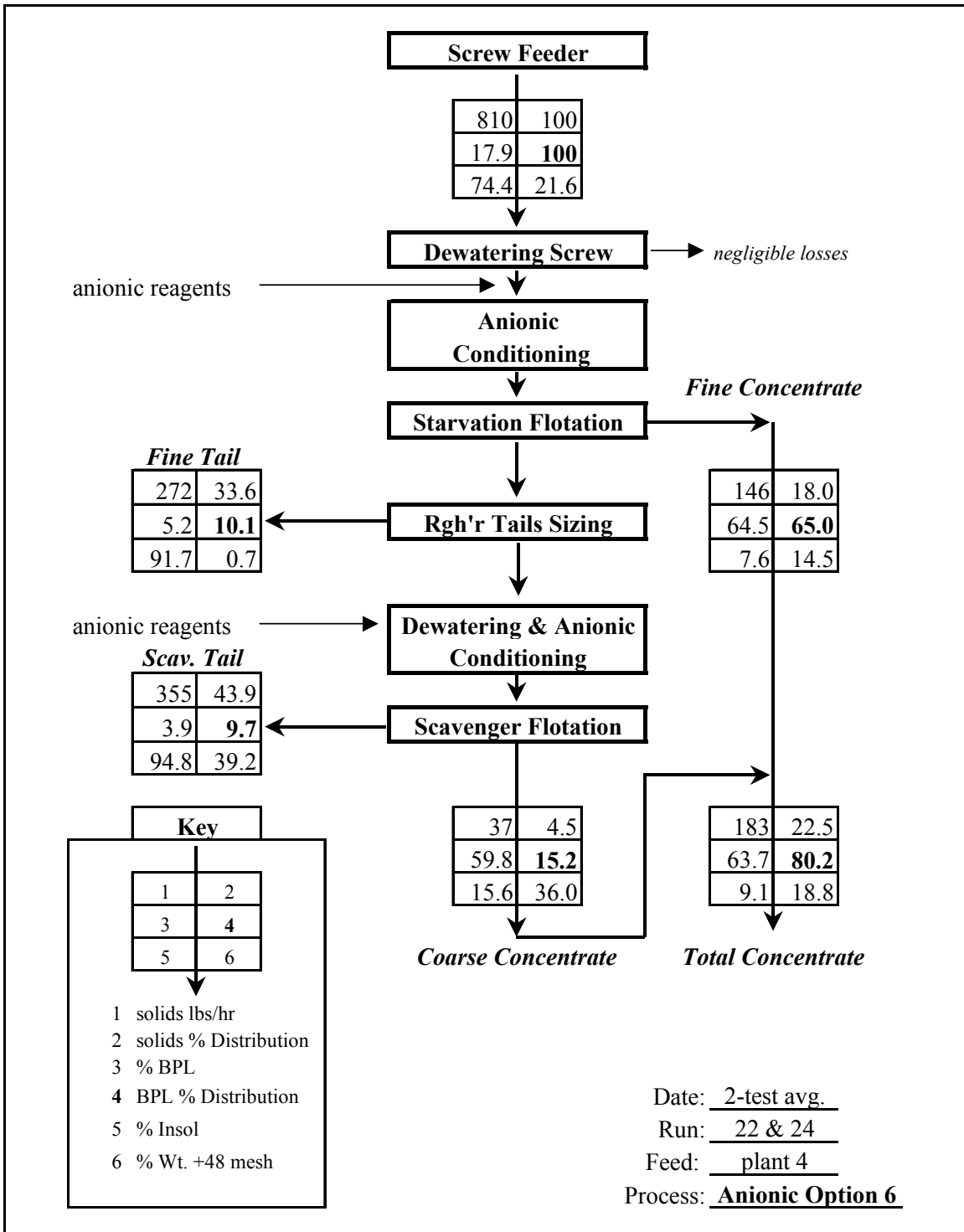
Anionic process options 4 and 5 were able to recover 2 to 3% more phosphate from Plant 4 feed than the Crago process. The concentrates from these two processes contained 8.3 to 11.5% Insol. Four of the runs testing Option 4 used Plant 4 collector.

Option 4 averaged 1% higher recovery than Option 5 and 10% higher recovery than Option 6. Option 4 was intermediate to Options 5 and 6 with regard to concentrate grade.

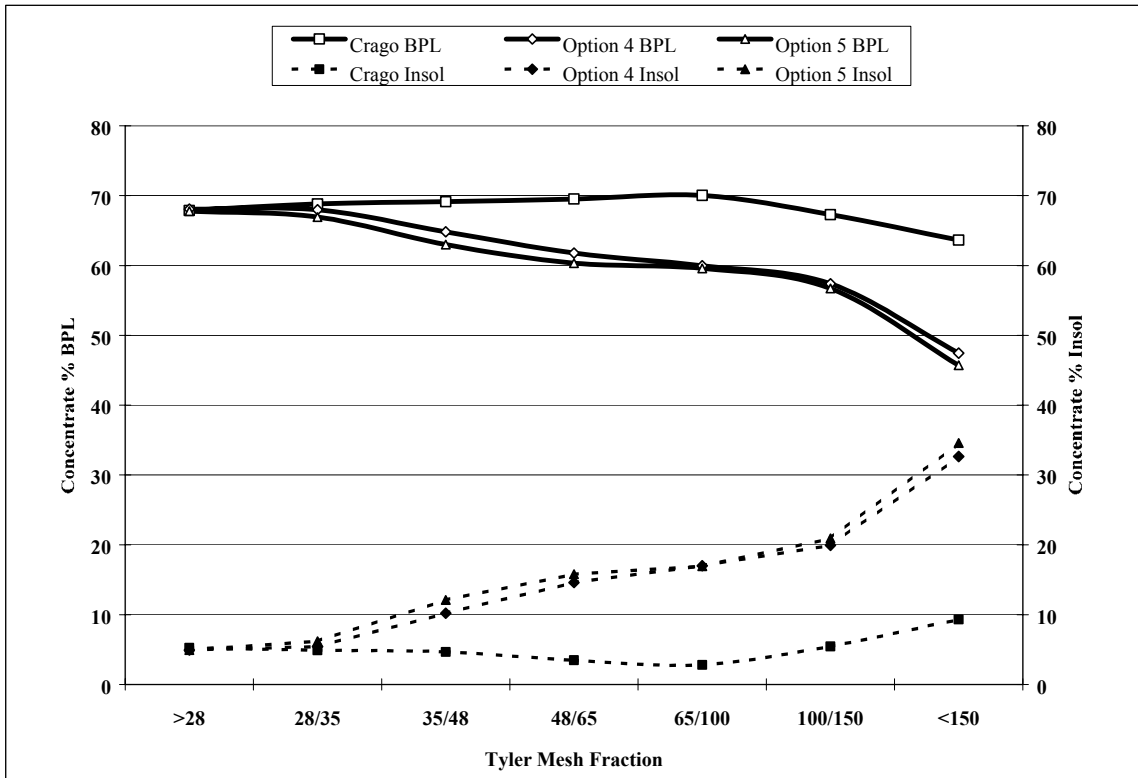
### **Particle Size Considerations**

Flotation feed preparation practices differ between operating companies. Some plants size the feed and float coarse and fine feeds separately, while others may float deslimed <14-mesh feed or <20-mesh feed. Typically particles coarser than 35-mesh are more difficult to recover in the froth product and particles finer than 65-mesh may be mechanically entrained in the froth product. Anionic (direct) flotation recovery losses result mostly from coarse grains of phosphate, while concentrate grade dilution results from fine grains of quartz. For cationic (inverse) flotation the relationship is reversed, with recovery losses from fine grains of phosphate and concentrate grade dilution from coarse grains of quartz.

The impacts of particle size on concentrate grade for the Crago process and anionic process options 4 and 5 are illustrated in Figure 22. The averaged sieve and chemical data, coinciding to the runs previously illustrated in Figures 12 through 21, are presented with equal weight for each of the three feeds tested.



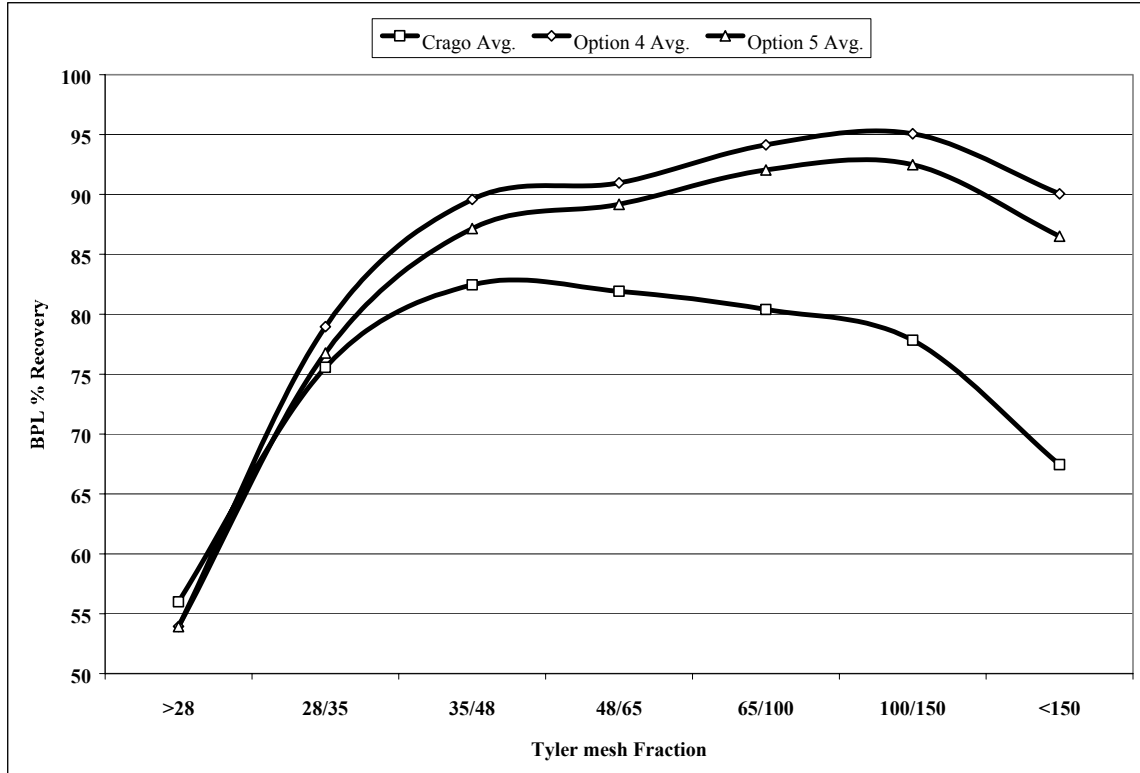
**Figure 21. Anionic Process Option 6 Results (Plant 4 Feed).**



**Figure 22. Final Concentrate Grade by Mesh Fraction.**

The Crago process provides a more uniform grade by mesh fraction than the anionic process options. The latter processes exhibit progressively lower BPL contents and higher Insol dilution as the particle size is decreased. The impact of particle size on concentrate grade is essentially identical for the two anionic processes.

Flotation recoveries corresponding to the above mentioned concentrate grade data are shown in Figure 23. The Crago process has lower recovery than the anionic process options except for the >28 mesh fraction. It is apparent that the improved grade of the Crago process is realized at the expense of lower recoveries in the finer size fractions. Anionic process option 4 maintains a recovery advantage over anionic process option 5 for almost all fractions.



**Figure 23. BPL % Recovery by Mesh Fraction.**

Anionic process options 1 and 6 were excluded from the above illustrations because they were not tested on all feeds and their data are not directly comparable. The performances of these two options were less favorable and their operation was more complicated than anionic process options 4 and 5.

### **Crago Process**

The above summary data confirm the preliminary laboratory test results showing that the Crago process is characterized by lower BPL % recovery but higher-grade concentrate than the anionic process options. Analytical and performance data, averaged for the Crago process for each of the three plant feeds are presented in Table 14. The data differs from feed to feed; however, the same trends exist for each feed.

### **Anionic Process Options 4 and 5**

Analytical and performance data, averaged for each of the three plant feeds, are presented in Tables 15 and 16 for Options 4 and 5, respectively. A comparison of these processes and the Crago process is given below.

**Rougher Flotation.** Conditioning the feed with anionic reagents and the subsequent rougher flotation step were essentially identical for all processes tested, except that Crago flotation was always performed with collector from the respective plant. Anionic option 4 also used plant collector for some tests. The BPL recovery for rougher flotation averaged about 90% for Option 4 and 88% for the other two processes.

**Cleaner Flotation.** Rougher concentrates from the anionic processes were re-floated without additional reagents to drop out sand. The Crago process used acid scrubbing and rinsing to remove reagents and water prior to cleaner flotation of the rougher concentrate with a cationic collector. The grade increase from rougher to final concentrate averaged about 11% BPL for the Crago process, 5% BPL for Option 4, and 3% BPL for Option 5. Comparing the final concentrate size fractions from Tables 15 and 16 with those in Table 14 reveals that anionic concentrate grade dilution results primarily from <48 mesh sand. Cationic flotation is more effective in removing this fine sand than re-floating the anionic concentrates.

Anionic option 5 sized the rougher concentrate and re-floated only the fine portion to drop out mechanically entrained sand. The upgrading achieved therefore depended on the efficiencies of sizing and flotation. For plant feeds 1 and 4 the rougher concentrates were sized with the pilot Derrick Screen, which recovered about 99% of the >48 mesh and 42% of the <48 mesh in the coarse concentrate. It may be argued that a sharper size separation would have improved concentrate grade; however, as shown in Figures 14 and 20, the fine cleaner concentrates for these tests had a higher Insol content than the coarse concentrates. For Plant 3 feed, the Option 5 rougher concentrates were sized with a density separator, which averaged only 43% and 20% recovery of >48 mesh and <48 mesh to the coarse concentrate, respectively. Hydraulic sizing efficiency was impaired with concentrate froth, which contained agglomerates (coarse and fine particles) having an apparent specific gravity lower than that of discrete particles.

**Recycle.** For anionic option 4, the middling recycle was controlled by sizing the cleaner tailing at nominally 48 mesh with a pilot Derrick Screen. Material passing through the screen was rejected as fine tailings, while screen oversize was recycled to the reagent conditioning tanks. This methodology maintained recovery of coarse phosphate particles and prevented fine sand from building up a circulating load. The Crago process avoids recycle streams by switching to inverse flotation for cleaning. Anionic option 5 similarly avoided the need for recycle by re-floating only the fine rougher concentrate and discarding the cleaner tails.

Phosphate losses to the rejected portion of the cleaner tailing averaged about 2% for Option 4, which compares favorably to Option 5 (about 3%) and the Crago process (about 6%).

**Table 14. Crago Process Averaged Data by Size Fraction.**

| Plant 1 Feed<br>4-test avg. | Rougher Tail |       |         | Cleaner Tail |       |         | Final Concentrate |       |         |            |
|-----------------------------|--------------|-------|---------|--------------|-------|---------|-------------------|-------|---------|------------|
|                             | % Wt.        | % BPL | % Insol | % Wt.        | % BPL | % Insol | % Wt.             | % BPL | % Insol | BPL % Rec. |
| >28                         | 1.1          | 29.5  | 58.1    | -            | -     | -       | 2.8               | 68.9  | 5.0     | 44.0       |
| 28/35                       | 5.3          | 7.0   | 89.3    | 0.9          | 17.6  | 76.0    | 7.5               | 68.7  | 6.3     | 64.8       |
| 35/48                       | 19.8         | 2.8   | 95.3    | 10.4         | 12.6  | 82.0    | 18.7              | 69.0  | 7.1     | 73.4       |
| 48/65                       | 38.3         | 1.9   | 96.8    | 34.4         | 14.6  | 78.9    | 32.5              | 70.2  | 4.7     | 75.3       |
| 65/100                      | 24.5         | 1.6   | 97.1    | 35.1         | 22.5  | 68.6    | 26.5              | 71.7  | 3.1     | 74.7       |
| 100/150                     | 8.5          | 1.8   | 96.7    | 13.2         | 27.0  | 62.4    | 9.3               | 69.6  | 4.5     | 70.6       |
| <150                        | 2.6          | 2.9   | 95.5    | 6.1          | 24.6  | 65.0    | 2.8               | 63.5  | 9.5     | 59.4       |

**Plant 3 Feed (2-test avg.)**

|         |      |      |      |      |      |      |      |      |      |      |
|---------|------|------|------|------|------|------|------|------|------|------|
| >28     | 5.6  | 18.1 | 71.1 | 0.5  | 48.2 | 31.6 | 29.1 | 66.6 | 5.2  | 86.6 |
| 28/35   | 12.1 | 1.4  | 97.3 | 3.6  | 32.7 | 62.1 | 22.8 | 68.9 | 3.7  | 95.1 |
| 35/48   | 23.3 | 0.6  | 98.5 | 16.5 | 23.9 | 66.8 | 21.0 | 69.4 | 3.1  | 91.2 |
| 48/65   | 31.4 | 0.4  | 98.3 | 34.7 | 25.9 | 63.5 | 15.1 | 69.1 | 2.3  | 79.7 |
| 65/100  | 20.2 | 0.4  | 98.7 | 31.6 | 27.5 | 60.9 | 9.3  | 69.4 | 2.3  | 72.6 |
| 100/150 | 6.4  | 0.3  | 98.7 | 10.5 | 24.2 | 65.5 | 2.5  | 67.5 | 4.3  | 70.3 |
| <150    | 1.1  | 0.4  | 98.6 | 2.6  | 17.9 | 74.2 | 0.3  | 62.3 | 11.2 | 58.1 |

**Plant 4 Feed (2-test avg.)**

|         |      |      |      |      |      |      |      |      |     |      |
|---------|------|------|------|------|------|------|------|------|-----|------|
| >28     | 0.8  | 45.0 | 30.5 | -    | -    | -    | 0.8  | 68.1 | 5.4 | 37.4 |
| 28/35   | 3.9  | 9.3  | 86.0 | -    | -    | -    | 2.8  | 68.9 | 4.6 | 66.7 |
| 35/48   | 17.2 | 3.5  | 93.9 | 3.0  | 9.5  | 81.9 | 11.8 | 69.0 | 3.8 | 82.8 |
| 48/65   | 41.3 | 1.9  | 96.3 | 21.3 | 7.1  | 87.9 | 34.3 | 69.2 | 3.4 | 90.7 |
| 65/100  | 25.5 | 1.2  | 97.3 | 47.1 | 8.3  | 86.6 | 35.6 | 69.0 | 3.1 | 93.9 |
| 100/150 | 7.5  | 0.6  | 98.2 | 19.9 | 12.0 | 77.8 | 11.5 | 64.7 | 7.6 | 92.6 |
| <150    | 4.2  | 0.7  | 97.7 | 8.8  | 17.8 | 67.7 | 3.4  | 65.1 | 7.2 | 84.9 |

**Table 15. Anionic Process Option 4 Averaged Data by Size Fraction.**

| Plant 1 Feed<br>3-test avg.       | Rougher Tail |       |         | Fine Tail |       |         | Final Concentrate |       |         |            |
|-----------------------------------|--------------|-------|---------|-----------|-------|---------|-------------------|-------|---------|------------|
|                                   | % Wt.        | % BPL | % Insol | % Wt.     | % BPL | % Insol | % Wt.             | % BPL | % Insol | BPL % Rec. |
| >28                               | 1.5          | 28.2  | 59.9    | 0.8       | 63.3  | 10.3    | 3.5               | 68.1  | 5.0     | 55.0       |
| 28/35                             | 5.6          | 6.4   | 90.1    | 1.1       | 47.8  | 33.4    | 7.3               | 66.3  | 7.6     | 74.5       |
| 35/48                             | 22.6         | 2.0   | 96.3    | 3.5       | 21.7  | 69.9    | 23.5              | 60.7  | 16.7    | 87.0       |
| 48/65                             | 46.0         | 1.1   | 97.8    | 45.8      | 13.3  | 81.6    | 31.8              | 57.4  | 21.7    | 86.0       |
| 65/100                            | 18.5         | 0.8   | 98.1    | 35.4      | 13.7  | 80.9    | 21.6              | 57.8  | 21.7    | 91.3       |
| 100/150                           | 4.2          | 1.0   | 98.0    | 9.0       | 18.4  | 74.1    | 9.8               | 56.4  | 21.7    | 93.8       |
| <150                              | 1.7          | 2.3   | 96.0    | 4.5       | 19.9  | 71.6    | 2.5               | 49.7  | 30.1    | 82.6       |
| <b>Plant 3 Feed (5-test avg.)</b> |              |       |         |           |       |         |                   |       |         |            |
| >28                               | 6.4          | 26.8  | 60.0    | 2.3       | 63.9  | 9.4     | 21.2              | 67.3  | 5.2     | 76.5       |
| 28/35                             | 12.3         | 2.8   | 95.3    | 1.4       | 49.3  | 29.1    | 20.2              | 68.5  | 4.1     | 94.0       |
| 35/48                             | 22.6         | 1.0   | 98.0    | 2.0       | 11.9  | 80.7    | 21.8              | 66.6  | 7.1     | 96.2       |
| 48/65                             | 30.4         | 0.5   | 98.4    | 36.6      | 5.1   | 91.9    | 18.5              | 63.6  | 11.5    | 96.3       |
| 65/100                            | 20.3         | 0.5   | 98.8    | 36.9      | 3.7   | 93.7    | 13.7              | 60.6  | 15.1    | 96.8       |
| 100/150                           | 6.8          | 0.4   | 99.1    | 16.4      | 2.9   | 95.0    | 4.0               | 56.8  | 20.4    | 96.5       |
| <150                              | 1.2          | 0.6   | 98.5    | 4.4       | 3.4   | 93.6    | 0.7               | 41.2  | 41.8    | 93.6       |
| <b>Plant 4 Feed (6-test avg.)</b> |              |       |         |           |       |         |                   |       |         |            |
| >28                               | 0.7          | 49.1  | 28.5    | -         | -     | -       | 0.5               | 68.7  | 4.7     | 30.0       |
| 28/35                             | 3.8          | 8.7   | 86.5    | -         | -     | -       | 2.6               | 69.1  | 4.6     | 68.4       |
| 35/48                             | 16.5         | 2.9   | 95.0    | 0.3       | 39.8  | 43.0    | 10.7              | 67.2  | 6.8     | 85.5       |
| 48/65                             | 34.5         | 1.4   | 97.1    | 27.7      | 26.1  | 62.8    | 31.4              | 64.3  | 10.6    | 90.7       |
| 65/100                            | 26.8         | 0.9   | 97.8    | 43.9      | 14.7  | 78.4    | 37.2              | 61.5  | 14.2    | 94.3       |
| 100/150                           | 13.8         | 0.6   | 98.3    | 21.0      | 9.5   | 85.4    | 13.7              | 58.9  | 17.7    | 94.9       |
| <150                              | 4.1          | 0.7   | 97.7    | 7.2       | 7.6   | 86.2    | 3.9               | 51.5  | 26.0    | 94.0       |

**Table 16. Anionic Process Option 5 Averaged Data by Size Fraction.**

| Plant 1 Feed<br>3-test avg.       | Rougher Tail |       |         | Fine Tail |       |         | Final Concentrate |       |         |            |
|-----------------------------------|--------------|-------|---------|-----------|-------|---------|-------------------|-------|---------|------------|
|                                   | % Wt.        | % BPL | % Insol | % Wt.     | % BPL | % Insol | % Wt.             | % BPL | % Insol | BPL % Rec. |
| >28                               | 1.1          | 28.6  | 59.1    | -         | -     | -       | 2.5               | 68.0  | 5.0     | 53.2       |
| 28/35                             | 5.3          | 7.0   | 89.4    | -         | -     | -       | 6.2               | 64.9  | 9.3     | 70.1       |
| 35/48                             | 21.4         | 2.6   | 95.7    | 0.9       | 38.7  | 46.4    | 19.8              | 57.4  | 21.2    | 81.2       |
| 48/65                             | 45.0         | 1.5   | 97.2    | 40.3      | 20.7  | 70.8    | 33.1              | 54.8  | 24.3    | 81.6       |
| 65/100                            | 19.3         | 1.4   | 97.4    | 38.4      | 21.3  | 70.6    | 25.3              | 57.6  | 21.4    | 87.1       |
| 100/150                           | 6.0          | 1.8   | 97.0    | 13.8      | 26.1  | 63.0    | 10.2              | 57.5  | 21.0    | 86.9       |
| <150                              | 1.9          | 2.9   | 95.0    | 6.6       | 26.1  | 62.3    | 2.8               | 48.0  | 32.7    | 75.2       |
| <b>Plant 3 Feed (5-test avg.)</b> |              |       |         |           |       |         |                   |       |         |            |
| >28                               | 7.5          | 30.6  | 54.9    | 5.0       | 63.3  | 9.0     | 18.3              | 67.0  | 4.9     | 67.0       |
| 28/35                             | 12.8         | 4.7   | 92.5    | 4.6       | 54.3  | 22.1    | 18.7              | 67.5  | 4.5     | 88.5       |
| 35/48                             | 22.5         | 1.5   | 97.0    | 8.0       | 31.7  | 53.6    | 19.4              | 65.4  | 7.8     | 93.1       |
| 48/65                             | 29.5         | 0.6   | 98.2    | 20.6      | 10.0  | 84.2    | 23.3              | 62.5  | 12.7    | 96.2       |
| 65/100                            | 19.8         | 0.5   | 98.4    | 36.0      | 4.2   | 93.3    | 15.1              | 57.9  | 17.9    | 96.7       |
| 100/150                           | 6.7          | 0.4   | 98.8    | 19.5      | 3.0   | 94.9    | 4.5               | 52.4  | 26.0    | 96.4       |
| <150                              | 1.2          | 0.5   | 98.8    | 6.4       | 2.4   | 96.2    | 0.8               | 37.7  | 44.4    | 93.0       |
| <b>Plant 4 Feed (2-test avg.)</b> |              |       |         |           |       |         |                   |       |         |            |
| >28                               | 0.6          | 46.5  | 32.0    | -         | -     | -       | 0.8               | 68.4  | 4.9     | 41.4       |
| 28/35                             | 3.5          | 8.3   | 87.3    | 0.1       | 63.7  | 12.6    | 2.7               | 68.5  | 4.8     | 71.5       |
| 35/48                             | 15.0         | 2.6   | 95.2    | 1.3       | 56.5  | 21.6    | 10.9              | 66.3  | 7.3     | 87.1       |
| 48/65                             | 34.1         | 1.3   | 97.1    | 22.1      | 34.6  | 52.2    | 29.7              | 63.8  | 10.3    | 89.7       |
| 65/100                            | 27.6         | 0.8   | 97.6    | 40.7      | 21.1  | 70.3    | 34.1              | 63.2  | 11.5    | 92.3       |
| 100/150                           | 14.6         | 0.4   | 98.5    | 24.8      | 13.5  | 79.9    | 17.5              | 60.3  | 15.8    | 94.1       |
| <150                              | 4.5          | 0.4   | 98.1    | 11.0      | 10.1  | 79.9    | 4.3               | 51.4  | 26.7    | 91.3       |



## Reagent Consumptions

Consumptive reagent data, averaged for each of the flotation processes tested, are compared in Table 17. As Options 1 and 6 were not tested on all feeds, their concentration ratios and costs per ton of concentrate are not comparable. The unit prices used for Crago process reagents are considered to be within the range of prevailing costs. Reagent unit prices were fixed for each process, except for the anionic collector. For anionic process options 1, 4, and 6 the anionic collector cost assumed a blend of Syllfat FA12 and plant collector  $[(\$0.135 + \$0.250)/2 = \$0.1925/\text{Lb}]$ . For Option 5, the anionic collector unit price assumed a 1:1 mixture of CENTURY MO-5 and \$0.12/Lb reagent  $[(\$0.35 + \$0.12)/2 = \$0.235/\text{Lb}]$ . These cost assumptions may be overly punitive to the anionic processes because Option 4 gave acceptable results with at least two plant collectors.

Anionic process option 4 offers reagent cost savings of 30% per ton of feed relative to the Crago process. The savings increase to 42% per ton of concentrate because Option 4 also improved flotation recovery and reduced concentrate grade.

**Table 17. Consumption and Cost of Reagents.**

|                           | Crago<br>Process | Anionic Processes |             |             |             |
|---------------------------|------------------|-------------------|-------------|-------------|-------------|
|                           |                  | Option 1          | Option 4    | Option 5    | Option 6    |
| <b>Reagent (Lbs/t)</b>    |                  |                   |             |             |             |
| Anionic collector         | 1.03             | 1.46              | 0.98        | 1.23        | 0.73        |
| Fuel oil                  | 0.43             | 0.87              | 0.40        | 0.37        | 0.60        |
| Soda ash                  | 0.57             | 0.35              | 0.37        | 0.57        | 0.78        |
| Sodium silicate           | 0.35             | 0.20              | 0.11        | 0.27        | 0.78        |
| Sulfuric acid             | 3.69             | -                 | -           | -           | -           |
| Cationic collector        | 0.29             | -                 | -           | -           | -           |
| <b>Reagent (\$/t)</b>     |                  |                   |             |             |             |
| Anionic collector         | 0.14             | 0.28              | 0.19        | 0.29        | 0.14        |
| Fuel oil                  | 0.03             | 0.07              | 0.03        | 0.03        | 0.05        |
| Soda ash                  | 0.05             | 0.03              | 0.03        | 0.05        | 0.07        |
| Sodium silicate           | 0.03             | 0.01              | 0.01        | 0.02        | 0.06        |
| Sulfuric acid             | 0.06             | -                 | -           | -           | -           |
| Cationic collector        | 0.07             | -                 | -           | -           | -           |
| <b>Total (\$/t feed)</b>  | <b>0.37</b>      | <b>0.39</b>       | <b>0.26</b> | <b>0.39</b> | <b>0.31</b> |
| Concentration ratio       | 4.93             | na                | 4.07        | 4.12        | na          |
| <b>Total (\$/t conc.)</b> | <b>1.81</b>      | na                | <b>1.05</b> | <b>1.59</b> | na          |

## CONCLUSIONS AND RECOMMENDATIONS

The test work performed in this program was limited to flotation feeds from four plants for laboratory testing and feeds from three plants for pilot testing. The sieve and chemical analyses of each feed varied, as did their responses to flotation. The conclusions drawn from this work are based on the feed samples tested.

Results from the pilot flotation tests confirmed that the Crago process facilitates the production of high-grade (low insol) concentrates. Concentrate with less than 5% Insol content was obtained from each feed tested. The Crago process is recommended for Florida phosphate if low-Insol concentrate production is an operational requirement.

On the other hand, if concentrates with increased Insol levels are acceptable, anionic flotation may be utilized to improve phosphate recovery and reduce reagent costs as well as the number of reagents used in flotation. Anionic process option 4 is superior to the other anionic processes tested. The relative advantages of Option 4 determined from the test program are itemized below:

1. Equivalent or superior concentrate grade
2. Equivalent or superior BPL % recovery
3. Equivalent or lower reagent consumption and cost
4. Cleaner tails sizing is less problematic than rougher concentrate sizing

Anionic process option 4 averaged at least a 30% reduction in reagent costs relative to the Crago process. Performance data for the Crago process and anionic process option 4 are compared in Table 18. Option 4 averaged 7% higher BPL recovery with a concentrate grade 6% BPL lower than the Crago process for the three feeds tested. Performance differences were exaggerated for the refractory feed from Plant 1. For the other two feeds the recovery advantage was reduced to 3% and the grade penalty was reduced to 4% BPL. The latter concentrates contained 9 to 10% Insol.

**Table 18. Process Performance Comparison.**

| Feed    | Concentrate % BPL |          |         | BPL % Recovery |          |           |
|---------|-------------------|----------|---------|----------------|----------|-----------|
|         | Crago             | Option 4 | Penalty | Crago          | Option 4 | Advantage |
| Plant 1 | 70                | 59       | (11)    | 72             | 85       | 13        |
| Plant 3 | 70                | 66       | (4)     | 86             | 90       | 5         |
| Plant 4 | 69                | 64       | (4)     | 88             | 91       | 2         |
| Average | 69                | 63       | (6)     | 82             | 89       | 7         |

As shown, the same trend in flotation performance exists for each feed; however, the magnitude of difference between the Crago process and Option 4 varies considerably from feed to feed.

The purpose of this program was to demonstrate an alternative flotation process for Florida phosphate that, relative to the Crago process, could achieve certain goals. The goals and corresponding result are listed below.

- Increase flotation recovery by 2 to 4%. Anionic process option 4 increased flotation recovery by 3 to 7%.
- Reduce the cost of flotation reagents by 33%. Anionic process option 4 reduced reagent costs as follows:
  - 30% per ton of feed
  - 42% per ton of concentrate.
- Eliminate sulfuric acid and cationic reagent usage. Goal will be accomplished if an anionic flotation process is adopted.
- Eliminate process requirements for good quality water (deep-well water). Goal will be accomplished if an anionic flotation process is adopted.

The program objective was achieved and the results met identified goals. Additional evaluation of anionic flotation by operating companies may be warranted if the production of low-Insol concentrates is not an operating requirement.

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**Appendix A**

**MATERIAL BALANCES – PLANT 1 FEED TEST RUNS**

**MATERIAL BALANCES – PLANT 1 FEED TEST RUNS**

**CIRCUIT: JACOBS Pilot Plant, Crago Flowsheet: Run 2**

| Stream        | Measured Values |       |        |        | Estimated Values |        |        |        |
|---------------|-----------------|-------|--------|--------|------------------|--------|--------|--------|
|               | Lbs/hr          | %BPL  | %Insol | %>48 M | Lbs/hr           | %BPL   | %Insol | %>48 M |
| Feed          | 941             | 12.30 | 82.86  | 28.80  | 962              | 12.34  | 82.61  | 28.44  |
| Rgh'r tail    | 773             | 3.04  | 95.14  | 27.90  | 797              | 3.04   | 95.41  | 28.18  |
| Rgh'r conc    | 168             | 54.90 | 26.37  | 32.94  | 165              | 57.40  | 20.62  | 29.70  |
| De-oil O'flow | 0               | 19.80 | 71.03  | 0.00   | 0                | 19.80  | 71.11  | 0.00   |
| Cln'r feed    | 173             | 59.71 | 18.10  | 29.09  | 164              | 57.47  | 20.52  | 29.76  |
| Cln'r conc.   | 132             | 70.70 | 3.22   | 34.57  | 120              | 69.81  | 3.23   | 36.54  |
| Cln'r tail    | 41              | 24.45 | 65.85  | 11.50  | 45               | 24.41  | 66.86  | 11.58  |
| DISTRIBUTIONS |                 |       |        |        |                  |        |        |        |
| Feed          |                 |       |        |        | 100.0%           | 100.0% | 100.0% | 100.0% |
| Rgh'r tail    |                 |       |        |        | 82.89%           | 20.41% | 95.73% | 82.13% |
| Rgh'r conc    |                 |       |        |        | 17.11%           | 79.59% | 4.27%  | 17.87% |
| De-oil O'flow |                 |       |        |        | 0.03%            | 0.05%  | 0.03%  | 0.00%  |
| Cln'r feed    |                 |       |        |        | 17.08%           | 79.54% | 4.24%  | 17.87% |
| Cln'r conc.   |                 |       |        |        | 12.43%           | 70.35% | 0.49%  | 15.98% |
| Cln'r tail    |                 |       |        |        | 4.64%            | 9.18%  | 3.76%  | 1.89%  |

**CIRCUIT: JACOBS Pilot Plant, Crago Flowsheet: Run 7**

| Stream        | Measured Values |       |        |        | Estimated Values |        |        |        |
|---------------|-----------------|-------|--------|--------|------------------|--------|--------|--------|
|               | Lbs/hr          | %BPL  | %Insol | %>48 M | Lbs/hr           | %BPL   | %Insol | %>48 M |
| Feed          | 909             | 13.74 | 80.62  | 29.30  | 883              | 12.42  | 81.55  | 29.21  |
| Rgh'r tail    | 807             | 3.70  | 93.80  | 29.50  | 753              | 4.21   | 92.72  | 29.57  |
| Rgh'r conc    | 102             | 61.38 | 14.60  | 27.72  | 129              | 60.22  | 16.54  | 27.13  |
| De-oil O'flow | 1               | 56.20 | 71.03  | 0.00   | 1                | 56.21  | 70.62  | 0.00   |
| Cln'r feed    | 135             | 58.93 | 18.27  | 26.84  | 128              | 60.25  | 16.05  | 27.37  |
| Cln'r conc.   | 110             | 68.07 | 5.72   | 30.30  | 108              | 68.03  | 5.66   | 30.33  |
| Cln'r tail    | 25              | 18.73 | 73.50  | 11.60  | 20               | 18.73  | 71.50  | 11.60  |
| DISTRIBUTIONS |                 |       |        |        |                  |        |        |        |
| Feed          |                 |       |        |        | 100.0%           | 100.0% | 100.0% | 100.0% |
| Rgh'r tail    |                 |       |        |        | 85.34%           | 28.93% | 97.03% | 86.39% |
| Rgh'r conc    |                 |       |        |        | 14.66%           | 71.07% | 2.97%  | 13.61% |
| De-oil O'flow |                 |       |        |        | 0.13%            | 0.60%  | 0.11%  | 0.00%  |
| Cln'r feed    |                 |       |        |        | 14.53%           | 70.47% | 2.86%  | 13.61% |
| Cln'r conc.   |                 |       |        |        | 12.24%           | 67.02% | 0.85%  | 12.70% |
| Cln'r tail    |                 |       |        |        | 2.29%            | 3.46%  | 2.01%  | 0.91%  |

**CIRCUIT: JACOBS Pilot Plant, Crago Flowsheet: Run 8**

| Stream               | Measured Values |       |        |         | Estimated Values |        |        |         |
|----------------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|                      | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed                 | 942             | 10.30 | 85.67  | 22.40   | 921              | 9.75   | 85.89  | 22.23   |
| Rgh'r tail           | 804             | 1.77  | 96.72  | 23.10   | 773              | 1.86   | 96.48  | 23.25   |
| Rgh'r conc           | 139             | 50.07 | 31.73  | 20.00   | 148              | 51.02  | 30.46  | 16.86   |
| De-oil O'flow        | 0               | 50.07 | 31.73  | 0.10    | 0                | 50.07  | 31.73  | 0.10    |
| Cln'r feed           | 149             | 51.67 | 29.60  | 16.11   | 148              | 51.02  | 30.46  | 16.86   |
| Cln'r conc.          | 95              | 70.72 | 3.97   | 19.70   | 93               | 70.45  | 3.97   | 20.93   |
| Cln'r tail           | 54              | 18.20 | 74.65  | 9.80    | 55               | 18.19  | 75.22  | 9.98    |
| <b>DISTRIBUTIONS</b> |                 |       |        |         |                  |        |        |         |
| Feed                 |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rgh'r tail           |                 |       |        |         | 83.95%           | 15.99% | 94.31% | 87.83%  |
| Rgh'r conc           |                 |       |        |         | 16.05%           | 84.01% | 5.69%  | 12.17%  |
| De-oil O'flow        |                 |       |        |         | 0.00%            | 0.01%  | 0.00%  | 0.00%   |
| Cln'r feed           |                 |       |        |         | 16.04%           | 84.01% | 5.69%  | 12.17%  |
| Cln'r conc.          |                 |       |        |         | 10.08%           | 72.87% | 0.47%  | 9.49%   |
| Cln'r tail           |                 |       |        |         | 5.96%            | 11.13% | 5.22%  | 2.68%   |

**CIRCUIT: JACOBS Pilot Plant, Crago Flowsheet: Run 9**

| Stream               | Measured Values |       |        |         | Estimated Values |        |        |         |
|----------------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|                      | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed                 | 909             | 9.70  | 85.90  | 25.10   | 904              | 9.57   | 86.07  | 24.57   |
| Rgh'r tail           | 753             | 1.55  | 96.82  | 24.80   | 746              | 1.57   | 96.65  | 25.23   |
| Rgh'r conc           | 156             | 46.66 | 36.62  | 26.54   | 158              | 47.37  | 36.11  | 21.42   |
| De-oil O'flow        | 0               | 46.66 | 36.62  | 0.10    | 0                | 46.66  | 36.62  | 0.10    |
| Cln'r feed           | 158             | 47.80 | 35.75  | 20.29   | 158              | 47.37  | 36.11  | 21.44   |
| Cln'r conc.          | 96              | 70.72 | 5.80   | 25.50   | 95               | 70.35  | 5.80   | 27.31   |
| Cln'r tail           | 62              | 12.60 | 81.73  | 12.28   | 63               | 12.59  | 81.97  | 12.56   |
| <b>DISTRIBUTIONS</b> |                 |       |        |         |                  |        |        |         |
| Feed                 |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rgh'r tail           |                 |       |        |         | 82.53%           | 13.51% | 92.67% | 84.76%  |
| Rgh'r conc           |                 |       |        |         | 17.47%           | 86.49% | 7.33%  | 15.24%  |
| De-oil O'flow        |                 |       |        |         | 0.01%            | 0.05%  | 0.00%  | 0.00%   |
| Cln'r feed           |                 |       |        |         | 17.46%           | 86.43% | 7.33%  | 15.24%  |
| Cln'r conc.          |                 |       |        |         | 10.52%           | 77.29% | 0.71%  | 11.69%  |
| Cln'r tail           |                 |       |        |         | 6.95%            | 9.14%  | 6.62%  | 3.55%   |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 3**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| New Feed      | 995             | 13.81 | 80.77  | 41.10   | 931              | 13.75  | 80.63  | 36.77   |
| Rghr Feed     | 1,015           | 13.97 | 80.49  | 40.94   | 958              | 13.99  | 80.25  | 36.65   |
| Rghr Tail     | 686             | 1.75  | 96.67  | 31.30   | 717              | 1.76   | 97.09  | 35.11   |
| Rghr Conc     | 329             | 50.20 | 31.45  | 45.00   | 241              | 50.35  | 30.20  | 41.25   |
| Clnr Conc     | 182             | 57.94 | 19.65  | 41.60   | 196              | 57.81  | 20.05  | 46.04   |
| Clnr Tail     | 147             | 18.47 | 73.77  | 20.00   | 45               | 18.13  | 74.01  | 20.54   |
| Sizer O'size  | 20              | 22.04 | 66.66  | 33.00   | 27               | 22.32  | 67.17  | 32.42   |
| Sizer U'size  | 20              | 11.82 | 83.37  | 3.30    | 19               | 12.04  | 83.93  | 3.30    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Feed     |                 |       |        |         | 102.9%           | 104.7% | 102.4% | 102.5%  |
| Rghr Tail     |                 |       |        |         | 77.0%            | 9.9%   | 92.7%  | 73.5%   |
| Rghr Conc     |                 |       |        |         | 25.9%            | 94.8%  | 9.7%   | 29.1%   |
| Clnr Conc     |                 |       |        |         | 21.0%            | 88.4%  | 5.2%   | 26.3%   |
| Clnr Tail     |                 |       |        |         | 4.9%             | 6.4%   | 4.5%   | 2.7%    |
| Sizer O'size  |                 |       |        |         | 2.9%             | 4.7%   | 2.4%   | 2.5%    |
| Sizer U'size  |                 |       |        |         | 2.0%             | 1.7%   | 2.1%   | 0.2%    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 4**

| Stream        | Measured Values |       |        |         | Estimated Values |       |        |         |
|---------------|-----------------|-------|--------|---------|------------------|-------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL  | %Insol | % >48 M |
| New Feed      | 995             | 13.91 | 80.16  | 32.80   | 1,050            | 14.00 | 79.98  | 33.37   |
| Rghr Feed     | 1,015           | 14.39 | 79.48  | 32.80   | 1,071            | 14.48 | 79.31  | 33.29   |
| Rghr Tail     | 772             | 3.20  | 94.71  | 35.20   | 849              | 3.02  | 95.10  | 34.23   |
| Rghr Conc     | 243             | 58.63 | 18.52  | 25.19   | 222              | 58.41 | 18.76  | 29.67   |
| Clnr Conc     | 209             | 63.00 | 12.50  | 41.80   | 188              | 63.06 | 12.41  | 31.04   |
| Clnr Tail     | 34              | 32.45 | 54.12  | 20.00   | 33               | 32.27 | 54.45  | 21.94   |
| Sizer O'size  | 20              | 38.50 | 45.53  | 33.00   | 21               | 38.66 | 45.25  | 29.02   |
| Sizer U'size  | 14              | 21.84 | 69.68  | 10.80   | 13               | 21.97 | 69.27  | 10.53   |
| DISTRIBUTIONS |                 |       |        |         |                  |       |        |         |
| New Feed      |                 |       |        |         | 100%             | 100%  | 100%   | 100%    |
| Rghr Feed     |                 |       |        |         | 102%             | 105%  | 101%   | 102%    |
| Rghr Tail     |                 |       |        |         | 81%              | 17%   | 96%    | 83%     |
| Rghr Conc     |                 |       |        |         | 21%              | 88%   | 5%     | 19%     |
| Clnr Conc     |                 |       |        |         | 18%              | 81%   | 3%     | 17%     |
| Clnr Tail     |                 |       |        |         | 3%               | 7%    | 2%     | 2%      |
| Sizer O'size  |                 |       |        |         | 2%               | 5%    | 1%     | 2%      |
| Sizer U'size  |                 |       |        |         | 1%               | 2%    | 1%     | 0%      |



**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 10**

| Stream               | Measured Values |       |        |         | Estimated Values |        |        |         |
|----------------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|                      | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| New Feed             | 983             | 9.46  | 86.80  | 20.60   | 1,022            | 9.49   | 86.76  | 21.08   |
| Rghr Feed            | 988             | 9.53  | 86.70  | 20.70   | 1,026            | 9.56   | 86.66  | 21.20   |
| Rghr Tail            | 808             | 1.37  | 97.56  | 22.70   | 847              | 1.34   | 97.64  | 21.71   |
| Rghr Conc            | 179             | 48.78 | 34.02  | 20.00   | 179              | 48.37  | 34.86  | 18.79   |
| Clnr Conc            | 149             | 54.27 | 27.10  | 19.60   | 152              | 54.62  | 26.65  | 20.46   |
| Clnr Tail            | 31              | 13.85 | 80.70  | 15.00   | 28               | 13.96  | 80.08  | 9.60    |
| Sizer O'size         | 4               | 26.72 | 63.05  | 43.00   | 4                | 26.66  | 63.04  | 50.15   |
| Sizer U'size         | 26              | 11.82 | 83.37  | 1.90    | 23               | 11.57  | 83.28  | 1.97    |
| <b>DISTRIBUTIONS</b> |                 |       |        |         |                  |        |        |         |
| New Feed             |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Feed            |                 |       |        |         | 100.4%           | 101.2% | 100.3% | 101.0%  |
| Rghr Tail            |                 |       |        |         | 82.9%            | 11.7%  | 93.3%  | 85.4%   |
| Rghr Conc            |                 |       |        |         | 17.6%            | 89.5%  | 7.1%   | 15.6%   |
| Clnr Conc            |                 |       |        |         | 14.9%            | 85.5%  | 4.6%   | 14.4%   |
| Clnr Tail            |                 |       |        |         | 2.7%             | 4.0%   | 2.5%   | 1.2%    |
| Sizer O'size         |                 |       |        |         | 0.4%             | 1.2%   | 0.3%   | 1.0%    |
| Sizer U'size         |                 |       |        |         | 2.3%             | 2.8%   | 2.2%   | 0.2%    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 5: Run 5**

| Stream        | Measured Values |       |        |         | Estimated Values |       |        |         |
|---------------|-----------------|-------|--------|---------|------------------|-------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL  | %Insol | % >48 M |
| Feed          | 1,024           | 13.67 | 80.92  | 31.70   | 932              | 13.26 | 80.83  | 31.62   |
| Rghr Tail     | 824             | 2.52  | 96.23  | 31.10   | 735              | 2.79  | 96.33  | 31.09   |
| Rghr Conc     | 200             | 36.33 | 22.42  | 34.17   | 197              | 52.28 | 23.06  | 33.59   |
| Sizer U'size  | 61              | 45.88 | 36.33  | 1.00    | 60               | 46.23 | 35.41  | 0.75    |
| Clnr Conc     | 51              | 55.80 | 24.35  | 0.60    | 49               | 54.03 | 24.55  | 0.67    |
| Clnr Tail     | 10              | 14.06 | 79.82  | 1.00    | 12               | 14.02 | 80.22  | 1.05    |
| Sizer O'size  | 139             | 59.26 | 17.80  | 47.90   | 137              | 54.95 | 17.60  | 48.08   |
| Comb. Conc    | 190             | 58.33 | 19.56  | 35.20   | 185              | 54.70 | 19.43  | 35.65   |
| DISTRIBUTIONS |                 |       |        |         |                  |       |        |         |
| Feed          |                 |       |        |         | 100%             | 100%  | 100%   | 100%    |
| Rghr Tail     |                 |       |        |         | 79%              | 17%   | 94%    | 78%     |
| Rghr Conc     |                 |       |        |         | 21%              | 83%   | 6%     | 22%     |
| Sizer U'size  |                 |       |        |         | 6%               | 23%   | 3%     | 0%      |
| Clnr Conc     |                 |       |        |         | 5%               | 21%   | 2%     | 0%      |
| Clnr Tail     |                 |       |        |         | 1%               | 1%    | 1%     | 0%      |
| Sizer O'size  |                 |       |        |         | 15%              | 61%   | 3%     | 22%     |
| Comb. Conc    |                 |       |        |         | 20%              | 82%   | 5%     | 22%     |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 5: Run 6**

| Stream        | Measured Values |       |        |         | Estimated Values |       |        |         |
|---------------|-----------------|-------|--------|---------|------------------|-------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL  | %Insol | % >48 M |
| Feed          | 951             | 13.87 | 80.00  | 27.70   | 885              | 13.56 | 80.15  | 25.97   |
| Rghr Tail     | 790             | 2.82  | 94.75  | 25.10   | 727              | 3.08  | 94.59  | 26.27   |
| Rghr Conc     | 161             | 61.74 | 13.70  | 40.46   | 158              | 61.71 | 13.83  | 24.62   |
| Sizer U'size  | 78              | 59.76 | 16.80  | 0.50    | 79               | 60.47 | 15.86  | 0.66    |
| Clnr Conc     | 68              | 64.02 | 11.20  | 0.50    | 69               | 63.50 | 11.49  | 0.36    |
| Clnr Tail     | 10              | 39.73 | 45.06  | 4.00    | 10               | 39.70 | 45.83  | 2.71    |
| Sizer O'size  | 76              | 62.73 | 11.92  | 43.80   | 80               | 62.95 | 11.82  | 48.33   |
| Comb. Conc    | 144             | 63.34 | 11.58  | 23.35   | 148              | 63.20 | 11.67  | 26.10   |
| DISTRIBUTIONS |                 |       |        |         |                  |       |        |         |
| Feed          |                 |       |        |         | 100%             | 100%  | 100%   | 100%    |
| Rghr Tail     |                 |       |        |         | 82%              | 19%   | 97%    | 83%     |
| Rghr Conc     |                 |       |        |         | 18%              | 81%   | 3%     | 17%     |
| Sizer U'size  |                 |       |        |         | 9%               | 40%   | 2%     | 0%      |
| Clnr Conc     |                 |       |        |         | 8%               | 36%   | 1%     | 0%      |
| Clnr Tail     |                 |       |        |         | 1%               | 3%    | 1%     | 0%      |
| Sizer O'size  |                 |       |        |         | 9%               | 42%   | 1%     | 17%     |
| Comb. Conc    |                 |       |        |         | 17%              | 78%   | 2%     | 17%     |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 5: Run 11**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| Feed          | 931             | 9.66  | 86.40  | 26.20   | 931              | 9.57   | 86.57  | 26.21   |
| Rghr Tail     | 763             | 1.70  | 97.17  | 27.30   | 763              | 1.76   | 96.95  | 27.30   |
| Rghr Conc     | 168             | 43.80 | 40.25  | 21.20   | 169              | 44.88  | 39.64  | 21.27   |
| Sizer U'size  | 92              | 38.00 | 48.62  | 1.00    | 92               | 37.95  | 49.25  | 0.55    |
| Clnr Conc     | 64              | 48.88 | 34.92  | 0.40    | 64               | 48.87  | 34.87  | 0.45    |
| Clnr Tail     | 28              | 13.05 | 82.10  | 0.70    | 28               | 13.05  | 82.07  | 0.77    |
| Sizer O'size  | 76              | 53.37 | 27.87  | 46.50   | 76               | 53.28  | 27.99  | 46.40   |
| Comb. Conc    | 140             | 51.33 | 31.08  | 25.50   | 140              | 51.27  | 31.14  | 25.38   |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| Feed          |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Tail     |                 |       |        |         | 81.9%            | 15.1%  | 91.7%  | 85.3%   |
| Rghr Conc     |                 |       |        |         | 18.1%            | 84.9%  | 8.3%   | 14.7%   |
| Sizer U'size  |                 |       |        |         | 9.9%             | 39.4%  | 5.6%   | 0.2%    |
| Clnr Conc     |                 |       |        |         | 6.9%             | 35.2%  | 2.8%   | 0.1%    |
| Clnr Tail     |                 |       |        |         | 3.0%             | 4.1%   | 2.9%   | 0.1%    |
| Sizer O'size  |                 |       |        |         | 8.2%             | 45.6%  | 2.6%   | 14.5%   |
| Comb. Conc    |                 |       |        |         | 15.1%            | 80.8%  | 5.4%   | 14.6%   |

## **Appendix B**

### **MATERIAL BALANCES – PLANT 3 FEED TEST RUNS**

**MATERIAL BALANCES – PLANT 3 FEED TEST RUNS**

**CIRCUIT: JACOBS Pilot Plant, Crago Flowsheet: Run 101A**

| Stream               | Measured Values |       |        |         | Estimated Values |        |        |         |
|----------------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|                      | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed                 | 756             | 19.16 | 72.35  | 49.40   | 772              | 19.55  | 72.14  | 48.77   |
| Rgh'r tail           | 541             | 1.96  | 95.99  | 41.90   | 545              | 1.94   | 96.25  | 42.22   |
| Rgh'r conc           | 215             | 62.07 | 14.06  | 68.29   | 227              | 61.88  | 14.18  | 64.53   |
| De-oil O'flow        | 0               | 48.80 | 27.37  | 0.00    | 0                | 48.80  | 27.37  | 0.00    |
| Cln'r feed           | 228             | 62.07 | 14.06  | 62.38   | 227              | 61.89  | 14.18  | 64.53   |
| Cln'r conc.          | 186             | 69.24 | 3.82   | 71.90   | 190              | 69.55  | 3.81   | 73.31   |
| Cln'r tail           | 42              | 22.94 | 67.81  | 19.90   | 37               | 22.95  | 66.91  | 19.92   |
| <b>DISTRIBUTIONS</b> |                 |       |        |         |                  |        |        |         |
| Feed                 |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rgh'r tail           |                 |       |        |         | 70.62            | 7.01   | 94.23  | 61.13   |
| Rgh'r conc           |                 |       |        |         | 29.38            | 92.99  | 5.77   | 38.87   |
| De-oil O'flow        |                 |       |        |         | 0.00             | 0.00   | 0.00   | 0.00    |
| Cln'r feed           |                 |       |        |         | 29.38            | 92.98  | 5.77   | 38.87   |
| Cln'r conc.          |                 |       |        |         | 24.55            | 87.32  | 1.29   | 36.90   |
| Cln'r tail           |                 |       |        |         | 4.83             | 5.67   | 4.48   | 1.97    |

**CIRCUIT: JACOBS Pilot Plant, Crago Flowsheet: Run 101B**

| Stream               | Measured Values |       |        |         | Estimated Values |        |        |         |
|----------------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|                      | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed                 | 749             | 19.36 | 72.64  | 46.60   | 738              | 19.28  | 72.44  | 46.76   |
| Rgh'r tail           | 541             | 1.17  | 96.94  | 40.00   | 511              | 1.19   | 97.19  | 39.92   |
| Rgh'r conc           | 207             | 59.68 | 16.92  | 63.83   | 227              | 59.90  | 16.86  | 62.12   |
| De-oil O'flow        | 0               | 0.00  | 0.00   | 0.00    | 0                | 0.00   | 0.00   | 0.00    |
| Cln'r feed           | 233             | 59.68 | 16.92  | 61.79   | 227              | 59.91  | 16.86  | 62.13   |
| Cln'r conc.          | 180             | 69.84 | 3.35   | 73.90   | 172              | 69.62  | 3.35   | 75.18   |
| Cln'r tail           | 54              | 29.61 | 58.69  | 21.30   | 55               | 29.60  | 59.05  | 21.33   |
| <b>DISTRIBUTIONS</b> |                 |       |        |         |                  |        |        |         |
| Feed                 |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rgh'r tail           |                 |       |        |         | 69.19            | 4.27   | 92.83  | 59.07   |
| Rgh'r conc           |                 |       |        |         | 30.81            | 95.73  | 7.17   | 40.93   |
| De-oil O'flow        |                 |       |        |         | 0.00             | 0.00   | 0.00   | 0.00    |
| Cln'r feed           |                 |       |        |         | 30.80            | 95.73  | 7.17   | 40.93   |
| Cln'r conc.          |                 |       |        |         | 23.33            | 84.26  | 1.08   | 37.52   |
| Cln'r tail           |                 |       |        |         | 7.47             | 11.47  | 6.09   | 3.41    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 1: Feed Sizing**

| Stream        | Measured Values |       |        |         | Estimated Values |         |         |         |
|---------------|-----------------|-------|--------|---------|------------------|---------|---------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL    | %Insol  | % >48 M |
| New Feed      | 808             | 20.16 | 71.46  | 47.80   | 851              | 19.66   | 72.10   | 46.58   |
| Crse feed     | 475             | 25.82 | 63.70  | 72.36   | 520              | 26.32   | 63.39   | 74.07   |
| fine feed     | 333             | 9.15  | 86.15  | 3.40    | 331              | 9.19    | 85.79   | 3.40    |
| DISTRIBUTIONS |                 |       |        |         |                  |         |         |         |
| New Feed      |                 |       |        |         | 100.00%          | 100.00% | 100.00% | 100.00% |
| Crse feed     |                 |       |        |         | 61.10%           | 81.81%  | 53.72%  | 97.16%  |
| fine feed     |                 |       |        |         | 38.90%           | 18.19%  | 46.28%  | 2.84%   |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 1: Run 104A**

| Stream        | Measured Values |       |        |         | Estimated Values |         |         |         |
|---------------|-----------------|-------|--------|---------|------------------|---------|---------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL    | %Insol  | % >48 M |
| New Feed      | 322             | 9.15  | 86.15  | 3.40    | 333              | 9.45    | 85.91   | 3.91    |
| Rghr Tail     | 236             | 0.54  | 98.00  | 3.70    | 270              | 0.53    | 98.44   | 3.21    |
| Rghr Conc     | 76              | 49.26 | 30.80  | 7.00    | 63               | 47.33   | 32.67   | 6.90    |
| Clnr Tail     | 13              | 6.17  | 89.30  | 3.80    | 12               | 6.17    | 87.58   | 3.78    |
| Clnr Conc     | 54              | 57.75 | 19.62  | 8.00    | 51               | 57.37   | 19.28   | 7.67    |
| DISTRIBUTIONS |                 |       |        |         |                  |         |         |         |
| New Feed      |                 |       |        |         | 100.00%          | 100.00% | 100.00% | 100.00% |
| Rghr Tail     |                 |       |        |         | 80.94%           | 4.58%   | 92.75%  | 66.37%  |
| Rghr Conc     |                 |       |        |         | 19.06%           | 95.42%  | 7.25%   | 33.63%  |
| Clnr Tail     |                 |       |        |         | 3.74%            | 2.44%   | 3.81%   | 3.61%   |
| Clnr Conc     |                 |       |        |         | 15.32%           | 92.98%  | 3.44%   | 30.02%  |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 1: Run 104B (coarse feed)**

| Stream        | Measured Values |       |        |         | Estimated Values |         |         |         |
|---------------|-----------------|-------|--------|---------|------------------|---------|---------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL    | %Insol  | % >48 M |
| New Feed      | 774             | 26.66 | 61.95  | 78.00   | 732              | 26.65   | 62.01   | 77.55   |
| Rghr Tail     | 390             | 5.37  | 91.03  | 82.30   | 399              | 5.37    | 90.96   | 82.57   |
| Rghr Conc     | 361             | 50.70 | 30.12  | 74.50   | 333              | 52.10   | 27.38   | 71.53   |
| Clnr Tail     | 47              | 1.09  | 97.80  | 44.30   | 53               | 1.09    | 99.82   | 44.48   |
| Clnr Conc     | 291             | 62.12 | 13.55  | 74.00   | 281              | 61.70   | 13.76   | 76.62   |
| DISTRIBUTIONS |                 |       |        |         |                  |         |         |         |
| New Feed      |                 |       |        |         | 100.00%          | 100.00% | 100.00% | 100.00% |
| Rghr Tail     |                 |       |        |         | 54.47%           | 10.98%  | 79.89%  | 58.00%  |
| Rghr Conc     |                 |       |        |         | 45.53%           | 89.02%  | 20.11%  | 42.00%  |
| Clnr Tail     |                 |       |        |         | 7.21%            | 0.29%   | 11.61%  | 4.14%   |
| Clnr Conc     |                 |       |        |         | 38.32%           | 88.73%  | 8.50%   | 37.87%  |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 1: Run 104C (coarse feed)**

| Stream        | Measured Values |       |        |         | Estimated Values |         |         |         |
|---------------|-----------------|-------|--------|---------|------------------|---------|---------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL    | %Insol  | % >48 M |
| New Feed      | 850             | 26.20 | 63.57  | 69.30   | 816              | 26.42   | 63.11   | 71.04   |
| Rghr Tail     | 434             | 4.65  | 92.60  | 73.90   | 485              | 4.65    | 93.18   | 72.72   |
| Rghr Conc     | 373             | 59.06 | 18.88  | 69.70   | 331              | 58.34   | 19.05   | 68.57   |
| Clnr Tail     | 42              | 2.92  | 93.50  | 29.50   | 42               | 2.92    | 93.29   | 29.51   |
| Clnr Conc     | 288             | 66.64 | 8.34   | 73.80   | 289              | 66.33   | 8.33    | 74.21   |
| DISTRIBUTIONS |                 |       |        |         |                  |         |         |         |
| New Feed      |                 |       |        |         | 100.00%          | 100.00% | 100.00% | 100.00% |
| Rghr Tail     |                 |       |        |         | 59.44%           | 10.45%  | 87.76%  | 60.85%  |
| Rghr Conc     |                 |       |        |         | 40.56%           | 89.55%  | 12.24%  | 39.15%  |
| Clnr Tail     |                 |       |        |         | 5.12%            | 0.57%   | 7.56%   | 2.12%   |
| Clnr Conc     |                 |       |        |         | 35.44%           | 88.98%  | 4.68%   | 37.02%  |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 1: Run 104D (coarse feed)**

| Stream        | Measured Values |       |        |         | Estimated Values |         |         |         |
|---------------|-----------------|-------|--------|---------|------------------|---------|---------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL    | %Insol  | % >48 M |
| New Feed      | 761             | 24.23 | 66.63  | 65.40   | 791              | 24.38   | 65.98   | 68.49   |
| Rghr Tail     | 462             | 2.95  | 95.00  | 70.40   | 490              | 2.95    | 95.82   | 68.18   |
| Rghr Conc     | 305             | 60.15 | 16.58  | 72.70   | 301              | 59.30   | 17.35   | 69.01   |
| Clnr Tail     | 38              | 8.28  | 86.57  | 34.40   | 35               | 8.28    | 85.50   | 34.46   |
| Clnr Conc     | 272             | 66.25 | 8.33   | 71.70   | 265              | 66.11   | 8.26    | 73.62   |
| DISTRIBUTIONS |                 |       |        |         |                  |         |         |         |
| New Feed      |                 |       |        |         | 100.00%          | 100.00% | 100.00% | 100.00% |
| Rghr Tail     |                 |       |        |         | 61.98%           | 7.50%   | 90.00%  | 61.69%  |
| Rghr Conc     |                 |       |        |         | 38.02%           | 92.50%  | 10.00%  | 38.31%  |
| Clnr Tail     |                 |       |        |         | 4.48%            | 1.52%   | 5.80%   | 2.25%   |
| Clnr Conc     |                 |       |        |         | 33.55%           | 90.98%  | 4.20%   | 36.06%  |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 102A**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| New Feed      | 756             | 19.62 | 72.53  | 48.70   | 784              | 19.67  | 72.46  | 47.80   |
| Rghr Feed     | 765             | 19.64 | 72.51  | 49.29   | 789              | 19.68  | 72.44  | 48.07   |
| Rghr Tail     | 543             | 2.43  | 95.95  | 41.00   | 556              | 2.40   | 96.11  | 42.04   |
| Rghr Conc     | 222             | 61.02 | 15.70  | 62.00   | 233              | 60.96  | 15.89  | 62.48   |
| Clnr Conc     | 247             | 64.59 | 11.02  | 64.10   | 218              | 64.58  | 10.93  | 64.62   |
| Clnr Tail     | 17              | 9.61  | 86.02  | 29.00   | 15               | 9.65   | 85.98  | 32.24   |
| Sizer O'size  | 9               | 20.91 | 70.43  | 99.00   | 6                | 20.84  | 70.35  | 84.93   |
| Sizer U'size  | 8               | 3.02  | 95.50  | 1.00    | 10               | 3.01   | 95.25  | 1.00    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rghr Feed     |                 |       |        |         | 100.73           | 100.77 | 100.71 | 101.30  |
| Rghr Tail     |                 |       |        |         | 71.01            | 8.68   | 94.19  | 62.45   |
| Rghr Conc     |                 |       |        |         | 29.72            | 92.09  | 6.52   | 38.84   |
| Clnr Conc     |                 |       |        |         | 27.76            | 91.13  | 4.19   | 37.52   |
| Clnr Tail     |                 |       |        |         | 1.96             | 0.96   | 2.33   | 1.32    |
| Sizer O'size  |                 |       |        |         | 0.73             | 0.77   | 0.71   | 1.30    |
| Sizer U'size  |                 |       |        |         | 1.23             | 0.19   | 1.62   | 0.03    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 102B**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| New Feed      | 770             | 19.41 | 73.30  | 45.40   | 736              | 19.31  | 73.12  | 45.20   |
| Rghr Feed     | 779             | 19.60 | 73.02  | 46.02   | 744              | 19.48  | 72.88  | 45.35   |
| Rghr Tail     | 606             | 2.48  | 95.92  | 38.90   | 535              | 2.54   | 96.32  | 39.35   |
| Rghr Conc     | 173             | 62.80 | 12.66  | 62.00   | 209              | 62.94  | 12.76  | 60.72   |
| Clnr Conc     | 201             | 65.67 | 9.01   | 60.90   | 195              | 65.69  | 8.96   | 62.47   |
| Clnr Tail     | 14              | 22.39 | 69.11  | 29.00   | 13               | 22.42  | 68.51  | 34.99   |
| Sizer O'size  | 9               | 36.12 | 49.11  | 99.00   | 7                | 36.08  | 49.23  | 59.84   |
| Sizer U'size  | 5               | 4.81  | 93.02  | 3.00    | 6                | 4.81   | 93.35  | 2.97    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rghr Feed     |                 |       |        |         | 101.02           | 101.90 | 100.68 | 101.35  |
| Rghr Tail     |                 |       |        |         | 72.68            | 9.57   | 95.74  | 63.28   |
| Rghr Conc     |                 |       |        |         | 28.33            | 92.33  | 4.94   | 38.06   |
| Clnr Conc     |                 |       |        |         | 26.53            | 90.23  | 3.25   | 36.66   |
| Clnr Tail     |                 |       |        |         | 1.80             | 2.09   | 1.69   | 1.40    |
| Sizer O'size  |                 |       |        |         | 1.02             | 1.90   | 0.68   | 1.35    |
| Sizer U'size  |                 |       |        |         | 0.79             | 0.20   | 1.01   | 0.05    |



**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 102C**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| New Feed      | 768             | 20.11 | 71.86  | 47.10   | 755              | 20.12  | 71.75  | 47.76   |
| Rghr Feed     | 777             | 20.31 | 71.58  | 47.25   | 759              | 20.22  | 71.62  | 47.86   |
| Rghr Tail     | 552             | 2.52  | 95.91  | 43.10   | 536              | 2.54   | 96.01  | 42.35   |
| Rghr Conc     | 225             | 62.50 | 13.35  | 62.00   | 223              | 62.68  | 13.02  | 61.11   |
| Clnr Conc     | 213             | 65.17 | 9.60   | 62.50   | 212              | 65.04  | 9.76   | 62.74   |
| Clnr Tail     | 14              | 17.44 | 76.01  | 35.00   | 11               | 17.59  | 75.51  | 29.93   |
| Sizer O'size  | 9               | 37.15 | 48.07  | 60.00   | 4                | 36.88  | 48.23  | 65.93   |
| Sizer U'size  | 5               | 4.91  | 92.58  | 6.10    | 7                | 4.88   | 93.50  | 6.19    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rghr Feed     |                 |       |        |         | 100.58           | 101.07 | 100.39 | 100.80  |
| Rghr Tail     |                 |       |        |         | 71.02            | 8.97   | 95.03  | 62.98   |
| Rghr Conc     |                 |       |        |         | 29.56            | 92.10  | 5.36   | 37.83   |
| Clnr Conc     |                 |       |        |         | 28.10            | 90.82  | 3.82   | 36.91   |
| Clnr Tail     |                 |       |        |         | 1.46             | 1.28   | 1.54   | 0.92    |
| Sizer O'size  |                 |       |        |         | 0.58             | 1.07   | 0.39   | 0.80    |
| Sizer U'size  |                 |       |        |         | 0.88             | 0.21   | 1.15   | 0.11    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 102D**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| New Feed      | 776             | 23.05 | 67.34  | 50.30   | 726              | 22.88  | 67.51  | 50.36   |
| Rghr Feed     | 787             | 23.37 | 66.88  | 50.92   | 737              | 23.22  | 67.03  | 50.89   |
| Rghr Tail     | 592             | 2.80  | 95.13  | 43.00   | 492              | 2.88   | 94.69  | 42.99   |
| Rghr Conc     | 196             | 64.15 | 11.37  | 68.70   | 244              | 64.21  | 11.29  | 66.81   |
| Clnr Conc     | 242             | 66.05 | 8.58   | 65.50   | 229              | 66.20  | 8.62   | 67.09   |
| Clnr Tail     | 16              | 34.92 | 51.42  | 58.10   | 16               | 34.89  | 50.64  | 62.71   |
| Sizer O'size  | 11              | 46.10 | 34.28  | 94.70   | 11               | 46.14  | 34.55  | 86.35   |
| Sizer U'size  | 5               | 9.28  | 86.53  | 8.90    | 5                | 9.28   | 87.29  | 8.87    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rghr Feed     |                 |       |        |         | 101.48           | 102.99 | 100.76 | 102.54  |
| Rghr Tail     |                 |       |        |         | 67.83            | 8.54   | 95.13  | 57.90   |
| Rghr Conc     |                 |       |        |         | 33.65            | 94.45  | 5.63   | 44.65   |
| Clnr Conc     |                 |       |        |         | 31.52            | 91.20  | 4.02   | 41.99   |
| Clnr Tail     |                 |       |        |         | 2.14             | 3.26   | 1.60   | 2.66    |
| Sizer O'size  |                 |       |        |         | 1.48             | 2.99   | 0.76   | 2.54    |
| Sizer U'size  |                 |       |        |         | 0.65             | 0.26   | 0.84   | 0.11    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 102E**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| New Feed      | 802             | 20.86 | 70.22  | 50.30   | 821              | 20.91  | 70.20  | 48.59   |
| Rghr Feed     | 824             | 21.71 | 69.04  | 51.55   | 842              | 21.73  | 69.06  | 49.72   |
| Rghr Tail     | 590             | 3.37  | 94.33  | 40.80   | 591              | 3.34   | 94.33  | 42.42   |
| Rghr Conc     | 234             | 64.45 | 9.33   | 68.40   | 251              | 64.98  | 9.59   | 66.89   |
| Clnr Conc     | 239             | 67.84 | 6.60   | 63.00   | 225              | 67.27  | 6.48   | 65.62   |
| Clnr Tail     | 27              | 45.40 | 36.60  | 74.10   | 26               | 45.31  | 36.30  | 77.78   |
| Sizer O'size  | 22              | 52.78 | 25.84  | 97.30   | 22               | 52.85  | 25.79  | 92.48   |
| Sizer U'size  | 5               | 9.68  | 86.05  | 8.30    | 5                | 9.68   | 85.94  | 8.29    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rghr Feed     |                 |       |        |         | 102.63           | 106.65 | 100.97 | 105.01  |
| Rghr Tail     |                 |       |        |         | 72.02            | 11.51  | 96.79  | 62.87   |
| Rghr Conc     |                 |       |        |         | 30.61            | 95.14  | 4.18   | 42.14   |
| Clnr Conc     |                 |       |        |         | 27.42            | 88.23  | 2.53   | 37.03   |
| Clnr Tail     |                 |       |        |         | 3.19             | 6.91   | 1.65   | 5.11    |
| Sizer O'size  |                 |       |        |         | 2.63             | 6.65   | 0.97   | 5.01    |
| Sizer U'size  |                 |       |        |         | 0.56             | 0.26   | 0.68   | 0.10    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 5: Run 103A**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed          | 737             | 19.63 | 71.25  | 48.10   | 730              | 19.72  | 71.10  | 47.81   |
| Rghr Tail     | 525             | 4.57  | 92.32  | 44.60   | 535              | 4.48   | 92.50  | 44.78   |
| Rghr Conc     | 212             | 61.00 | 13.00  | 64.70   | 196              | 61.39  | 12.59  | 56.06   |
| Sizer U'size  | 100             | 58.57 | 16.80  | 38.50   | 95               | 59.10  | 15.71  | 36.67   |
| Clnr Conc     | 94              | 63.53 | 9.70   | 36.70   | 89               | 62.99  | 10.06  | 38.88   |
| Clnr Tail     | 6               | 6.97  | 88.70  | 7.00    | 7                | 6.97   | 91.46  | 7.01    |
| Sizer O'size  | 100             | 63.53 | 9.62   | 71.70   | 100              | 63.57  | 9.64   | 74.43   |
| Comb. Conc    | 194             | 63.53 | 9.66   | 54.70   | 189              | 63.29  | 9.84   | 57.78   |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| Feed          |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rghr Tail     |                 |       |        |         | 73.21            | 16.62  | 95.26  | 68.59   |
| Rghr Conc     |                 |       |        |         | 26.79            | 83.38  | 4.74   | 31.41   |
| Sizer U'size  |                 |       |        |         | 13.03            | 39.04  | 2.88   | 9.99    |
| Clnr Conc     |                 |       |        |         | 12.12            | 38.72  | 1.72   | 9.86    |
| Clnr Tail     |                 |       |        |         | 0.90             | 0.32   | 1.16   | 0.13    |
| Sizer O'size  |                 |       |        |         | 13.76            | 44.34  | 1.87   | 21.42   |
| Comb. Conc    |                 |       |        |         | 25.88            | 83.06  | 3.58   | 31.28   |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 5: Run 103B**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed          | 765             | 18.95 | 71.66  | 46.00   | 769              | 19.14  | 71.69  | 45.52   |
| Rghr Tail     | 543             | 4.03  | 93.02  | 43.00   | 562              | 3.87   | 92.98  | 43.31   |
| Rghr Conc     | 222             | 61.00 | 14.90  | 61.90   | 207              | 60.61  | 13.86  | 51.50   |
| Sizer U'size  | 104             | 59.93 | 14.45  | 40.90   | 110              | 58.52  | 16.16  | 30.84   |
| Clnr Conc     | 96              | 60.80 | 12.50  | 27.60   | 103              | 62.00  | 11.45  | 32.46   |
| Clnr Tail     | 8               | 6.20  | 90.00  | 6.40    | 7                | 6.20   | 87.12  | 6.42    |
| Sizer O'size  | 100             | 63.14 | 11.13  | 71.50   | 97               | 62.98  | 11.24  | 75.07   |
| Comb. Conc    | 196             | 62.19 | 11.03  | 49.96   | 200              | 62.47  | 11.35  | 53.05   |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| Feed          |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rghr Tail     |                 |       |        |         | 73.09            | 14.79  | 94.80  | 69.55   |
| Rghr Conc     |                 |       |        |         | 26.91            | 85.21  | 5.20   | 30.45   |
| Sizer U'size  |                 |       |        |         | 14.34            | 43.84  | 3.23   | 9.72    |
| Clnr Conc     |                 |       |        |         | 13.45            | 43.55  | 2.15   | 9.59    |
| Clnr Tail     |                 |       |        |         | 0.89             | 0.29   | 1.08   | 0.13    |
| Sizer O'size  |                 |       |        |         | 12.57            | 41.37  | 1.97   | 20.74   |
| Comb. Conc    |                 |       |        |         | 26.02            | 84.92  | 4.12   | 30.33   |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 5: Run 103C**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed          | 702             | 19.92 | 71.26  | 47.90   | 767              | 20.15  | 70.74  | 48.30   |
| Rghr Tail     | 530             | 3.23  | 94.00  | 42.40   | 546              | 3.12   | 94.65  | 42.18   |
| Rghr Conc     | 172             | 61.20 | 12.47  | 65.30   | 221              | 62.25  | 11.63  | 63.43   |
| Sizer U'size  | 173             | 61.10 | 12.80  | 58.70   | 158              | 61.65  | 12.24  | 54.40   |
| Clnr Conc     | 168             | 63.92 | 9.45   | 52.80   | 153              | 63.32  | 9.87   | 55.73   |
| Clnr Tail     | 4               | 6.50  | 89.07  | 10.60   | 5                | 6.50   | 90.77  | 10.61   |
| Sizer O'size  | 65              | 63.73 | 10.08  | 86.40   | 63               | 63.72  | 10.14  | 85.90   |
| Comb. Conc    | 234             | 63.87 | 9.63   | 62.20   | 216              | 63.44  | 9.94   | 64.56   |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| Feed          |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rghr Tail     |                 |       |        |         | 71.19            | 11.02  | 95.26  | 62.17   |
| Rghr Conc     |                 |       |        |         | 28.81            | 88.98  | 4.74   | 37.83   |
| Sizer U'size  |                 |       |        |         | 20.55            | 62.88  | 3.56   | 23.15   |
| Clnr Conc     |                 |       |        |         | 19.95            | 62.69  | 2.78   | 23.02   |
| Clnr Tail     |                 |       |        |         | 0.60             | 0.19   | 0.77   | 0.13    |
| Sizer O'size  |                 |       |        |         | 8.25             | 26.10  | 1.18   | 14.68   |
| Comb. Conc    |                 |       |        |         | 28.21            | 88.78  | 3.97   | 37.70   |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 5: Run 103D**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed          | 876             | 20.18 | 71.00  | 49.00   | 929              | 20.43  | 70.81  | 47.78   |
| Rghr Tail     | 631             | 3.27  | 94.65  | 42.30   | 653              | 3.15   | 94.89  | 42.97   |
| Rghr Conc     | 245             | 62.30 | 12.72  | 66.00   | 276              | 61.26  | 13.89  | 59.13   |
| Sizer U'size  | 221             | 61.60 | 14.50  | 66.20   | 220              | 60.88  | 14.52  | 56.47   |
| Clnr Conc     | 209             | 63.73 | 11.71  | 57.90   | 209              | 63.55  | 10.94  | 58.13   |
| Clnr Tail     | 12              | 11.30 | 83.10  | 25.40   | 11               | 11.30  | 81.04  | 25.50   |
| Sizer O'size  | 57              | 62.98 | 11.67  | 72.60   | 56               | 62.74  | 11.47  | 69.50   |
| Comb. Conc    | 266             | 62.19 | 11.03  | 49.96   | 265              | 63.38  | 11.05  | 60.55   |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| Feed          |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rghr Tail     |                 |       |        |         | 70.27            | 10.85  | 94.17  | 63.21   |
| Rghr Conc     |                 |       |        |         | 29.73            | 89.15  | 5.83   | 36.79   |
| Sizer U'size  |                 |       |        |         | 23.66            | 70.51  | 4.85   | 27.96   |
| Clnr Conc     |                 |       |        |         | 22.45            | 69.84  | 3.47   | 27.32   |
| Clnr Tail     |                 |       |        |         | 1.21             | 0.67   | 1.38   | 0.64    |
| Sizer O'size  |                 |       |        |         | 6.07             | 18.64  | 0.98   | 8.83    |
| Comb. Conc    |                 |       |        |         | 28.52            | 88.48  | 4.45   | 36.15   |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 5: Run 103E**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed          | 852             | 21.66 | 69.16  | 48.80   | 853              | 21.48  | 69.33  | 47.56   |
| Rghr Tail     | 604             | 2.70  | 95.70  | 41.80   | 584              | 2.75   | 95.47  | 42.43   |
| Rghr Conc     | 248             | 64.06 | 11.03  | 62.20   | 270              | 62.04  | 12.74  | 58.69   |
| Sizer U'size  | 195             | 62.08 | 13.36  | 61.60   | 201              | 61.61  | 12.96  | 55.60   |
| Clnr Conc     | 179             | 65.05 | 9.32   | 59.20   | 187              | 64.43  | 9.21   | 56.86   |
| Clnr Tail     | 16              | 23.93 | 65.75  | 38.50   | 14               | 23.94  | 63.27  | 38.73   |
| Sizer O'size  | 67              | 63.67 | 12.25  | 71.80   | 68               | 63.29  | 12.07  | 67.78   |
| Comb. Conc    | 247             | 62.19 | 11.03  | 49.96   | 256              | 64.12  | 9.97   | 59.78   |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| Feed          |                 |       |        |         | 100.00           | 100.00 | 100.00 | 100.00  |
| Rghr Tail     |                 |       |        |         | 68.41            | 8.75   | 94.20  | 61.02   |
| Rghr Conc     |                 |       |        |         | 31.59            | 91.25  | 5.80   | 38.98   |
| Sizer U'size  |                 |       |        |         | 23.59            | 67.66  | 4.41   | 27.58   |
| Clnr Conc     |                 |       |        |         | 21.95            | 65.84  | 2.91   | 26.24   |
| Clnr Tail     |                 |       |        |         | 1.64             | 1.83   | 1.50   | 1.34    |
| Sizer O'size  |                 |       |        |         | 8.01             | 23.59  | 1.39   | 11.41   |
| Comb. Conc    |                 |       |        |         | 29.95            | 89.42  | 4.31   | 37.65   |

**Appendix C**

**MATERIAL BALANCES – PLANT 4 FEED TEST RUNS**

**MATERIAL BALANCES – PLANT 4 FEED TEST RUNS**

**CIRCUIT: JACOBS Pilot Plant, Crago Flowsheet: Run 12**

| Stream               | Measured Values |       |        |         | Estimated Values |        |        |         |
|----------------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|                      | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed                 | 916             | 22.30 | 69.12  | 15.30   | 954              | 22.79  | 68.28  | 15.47   |
| Rgh'r tail           | 631             | 3.33  | 93.95  | 18.60   | 635              | 3.28   | 94.98  | 18.44   |
| Rgh'r conc           | 285             | 61.70 | 15.24  | 8.00    | 319              | 61.59  | 15.16  | 9.55    |
| De-oil O'flow        | 0               | 52.43 | 27.15  | 0.10    | 0                | 52.43  | 27.15  | 0.10    |
| Cln'r feed           | 345             | 61.74 | 14.86  | 11.03   | 319              | 61.60  | 15.15  | 9.56    |
| Cln'r conc.          | 298             | 69.60 | 4.12   | 12.50   | 274              | 69.79  | 4.11   | 10.85   |
| Cln'r tail           | 47              | 11.80 | 83.10  | 1.70    | 45               | 11.80  | 82.24  | 1.69    |
| <b>DISTRIBUTIONS</b> |                 |       |        |         |                  |        |        |         |
| Feed                 |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rgh'r tail           |                 |       |        |         | 66.55%           | 9.59%  | 92.58% | 79.35%  |
| Rgh'r conc           |                 |       |        |         | 33.45%           | 90.41% | 7.42%  | 20.65%  |
| De-oil O'flow        |                 |       |        |         | 0.03%            | 0.07%  | 0.01%  | 0.00%   |
| Cln'r feed           |                 |       |        |         | 33.42%           | 90.33% | 7.41%  | 20.65%  |
| Cln'r conc.          |                 |       |        |         | 28.69%           | 87.89% | 1.73%  | 20.14%  |
| Cln'r tail           |                 |       |        |         | 4.72%            | 2.44%  | 5.69%  | 0.52%   |

**CIRCUIT: JACOBS Pilot Plant, Crago Flowsheet: Run 18**

| Stream               | Measured Values |       |        |         | Estimated Values |        |        |         |
|----------------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|                      | Lbs/hr          | %BPL  | %Insol | % >48 M | Lbs/hr           | %BPL   | %Insol | % >48 M |
| Feed                 | 898             | 17.17 | 74.88  | 21.50   | 903              | 16.88  | 74.91  | 21.75   |
| Rgh'r tail           | 653             | 1.88  | 96.20  | 24.90   | 653              | 1.90   | 96.18  | 24.66   |
| Rgh'r conc           | 245             | 57.67 | 17.30  | 12.43   | 251              | 55.87  | 19.54  | 14.19   |
| De-oil O'flow        | 0               | 48.80 | 27.37  | 0.01    | 0                | 48.80  | 27.37  | 0.01    |
| Cln'r feed           | 245             | 54.18 | 22.51  | 15.30   | 251              | 55.87  | 19.54  | 14.19   |
| Cln'r conc.          | 190             | 67.62 | 3.62   | 18.50   | 202              | 67.55  | 3.60   | 16.61   |
| Cln'r tail           | 55              | 7.61  | 87.95  | 4.20    | 49               | 7.61   | 85.45  | 4.18    |
| <b>DISTRIBUTIONS</b> |                 |       |        |         |                  |        |        |         |
| Feed                 |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rgh'r tail           |                 |       |        |         | 72.24%           | 8.11%  | 92.76% | 81.89%  |
| Rgh'r conc           |                 |       |        |         | 27.76%           | 91.89% | 7.24%  | 18.11%  |
| De-oil O'flow        |                 |       |        |         | 0.01%            | 0.03%  | 0.00%  | 0.00%   |
| Cln'r feed           |                 |       |        |         | 27.75%           | 91.85% | 7.24%  | 18.11%  |
| Cln'r conc.          |                 |       |        |         | 22.35%           | 89.42% | 1.07%  | 17.07%  |
| Cln'r tail           |                 |       |        |         | 5.40%            | 2.44%  | 6.16%  | 1.04%   |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 13**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| New Feed      | 1,053           | 21.75 | 69.38  | 14.50   | 948              | 21.63  | 69.50  | 14.59   |
| Rghr Feed     | 1,057           | 21.85 | 69.25  | 14.72   | 957              | 21.86  | 69.19  | 15.09   |
| Rghr Tail     | 600             | 2.22  | 96.07  | 17.20   | 622              | 2.24   | 96.00  | 16.79   |
| Rghr Conc     | 457             | 57.20 | 21.20  | 13.00   | 334              | 58.39  | 19.29  | 11.93   |
| Clnr Conc     | 420             | 65.43 | 10.80  | 10.80   | 291              | 64.15  | 11.23  | 11.37   |
| Clnr Tail     | 38              | 19.57 | 72.06  | 15.00   | 43               | 19.34  | 73.85  | 15.69   |
| Sizer O'size  | 7               | 46.71 | 36.08  | 70.70   | 9                | 46.96  | 36.13  | 68.30   |
| Sizer U'size  | 33              | 11.85 | 82.65  | 1.90    | 34               | 12.10  | 83.74  | 1.89    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Feed     |                 |       |        |         | 100.9%           | 102.0% | 100.5% | 104.4%  |
| Rghr Tail     |                 |       |        |         | 65.7%            | 6.8%   | 90.7%  | 75.6%   |
| Rghr Conc     |                 |       |        |         | 35.3%            | 95.3%  | 9.8%   | 28.8%   |
| Clnr Conc     |                 |       |        |         | 30.7%            | 91.2%  | 5.0%   | 24.0%   |
| Clnr Tail     |                 |       |        |         | 4.5%             | 4.1%   | 4.8%   | 4.9%    |
| Sizer O'size  |                 |       |        |         | 0.9%             | 2.0%   | 0.5%   | 4.4%    |
| Sizer U'size  |                 |       |        |         | 3.6%             | 2.0%   | 4.3%   | 0.5%    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 15**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| New Feed      | 1,050           | 22.20 | 69.25  | 12.90   | 1,029            | 22.23  | 69.17  | 13.19   |
| Rghr Feed     | 1,057           | 22.41 | 68.98  | 13.25   | 1,037            | 22.47  | 68.85  | 13.67   |
| Rghr Tail     | 631             | 2.40  | 95.90  | 15.50   | 682              | 2.38   | 96.16  | 14.84   |
| Rghr Conc     | 427             | 60.53 | 17.13  | 13.20   | 355              | 60.99  | 16.48  | 11.42   |
| Clnr Conc     | 384             | 66.97 | 8.86   | 10.20   | 311              | 66.42  | 9.01   | 11.00   |
| Clnr Tail     | 43              | 23.30 | 67.77  | 18.50   | 45               | 23.28  | 68.19  | 14.35   |
| Sizer O'size  | 7               | 53.40 | 27.85  | 65.10   | 8                | 53.41  | 27.88  | 75.24   |
| Sizer U'size  | 36              | 16.67 | 76.12  | 1.00    | 37               | 16.68  | 77.03  | 1.01    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Feed     |                 |       |        |         | 100.8%           | 101.9% | 100.3% | 104.5%  |
| Rghr Tail     |                 |       |        |         | 66.2%            | 7.1%   | 92.1%  | 74.6%   |
| Rghr Conc     |                 |       |        |         | 34.5%            | 94.8%  | 8.2%   | 29.9%   |
| Clnr Conc     |                 |       |        |         | 30.2%            | 90.2%  | 3.9%   | 25.2%   |
| Clnr Tail     |                 |       |        |         | 4.4%             | 4.6%   | 4.3%   | 4.7%    |
| Sizer O'size  |                 |       |        |         | 0.8%             | 1.9%   | 0.3%   | 4.5%    |
| Sizer U'size  |                 |       |        |         | 3.6%             | 2.7%   | 4.0%   | 0.3%    |



**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 16**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| New Feed      | 1,004           | 22.00 | 69.62  | 15.30   | 992              | 21.99  | 69.45  | 15.29   |
| Rghr Feed     | 1,017           | 22.39 | 69.10  | 15.92   | 1,005            | 22.41  | 68.90  | 16.05   |
| Rghr Tail     | 638             | 2.48  | 95.58  | 17.70   | 662              | 2.48   | 96.04  | 17.61   |
| Rghr Conc     | 378             | 60.53 | 17.13  | 13.50   | 343              | 60.82  | 16.61  | 13.04   |
| Clnr Conc     | 335             | 66.17 | 9.55   | 11.50   | 298              | 65.87  | 9.69   | 11.77   |
| Clnr Tail     | 43              | 27.63 | 61.46  | 25.20   | 45               | 27.69  | 61.98  | 21.41   |
| Sizer O'size  | 13              | 53.50 | 27.36  | 65.40   | 13               | 53.43  | 27.38  | 73.23   |
| Sizer U'size  | 31              | 17.17 | 75.80  | 0.10    | 32               | 17.10  | 76.21  | 0.10    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Feed     |                 |       |        |         | 101.3%           | 103.2% | 100.5% | 106.4%  |
| Rghr Tail     |                 |       |        |         | 66.7%            | 7.5%   | 92.2%  | 76.8%   |
| Rghr Conc     |                 |       |        |         | 34.6%            | 95.7%  | 8.3%   | 29.5%   |
| Clnr Conc     |                 |       |        |         | 30.0%            | 90.0%  | 4.2%   | 23.1%   |
| Clnr Tail     |                 |       |        |         | 4.6%             | 5.8%   | 4.1%   | 6.4%    |
| Sizer O'size  |                 |       |        |         | 1.3%             | 3.2%   | 0.5%   | 6.4%    |
| Sizer U'size  |                 |       |        |         | 3.2%             | 2.5%   | 3.6%   | 0.0%    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 17**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| New Feed      | 976             | 21.70 | 69.60  | 14.30   | 966              | 21.70  | 69.82  | 13.88   |
| Rghr Feed     | 989             | 22.18 | 68.96  | 14.96   | 980              | 22.21  | 69.13  | 14.68   |
| Rghr Tail     | 669             | 3.86  | 94.32  | 15.40   | 674              | 3.85   | 93.81  | 15.94   |
| Rghr Conc     | 321             | 62.45 | 15.13  | 12.10   | 306              | 62.59  | 14.85  | 11.91   |
| Clnr Conc     | 272             | 67.68 | 8.20   | 10.10   | 256              | 67.52  | 8.27   | 10.30   |
| Clnr Tail     | 49              | 37.30 | 48.60  | 22.60   | 50               | 37.33  | 48.62  | 20.13   |
| Sizer O'size  | 13              | 58.32 | 20.76  | 64.70   | 14               | 58.30  | 20.78  | 70.59   |
| Sizer U'size  | 36              | 29.44 | 58.70  | 1.00    | 36               | 29.38  | 59.17  | 1.00    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Feed     |                 |       |        |         | 101.4%           | 103.8% | 100.4% | 107.2%  |
| Rghr Tail     |                 |       |        |         | 69.7%            | 12.4%  | 93.7%  | 80.0%   |
| Rghr Conc     |                 |       |        |         | 31.7%            | 91.5%  | 6.7%   | 27.2%   |
| Clnr Conc     |                 |       |        |         | 26.5%            | 82.6%  | 3.1%   | 19.7%   |
| Clnr Tail     |                 |       |        |         | 5.2%             | 8.9%   | 3.6%   | 7.5%    |
| Sizer O'size  |                 |       |        |         | 1.4%             | 3.8%   | 0.4%   | 7.2%    |
| Sizer U'size  |                 |       |        |         | 3.8%             | 5.1%   | 3.2%   | 0.3%    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 19**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| New Feed      | 925             | 17.07 | 74.93  | 21.20   | 885              | 16.98  | 74.97  | 21.69   |
| Rghr Feed     | 949             | 17.96 | 73.68  | 22.43   | 910              | 17.98  | 73.57  | 22.79   |
| Rghr Tail     | 642             | 1.21  | 97.47  | 25.80   | 630              | 1.22   | 97.56  | 24.96   |
| Rghr Conc     | 307             | 54.78 | 20.60  | 16.50   | 281              | 55.54  | 19.80  | 17.91   |
| Clnr Conc     | 253             | 62.44 | 10.68  | 16.90   | 222              | 61.73  | 10.85  | 15.61   |
| Clnr Tail     | 54              | 32.20 | 52.70  | 24.60   | 59               | 32.07  | 53.73  | 26.63   |
| Sizer O'size  | 24              | 52.20 | 25.40  | 70.00   | 26               | 52.29  | 25.41  | 60.36   |
| Sizer U'size  | 30              | 16.17 | 75.85  | 0.20    | 33               | 16.22  | 75.93  | 0.20    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Feed     |                 |       |        |         | 102.9%           | 109.0% | 101.0% | 108.1%  |
| Rghr Tail     |                 |       |        |         | 71.2%            | 5.1%   | 92.6%  | 81.9%   |
| Rghr Conc     |                 |       |        |         | 31.8%            | 103.9% | 8.4%   | 26.2%   |
| Clnr Conc     |                 |       |        |         | 25.1%            | 91.4%  | 3.6%   | 18.1%   |
| Clnr Tail     |                 |       |        |         | 6.6%             | 12.5%  | 4.7%   | 8.1%    |
| Sizer O'size  |                 |       |        |         | 2.9%             | 9.0%   | 1.0%   | 8.1%    |
| Sizer U'size  |                 |       |        |         | 3.7%             | 3.5%   | 3.8%   | 0.0%    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 20**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| New Feed      | 888             | 17.37 | 74.84  | 22.00   | 884              | 17.15  | 74.91  | 21.85   |
| Rghr Feed     | 895             | 17.61 | 74.49  | 22.44   | 892              | 17.44  | 74.49  | 22.52   |
| Rghr Tail     | 637             | 1.30  | 97.05  | 24.90   | 643              | 1.34   | 96.96  | 24.97   |
| Rghr Conc     | 259             | 57.77 | 18.32  | 15.30   | 250              | 58.93  | 16.60  | 16.22   |
| Clnr Conc     | 228             | 65.73 | 8.07   | 16.00   | 214              | 64.77  | 8.36   | 15.14   |
| Clnr Tail     | 31              | 23.83 | 64.80  | 38.00   | 36               | 23.78  | 66.21  | 22.72   |
| Sizer O'size  | 7               | 46.72 | 32.00  | 76.00   | 9                | 46.74  | 32.10  | 90.36   |
| Sizer U'size  | 23              | 16.17 | 75.77  | 0.40    | 27               | 16.21  | 77.47  | 0.40    |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| New Feed      |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Feed     |                 |       |        |         | 101.0%           | 102.7% | 100.4% | 104.1%  |
| Rghr Tail     |                 |       |        |         | 72.8%            | 5.7%   | 94.2%  | 83.2%   |
| Rghr Conc     |                 |       |        |         | 28.2%            | 97.0%  | 6.3%   | 21.0%   |
| Clnr Conc     |                 |       |        |         | 24.2%            | 91.5%  | 2.7%   | 16.8%   |
| Clnr Tail     |                 |       |        |         | 4.0%             | 5.6%   | 3.6%   | 4.2%    |
| Sizer O'size  |                 |       |        |         | 1.0%             | 2.7%   | 0.4%   | 4.1%    |
| Sizer U'size  |                 |       |        |         | 3.0%             | 2.9%   | 3.1%   | 0.1%    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 4: Run 21**

| Stream               | Measured Values |       |        |         | Estimated Values |        |        |         |
|----------------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|                      | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| New Feed             | 965             | 17.47 | 74.18  | 20.50   | 939              | 17.43  | 74.20  | 21.08   |
| Rghr Feed            | 976             | 17.79 | 73.72  | 21.08   | 951              | 17.77  | 73.71  | 21.79   |
| Rghr Tail            | 699             | 1.62  | 96.55  | 24.40   | 678              | 1.63   | 96.54  | 23.14   |
| Rghr Conc            | 277             | 57.46 | 17.37  | 18.80   | 273              | 57.80  | 17.06  | 18.42   |
| Clnr Conc            | 241             | 63.08 | 9.91   | 17.30   | 235              | 62.78  | 10.00  | 17.36   |
| Clnr Tail            | 37              | 26.82 | 60.58  | 29.00   | 38               | 26.89  | 60.89  | 25.04   |
| Sizer O'size         | 11              | 44.85 | 35.10  | 70.50   | 12               | 44.78  | 35.12  | 77.94   |
| Sizer U'size         | 25              | 18.85 | 72.40  | 1.00    | 26               | 18.75  | 72.61  | 1.00    |
| <b>DISTRIBUTIONS</b> |                 |       |        |         |                  |        |        |         |
| New Feed             |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Feed            |                 |       |        |         | 101.3%           | 103.2% | 100.6% | 104.7%  |
| Rghr Tail            |                 |       |        |         | 72.2%            | 6.8%   | 93.9%  | 79.2%   |
| Rghr Conc            |                 |       |        |         | 29.1%            | 96.5%  | 6.7%   | 25.4%   |
| Clnr Conc            |                 |       |        |         | 25.1%            | 90.3%  | 3.4%   | 20.6%   |
| Clnr Tail            |                 |       |        |         | 4.0%             | 6.2%   | 3.3%   | 4.8%    |
| Sizer O'size         |                 |       |        |         | 1.3%             | 3.2%   | 0.6%   | 4.7%    |
| Sizer U'size         |                 |       |        |         | 2.8%             | 3.0%   | 2.7%   | 0.1%    |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 5: Run 14**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| Feed          | 884             | 21.65 | 70.03  | 14.50   | 876              | 21.53  | 69.83  | 13.74   |
| Rghr Tail     | 580             | 2.29  | 95.88  | 14.90   | 570              | 2.31   | 96.13  | 15.42   |
| Rghr Conc     | 304             | 57.40 | 20.95  | 11.40   | 306              | 57.36  | 20.79  | 10.61   |
| Sizer U'size  | 195             | 53.93 | 24.93  | 0.10    | 197              | 54.22  | 24.97  | 0.12    |
| Clnr Conc     | 147             | 65.33 | 10.22  | 0.01    | 150              | 65.11  | 10.23  | 0.01    |
| Clnr Tail     | 49              | 19.77 | 71.38  | 1.00    | 47               | 19.76  | 71.60  | 0.48    |
| Sizer O'size  | 108             | 62.95 | 13.20  | 28.40   | 109              | 63.02  | 13.22  | 29.57   |
| Comb. Conc    | 255             | 64.32 | 11.49  | 12.07   | 259              | 64.23  | 11.49  | 12.47   |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| Feed          |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Tail     |                 |       |        |         | 65.1%            | 7.0%   | 89.6%  | 73.0%   |
| Rghr Conc     |                 |       |        |         | 34.9%            | 93.0%  | 10.4%  | 27.0%   |
| Sizer U'size  |                 |       |        |         | 22.5%            | 56.6%  | 8.0%   | 0.2%    |
| Clnr Conc     |                 |       |        |         | 17.1%            | 51.6%  | 2.5%   | 0.0%    |
| Clnr Tail     |                 |       |        |         | 5.4%             | 5.0%   | 5.5%   | 0.2%    |
| Sizer O'size  |                 |       |        |         | 12.4%            | 36.4%  | 2.4%   | 26.8%   |
| Comb. Conc    |                 |       |        |         | 29.5%            | 88.1%  | 4.9%   | 26.8%   |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 5: Run 23**

| Stream        | Measured Values |       |        |         | Estimated Values |        |        |         |
|---------------|-----------------|-------|--------|---------|------------------|--------|--------|---------|
|               | Mass            | %BPL  | %Insol | % >48 M | Mass             | %BPL   | %Insol | % >48 M |
| Feed          | 912             | 18.34 | 74.32  | 20.73   | 855              | 17.90  | 73.92  | 21.13   |
| Rghr Tail     | 669             | 1.18  | 96.87  | 23.50   | 607              | 1.21   | 97.39  | 23.13   |
| Rghr Conc     | 242             | 58.81 | 16.32  | 19.20   | 248              | 58.75  | 16.45  | 16.22   |
| Sizer U'size  | 146             | 54.21 | 21.79  | 1.00    | 147              | 54.54  | 22.22  | 0.56    |
| Clnr Conc     | 122             | 60.75 | 13.83  | 0.25    | 124              | 60.58  | 13.72  | 0.27    |
| Clnr Tail     | 23              | 23.07 | 67.36  | 1.80    | 24               | 23.07  | 66.50  | 2.03    |
| Sizer O'size  | 97              | 64.74 | 7.99   | 37.30   | 101              | 64.91  | 8.00   | 39.16   |
| Comb. Conc    | 219             | 62.51 | 11.25  | 16.59   | 224              | 62.52  | 11.16  | 17.72   |
| DISTRIBUTIONS |                 |       |        |         |                  |        |        |         |
| Feed          |                 |       |        |         | 100.0%           | 100.0% | 100.0% | 100.0%  |
| Rghr Tail     |                 |       |        |         | 71.0%            | 4.8%   | 93.5%  | 77.7%   |
| Rghr Conc     |                 |       |        |         | 29.0%            | 95.2%  | 6.5%   | 22.3%   |
| Sizer U'size  |                 |       |        |         | 17.2%            | 52.5%  | 5.2%   | 0.5%    |
| Clnr Conc     |                 |       |        |         | 14.5%            | 48.9%  | 2.7%   | 0.2%    |
| Clnr Tail     |                 |       |        |         | 2.8%             | 3.6%   | 2.5%   | 0.3%    |
| Sizer O'size  |                 |       |        |         | 11.8%            | 42.7%  | 1.3%   | 21.8%   |
| Comb. Conc    |                 |       |        |         | 26.2%            | 91.6%  | 4.0%   | 22.0%   |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 6: Run 22**

| Stream        | Measured Values |       |        |        | Estimated Values |        |        |        |
|---------------|-----------------|-------|--------|--------|------------------|--------|--------|--------|
|               | Mass            | %BPL  | %Insol | %>48 m | Mass             | %BPL   | %Insol | %>48 m |
| Feed          | 834             | 17.17 | 74.46  | 21.30  | 818              | 17.65  | 75.00  | 21.41  |
| Rgh'r conc    | 137             | 64.36 | 7.80   | 13.30  | 128              | 64.09  | 7.80   | 13.29  |
| Rgh'r tail    | 697             | 7.87  | 87.59  | 22.88  | 690              | 9.05   | 87.45  | 22.91  |
| -48 mesh tail | 242             | 5.94  | 90.15  | 1.00   | 259              | 5.71   | 89.96  | 1.00   |
| Scav'r feed   | 455             | 10.75 | 85.62  | 35.57  | 431              | 11.06  | 85.94  | 36.10  |
| Scav'r conc   | 41              | 63.77 | 9.57   | 28.20  | 37               | 63.49  | 9.57   | 28.16  |
| Scav'r tail   | 348             | 6.63  | 93.80  | 37.60  | 394              | 6.12   | 93.13  | 36.85  |
| DISTRIBUTIONS |                 |       |        |        |                  |        |        |        |
| Feed          |                 |       |        |        | 100.0%           | 100.0% | 100.0% | 100.0% |
| Rgh'r conc    |                 |       |        |        | 15.6%            | 56.8%  | 5.9%   | 3.7%   |
| Rgh'r tail    |                 |       |        |        | 84.4%            | 43.2%  | 50.4%  | 54.0%  |
| -48 mesh tail |                 |       |        |        | 31.7%            | 10.2%  | 12.3%  | 0.6%   |
| Scav'r feed   |                 |       |        |        | 52.7%            | 33.0%  | 37.8%  | 63.7%  |
| Scav'r conc   |                 |       |        |        | 4.5%             | 16.3%  | 2.1%   | 2.7%   |
| Scav'r tail   |                 |       |        |        | 48.1%            | 16.7%  | 20.7%  | 35.7%  |

**CIRCUIT: JACOBS Pilot Plant, Anionic Option 6: Run 24**

| Stream        | Measured Values |       |        |        | Estimated Values |        |        |        |
|---------------|-----------------|-------|--------|--------|------------------|--------|--------|--------|
|               | Mass            | %BPL  | %Insol | %>48 m | Mass             | %BPL   | %Insol | %>48 m |
| Feed          | 835             | 18.15 | 74.37  | 22.20  | 801              | 18.11  | 73.83  | 21.76  |
| Rgh'r conc    | 162             | 64.89 | 7.46   | 15.70  | 163              | 64.99  | 7.46   | 15.74  |
| Rgh'r tail    | 672             | 6.87  | 90.51  | 23.77  | 638              | 6.10   | 90.84  | 23.30  |
| -48 mesh tail | 288             | 4.57  | 93.22  | 0.30   | 285              | 4.67   | 93.37  | 0.30   |
| Scav'r feed   | 385             | 8.59  | 88.49  | 41.30  | 353              | 7.25   | 88.80  | 41.84  |
| Scav'r conc   | 24              | 55.68 | 21.68  | 43.80  | 37               | 56.17  | 21.68  | 43.91  |
| Scav'r tail   | 361             | 1.55  | 96.70  | 40.80  | 317              | 1.60   | 96.55  | 41.60  |
| DISTRIBUTIONS |                 |       |        |        |                  |        |        |        |
| Feed          |                 |       |        |        | 100.0%           | 100.0% | 100.0% | 100.0% |
| Rgh'r conc    |                 |       |        |        | 20.4%            | 73.2%  | 7.4%   | 5.4%   |
| Rgh'r tail    |                 |       |        |        | 79.6%            | 26.8%  | 33.0%  | 35.3%  |
| -48 mesh tail |                 |       |        |        | 35.5%            | 9.2%   | 11.6%  | 0.2%   |
| Scav'r feed   |                 |       |        |        | 44.1%            | 17.6%  | 21.2%  | 40.8%  |
| Scav'r conc   |                 |       |        |        | 4.6%             | 14.1%  | 4.2%   | 8.4%   |
| Scav'r tail   |                 |       |        |        | 39.5%            | 3.5%   | 4.6%   | 8.7%   |