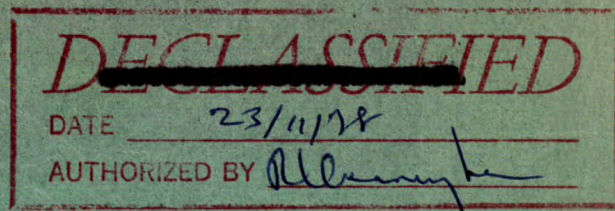


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**CANADA**

**DEPARTMENT OF MINES AND TECHNICAL SURVEYS**

**OTTAWA**

**MINES BRANCH INVESTIGATION REPORT IR 66-3**

**BENEFICIATION OF FLUORITE FROM  
UDAIPUR, INDIA  
(PROJECT MP-IM-6408)**

by

**F. H. HARTMAN AND R. A. WYMAN**

**MINERAL PROCESSING DIVISION**

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F. H. Hartman\* and R. A. Wyman\*\*

- - - -

SUMMARY

A thorough study of the beneficiation prospects for fluorite samples from India was undertaken at the request of Wright Engineers of Vancouver, British Columbia.

A process was worked out for material assaying 17.8%  $\text{CaF}_2$ . This consisted of normal flotation with 4 lb of sodium carbonate per ton and 1 lb of oleic acid per ton of feed ground to at least 85 per cent minus 200 mesh, regrinding the float to at least 90 per cent minus 325 mesh, hot conditioning, adding 0.5 lb quebracho per ton and cleaning by further flotation to an acid grade product, e. g. an 86.4% recovery of 98.1%  $\text{CaF}_2$ ; a 92.1% recovery of 97.2%  $\text{CaF}_2$ .

Metallurgical grade fluorite could be obtained by diverting some of the flow from the above circuit after the first cleaning stage. Metallurgical grade was also obtained by sink-float treatment of one portion of the sample containing approximately 24%  $\text{CaF}_2$  at 1/2 inch size.

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## INTRODUCTION

The National Metallurgical Laboratory of India investigated the possibility of beneficiating fluorite ores from Udaipur for the Government of Rajasthan. Their batch and pilot plant studies indicated that recovery of fluorite from this source would be possible.

Wright Engineers Limited of Vancouver, were asked by the Government of Rajasthan to make a feasibility study of a 300 tpd fluorite mill. Wright Engineers Limited then requested the Mines Branch to provide metallurgical information to be used in development of this study and plant design. Three samples, one of very low grade and two of low grade by the usual standards, were sent from India. The Mines Branch undertook to make trials on the higher grade materials and, if these proved successful, to continue the test work on the lower grade feed derived from mixing the three samples.

The text of this report develops numerous factors pertaining to the problem. A highly effective process was evolved. A flow sheet is suggested. A complete record of the tests performed and the results obtained is given in the Appendix.

## DESCRIPTION OF SAMPLES

Two boxes, containing a total of three bags, were received from India. The three samples may be described as follows:

Bag 1: Marked "2 of 2, 7%"; approximately 100 lb of  
1 1/2 inch pieces.

Bag 2: Marked "1 of 2, 26%"; approximately 150 lb of  
3/4 inch to fines.

Bag 3: Marked "1 of 2, 26%"; approximately 70 lb of  
1/2 inch to fines.

Representative portions riffled from each bag, were analysed for  $\text{CaF}_2$  only (see Table 1), because extensive chemical analyses of the various deposits had already been reported by the National Metallurgical Laboratory (NML) of India.

TABLE 1

Fluorite Content of Samples

| Sample             | %CaF <sub>2</sub> |
|--------------------|-------------------|
| Bag 1 (Marked 7%)  | 4.29              |
| Bag 2 (Marked 26%) | 23.36             |
| Bag 3 (Marked 26%) | 24.81             |

Throughout this report the term "high grade" has been applied to mixtures of the Bag 2 and 3 material. The term "mixed grade" has been applied to mixtures of all three samples in proportion to weight received. On the basis of the above analysis, the mixed grade material would contain 17.7% CaF<sub>2</sub>.

A mineralogical examination\* indicated quartz to be the principal constituent of each sample. Fluorite was second in abundance in Bags 2 and 3, followed by feldspar. In Bag 1, the order was feldspar followed by fluorite. Minor amounts of mica, chlorite and calcite were observed in all three samples, with larger amounts of these minerals in Bag 1 than in the other two.

ANALYSIS

Since the pertinent information required throughout the test program was distribution of fluorite in the products of the experiments performed, CaF<sub>2</sub> determinations\*\* only were used to assess the results obtained.

INITIAL TEST WORK

Sink-Float Tests

Sink and float separations in tetrabromoethane (sp gr 2.96) were made on fractions of the high grade material between 1/2 inch and 35 mesh. The results obtained are summarized in Table 2.

---

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\*\*All determinations were made by S. T. Lepage, technician, under the supervision of G. A. Kent, Senior Scientific Officer, Mineral Processing Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

TABLE 2

Sink-Float Tests on High Grade

| Fraction      | Wt %  | Sink |                  |                    |
|---------------|-------|------|------------------|--------------------|
|               |       | Wt % | Wt % of Fraction | CaF <sub>2</sub> % |
| +1/2 in.      | 19.4  | 23.1 | 4.5              | 79.59              |
| -1/2 in. +4 m | 57.8  | 9.0  | 5.2              | 86.82              |
| -4 +10 m      | 14.2  | 17.0 | 2.4              | 88.63              |
| -10 +35 m     | 5.9   | 22.8 | 1.4              | 91.28              |
| -35 m         | 2.7   | -    | -                | -                  |
|               | 100.0 | -    | 13.5*            | 86.40              |

\*Based on 24% CaF<sub>2</sub> in feed, this represents a recovery of 49%.

Comminution

The Bag 1 sample was reduced to minus 1/2 inch to make it similar in size to the other two samples.

Portions of Bag 2 and 3 samples were mixed in proportion to weight received and the size distribution was obtained, as shown in Table 3.

TABLE 3

Screen Analysis of High Grade

| Fraction      | Weight % |
|---------------|----------|
| +1/2 in.      | 19.4     |
| -1/2 in. +4 m | 57.8     |
| -4 +10 m      | 14.2     |
| -10 +35 m     | 5.9      |
| -35 m         | 2.7      |
|               | 100.0    |

Further reduction of this material by jaw and rolls crushing produced a minus 28 mesh product with the size distribution indicated in Table 4.



TABLE 4

Screen Analysis of Crushed High Grade

| Fraction    | Weight % |
|-------------|----------|
| - 28 + 35 m | 22.6     |
| - 35 + 48 m | 19.7     |
| - 48 + 65 m | 14.9     |
| - 65 +100 m | 11.0     |
| -100 +150 m | 7.9      |
| -150 +200 m | 5.8      |
| -200 m      | 18.1     |
|             | 100.0    |

Grinding tests were carried out on the minus 28 mesh material in an Abbé jar mill. The load was calculated to slightly more than fill the voids in the 3000 gram flint pebble charge. The density was 50% solids. Size distributions of the products obtained for several grinding times are given in Table 5.

TABLE 5

Grinding Tests on High Grade

| Fraction (mesh) | 15 min (%) | 30 min (%) | 45 min (%) |
|-----------------|------------|------------|------------|
| - 28 + 35       | 0.2        | -          | -          |
| - 35 + 48       | 3.6        | -          | -          |
| - 48 + 65       | 7.6        | 1.0        | 0.2        |
| - 65 +100       | 16.2       | 3.8        | 0.7        |
| -100 +150       | 19.2       | 12.0       | 4.2        |
| -150 +200       | 10.8       | 13.4       | 7.8        |
| -200            | 42.4       | 69.8       | 87.1       |
|                 | 100.0      | 100.0      | 100.0      |

Flotation

Two flotation tests were performed, one with the material ground for 30 minutes and the other with material ground for 45 minutes. "Hot conditioning" was used, i. e. a pulp made up of the feed plus sodium carbonate and oleic acid in water was heated to boiling. This was cooled by dilution, quebracho added, and the flotation carried out. Results are compared in Table 6. Conditions other than particle size are the same.

TABLE 6

Flotation of High Grade

| Test No. | % -200 m | Wt % | CaF <sub>2</sub> % | Recovery % |
|----------|----------|------|--------------------|------------|
| 1        | 69.8     | 18.9 | 92.53              | 81.3       |
| 2        | 87.1     | 18.4 | 96.10              | 80.2       |

The above comparison is made on the basis of 3 cleaning stages; Test 2, when carried to 5 cleaning stages, produced a grade of 97.36% CaF<sub>2</sub> for a recovery of 75.5%.

Summary

The results of these tests were considered to be a sufficient indication that the program on mixed grade material should go forward.

TEST WORK ON MIXED GRADE FEED

The tests on high grade material were conducted with the hot conditioning technique, known to be selective for fluorite, but involving the added expense of heating the pulp prior to flotation. In order to assess the prospects fully, it was decided that test work should be conducted on the mixed grade material both with hot conditioning and normal conditioning. The latter simply means that the tests are conducted at room temperature with water delivered through the regular mains.

In the hot conditioning technique, the pH regulator (sodium carbonate or "soda ash") and the promoter (oleic acid) are added in the required amounts prior to heating. The control agent (quebracho) is usually added after heating.

For normal conditioning, the pH regulator is added first, then the promoter. The promoter may be added in one lot (bulk addition), or in a series of smaller amounts with a period of flotation between each addition (step addition). The control agent may be added both in the rougher (initial) flotation stage and in the cleaner (subsequent) flotation stages. Normal conditioning requires roughly twice the number of cleaning stages as hot conditioning to produce approximately the same grade.

Alphabetical designations included as part of the Nos. for tests on the mixed grade material indicate different lots made up from the original samples. The analyses of these varied from 16.8 to 18.2 %  $\text{CaF}_2$  with an average of 17.4%, substantial agreement with the original analysis of 17.7%  $\text{CaF}_2$  for mixed grade.

A: Normal Conditioning

1) Bulk vs Step Addition of Promoter

This applies only to the rougher stage and two tests are compared in Table 7. All conditions for these tests are the same except for the method of promoter addition.

TABLE 7

Bulk vs Step Addition of Promoter

| Test No.          | 20-D                     | 26-D                     |
|-------------------|--------------------------|--------------------------|
| Promoter addition | Bulk                     | 7 steps                  |
| Fraction          | Loss of $\text{CaF}_2$ % | Loss of $\text{CaF}_2$ % |
| Rougher tails     | 5.1                      | 2.9                      |

It will be observed from Table 7 that the step addition gave a more complete recovery of the fluorite in the rougher and for this reason the step addition was used in most of the tests involving normal rougher flotation. Losses were higher in each comparable cleaner stage following the step addition to produce an overall lower recovery. Carrying test 26-D to twelve cleaners produced a 64.4% recovery, grading 97.00%  $\text{CaF}_2$  (see appendix).

It was observed in the later stages of the investigation that it was only necessary to extend the rougher float time to approximately that of the step method in order to obtain similar rougher results by the bulk addition method. This is indicated by Table 8, which reports rougher tails for the tests involved. Test 6-B, employing step addition, is included for comparison.

TABLE 8

Rougher Flotation Results

| Test No.                                    | 20-D | 39-E | 42-E | 6-B  |
|---|------|------|------|------|
| Promoter addition                           | Bulk | Bulk | Bulk | Bulk |
| Float time (min)                            | 15   | 24   | 24   | 24   |
| Rougher Tails (% loss of CaF <sub>2</sub> ) | 5.1  | 2.6  | 2.6  | 2.1  |

2) The Effect of Quebracho

The effect of quebracho in grade control was studied using normal conditioning, and step addition of promoter in the rougher stage. Rougher results are remarkably similar for the six tests included, and range from 2.1 to 3.0% fluorite loss to rougher tails. An addition of 0.5 lb of quebracho per ton was used in the rougher in each test, with further additions to cleaning stages as indicated in Table 9. In this table, the products for the various tests at comparable cleaning stages are shown. Test 6-B, with no quebracho added to the cleaning stages, is included for comparison. In Test 17-C, only 5 cleaning stages were used.

TABLE 9

The Effect of Quebracho

| With 7 Cleaning Stages |                                      |                                |                 |       |
|------------------------|--------------------------------------|--------------------------------|-----------------|-------|
| Test No.               | Quebracho added to Cleaners (lb/ton) | Grade--G (% CaF <sub>2</sub> ) | Recovery--R (%) | G x R |
| 6-B                    | 0                                    | 87.25                          | 81.3            | 7085  |
| 7-B                    | 0.12 to C1 5                         | 98.13                          | 18.2            | 1786  |
| 11-C                   | 0.0015 to C1 2, 4                    | 88.25                          | 83.2            | 7350  |
| 26-D                   | 0.0015 to all C1                     | 92.00                          | 80.1            | 7375  |
| 16-C                   | 0.0030 to all C1                     | 95.55                          | 40.7            | 3895  |
| 17-C                   | 0.0045 to all C1                     | 96.86                          | 13.4            | 1296  |

| With 10 Cleaning Stages |                                      |                                |                 |       |
|-------------------------|--------------------------------------|--------------------------------|-----------------|-------|
| Test No.                | Quebracho added to Cleaners (lb/ton) | Grade--G (% CaF <sub>2</sub> ) | Recovery--R (%) | G x R |
| 6-B                     | 0                                    | 89.18                          | 80.5            | 7175  |
| 11-C                    | 0.0015 to C1 2                       | 93.83                          | 75.4            | 7065  |
|                         | 0.0015 to C1 4                       |                                |                 |       |
|                         | 0.0030 to C1 8                       |                                |                 |       |
|                         | 0.0045 to C1 9                       |                                |                 |       |
|                         | 0.0060 to C1 10                      |                                |                 |       |
|                         | 0.0165 Total                         |                                |                 |       |
| 26-D                    | 0.0015 to all C1                     | 96.00                          | 73.2            | 7025  |
|                         | 0.0150 Total                         |                                |                 |       |
| With 12 Cleaning Stages |                                      |                                |                 |       |
| 26-D                    | 0.0015 to all C1                     | 97.00                          | 64.4            | 6250  |
|                         | 0.0165 Total                         |                                |                 |       |

The effect of quebracho is to increase grade at the expense of recovery. However it does provide a control mechanism to allow production of higher grades. The net change between cleaners 7 and 10 in Test 6-B was a 2% grade increase and a 1% recovery loss. For Test 26-D, the quebracho caused a gain of 4% in grade with a 7% sacrifice of recovery between the same cleaning stages. The grade level after 10 cleaners is, however, 96% for Test 26-D and only 89% for Test 6-B. Tests such as 7-B, 16-C and 17-C show the effect of too much quebracho. The control agent must obviously be used with care in this system.

### 3) Cleaning Time

The effect of shortening the cleaning time is to leave more fluorite in each cleaner tails with a resulting product of improved grade, but at a large sacrifice in recovery. Two tests, employing the same conditions in all respects but float time in the cleaning stages, are compared in Table 10.

TABLE 10

The Effect of Cleaning Time

| Fraction                                    | Test 6-B                      |                     | Test 8-B                      |                     |
|---|-------------------------------|---------------------|-------------------------------|---------------------|
|   | % Dist<br>of CaF <sub>2</sub> | Float time<br>(min) | % Dist<br>of CaF <sub>2</sub> | Float time<br>(min) |
| Concentrate<br>(% CaF <sub>2</sub> in Conc) | 80.5<br>(89.18)               |                     | 61.0<br>(93.63)               |                     |
| C1 10 Tails                                 | 0.3                           | 3.5                 | 2.7                           | 2.0                 |
| C1 9 "                                      | 0.2                           | 3.5                 | 3.0                           | 2.0                 |
| C1 8 "                                      | 0.3                           | 3.5                 | 3.2                           | 2.0                 |
| C1 7 "                                      | 0.3                           | 3.5                 | 2.5                           | 2.5                 |
| C1 6 "                                      | 0.3                           | 4.0                 | 2.6                           | 2.5                 |
| C1 5 "                                      | 0.5                           | 4.0                 | 2.6                           | 3.0                 |
| C1 4 "                                      | 0.6                           | 4.0                 | 2.5                           | 3.0                 |
| C1 3 "                                      | 1.2                           | 5.0                 | 3.2                           | 3.0                 |
| C1 2 "                                      | 2.6                           | 6.5                 | 4.6                           | 4.0                 |
| C1 1 "                                      | 11.1                          | 7.5                 | 9.2                           | 7.0                 |
| Rougher "                                   | 2.1                           | 24.5                | 2.9                           | 24.0                |

4) The Effect of Temperature

A comparison was made between flotation with water at the delivered temperature of 6 to 8° C, and at 25° C. The effect of lower water temperature was similar to that of shorter flotation time -- more fluorite remained in each cleaner with a resulting high grade for low recovery. For the tests shown in Table 11, all conditions but pulp temperature are the same.

TABLE 11

The Effect of Temperature

| Fraction                                    | Test 25-D               |         | Test 26-D               |         |
|---|-------------------------|---------|-------------------------|---------|
|   | Dist CaF <sub>2</sub> % | Temp °C | Dist CaF <sub>2</sub> % | Temp °C |
| Concentrate<br>(% CaF <sub>2</sub> in Conc) | 87.5<br>(91.64)         |         | 64.4<br>(97.00)         |         |
| Cl 12 Tails                                 | 1.0                     | 25      | 5.3                     | 6       |
| Cl 11 "                                     | 0.8                     | 25      | 3.5                     | 6       |
| Cl 10 "                                     | 0.6                     | 25      | 3.2                     | 6       |
| Cl 9 "                                      | 0.6                     | 25      | 2.3                     | 6       |
| Cl 8 "                                      | 0.7                     | 25      | 1.4                     | 6       |
| Cl 7 "                                      | 0.6                     | 25      | 1.3                     | 6       |
| Cl 6 "                                      | 0.5                     | 25      | 1.2                     | 6       |
| Cl 5 "                                      | 0.5                     | 25      | 1.0                     | 7       |
| Cl 4 "                                      | 0.7                     | 25      | 1.1                     | 7       |
| Cl 3 "                                      | 0.8                     | 25      | 1.3                     | 8       |
| Cl 2 "                                      | 1.2                     | 25      | 1.8                     | 9       |
| Cl 1 "                                      | 2.1                     | 25      | 9.3                     | 10      |
| Rougher "                                   | 2.4                     | 25      | 2.9                     | 15      |

5) Summary

The normal conditioning method provides low losses to rougher tails. However, a large number of cleaning stages are necessary, even with quebracho as a control agent, to obtain a high grade product. Lower pulp temperature for cleaning, and reduced cleaning time, both aid in developing a high product grade at the expense of recovery.

B: Hot Conditioning

1) Hot vs Normal Conditioning

A comparison of the products obtained with hot and normal conditioning systems is given in Table 12. Tests 18-C and 22-D, examples of hot conditioning, differ only in the fatty acid used as promoter. Each has 6 cleaning stages. Test 26-D, the example of normal conditioning, has 12 cleaning stages, and also 0.0165 lb/ton more quebracho than tests 18-C and 22-D. The additional quebracho was used in the cleaning stages for grade control.

TABLE 12

Hot vs Normal Conditioning

| Test No. | Wt % | CaF <sub>2</sub> % | Recov % | Remarks             |
|----------|------|--------------------|---------|---------------------|
| 18-C     | 13.6 | 97.29              | 80.0    | Hot: 6 Cleaners     |
| 22-D     | 15.7 | 96.71              | 88.2    | " "                 |
| 26-D     | 11.4 | 97.00              | 64.4    | Normal: 12 Cleaners |

2) The Effect of Feed Fineness

An indication of the fineness to which this feed must be ground prior to hot conditioning is given in Table 13. Here the products of two tests that are similar with the exception of feed fineness are compared.

TABLE 13

The Effect of Feed Fineness

| Test No. | % -200 m | Wt % | CaF <sub>2</sub> % | Recovery % |
|----------|----------|------|--------------------|------------|
| 3-B      | 87.1     | 15.5 | 96.37              | 82.6       |
| 4-B      | 69.8     | 13.4 | 95.83              | 70.4       |

3) Quebracho Requirement

The question of whether quebracho is required in the hot conditioning system was explored with two different fatty acid promoters. The conditions for each test reported in Table 14 are similar except as follows:

- 1) In Test 18-C and 31-E the promoter was Harfat 231 (old); no quebracho was used in 31-E.
- 2) In Tests 22-D and 30-D the promoter was Distilled Oleic Acid; no quebracho was used in 30-D. The products obtained are compared in Table 14.



TABLE 14

Quebracho vs No Quebracho

| Test No. | Promoter   | Quebracho | Wt % | CaF <sub>2</sub> % | Recovery % |
|----------|------------|-----------|------|--------------------|------------|
| 18-C     | Harfat     | yes       | 13.6 | 97.29              | 80.0       |
| 31-E     | "          | no        | 13.1 | 95.92              | 69.6       |
| 22-D     | Dist Oleic | yes       | 15.7 | 96.71              | 88.2       |
| 30-D     | " "        | no        | 15.3 | 93.53              | 87.1       |

Both recovery and grade are shown to be improved through the use of quebracho.

4) Method of Quebracho Application

With similar test conditions, except for the point of quebracho addition, the products from two tests are compared in Table 15. In Test 18-C, the quebracho was added following the hot conditioning, and in 19-C before this step.

TABLE 15

Point of Quebracho Addition

| Test No. | Q.addition     | Wt % | CaF <sub>2</sub> % | Recovery % |
|----------|----------------|------|--------------------|------------|
| 18-C     | After Heating  | 13.6 | 97.29              | 80.0       |
| 19-C     | Before Heating | 16.2 | 92.41              | 89.6       |

These results indicate that better cleaning is achieved when the quebracho is added after the hot conditioning step.

5) Response to Promoters

Five fatty acids were compared for their promoting qualities. All test conditions were the same with the exception of the promoter employed. The products of the tests are shown in Table 16.

TABLE 16

Effect of Various Promoters

| Test No. | Promoter          | Wt % | CaF <sub>2</sub> % | Recovery % |
|----------|-------------------|------|--------------------|------------|
| 18-C     | Harfat 231 (old)  | 13.6 | 97.29              | 80.0       |
| 21-D     | " " (new)         | 13.7 | 95.29              | 76.2       |
| 22-D     | Dist Oleic Acid   | 15.7 | 96.71              | 88.2       |
| 23-D     | Oleic No. 4       | 15.7 | 95.42              | 87.6       |
| 24-D     | Undistilled Oleic | 14.5 | 97.20              | 81.8       |

The best overall result was obtained with Distilled Oleic Acid. However, it is apparent that good results may be achieved by this method with a variety of fatty acid promoters.

6) The Effect of Pulp Density

To obtain an indication of the part pulp density plays in this process, the tests recorded in Table 17 were performed. Distilled Oleic Acid was used, and all conditions were the same except for the pulp density at the various stages of roughing and cleaning.

TABLE 17

The Effect of Pulp Density

| Fraction                                    | Test 22-D               |          | Test 36-E               |          |
|---|-------------------------|----------|-------------------------|----------|
|   | Dist CaF <sub>2</sub> % | % Solids | Dist CaF <sub>2</sub> % | % Solids |
| Concentrate<br>(% CaF <sub>2</sub> in Conc) | 88.2<br>(96.71)         |          | 81.9<br>(94.25)         |          |
| C1 Cleaner 6                                |                         | 3.8      |                         | 7.9      |
| C1 6 Tails                                  | 2.2                     |          | 3.3                     |          |
| C1 Cleaner 5                                |                         | 3.8      |                         | 8.2      |
| C1 5 Tails                                  | 0.9                     |          | 2.4                     |          |
| C1 Cleaner 4                                |                         | 3.9      |                         | 8.3      |
| C1 4 Tails                                  | 0.8                     |          | 2.5                     |          |
| C1 Cleaner 3                                |                         | 4.0      |                         | 8.9      |
| C1 3 Tails                                  | 1.1                     |          | 2.3                     |          |
| C1 Cleaner 2                                |                         | 4.4      |                         | 10.0     |
| C1 2 Tails                                  | 1.4                     |          | 3.3                     |          |
| C1 Cleaner 1                                |                         | 5.9      |                         | 13.6     |
| C1 1 Tails                                  | 2.4                     |          | 2.4                     |          |
| Rougher                                     |                         | 20.0     |                         | 35.4     |
| Rougher Tails                               | 3.0                     |          | 1.9                     |          |

A lower loss to rougher tails is indicated for the denser roughing operation, but higher density in the cleaners causes an overall decrease in cleaning efficiency. This could in part be compensated for by increasing the cleaner flotation times, which would cause a recovery increase at the expense of product grade. These tests suggest that roughing at above 30% solids would be appropriate, but that low density cleaning would be preferable.

7) The Effect of Pulp Temperature

In order to gain some knowledge of the effect of pulp temperature on the flotation system, a comparison was made between two tests run with all conditions similar except for the temperature of the pulp during each stage. The results are shown in Table 18.

TABLE 18

The Effect of Pulp Temperature

| Fraction                                    | Test 34-E               |         | Test 35-E               |         |
|---|-------------------------|---------|-------------------------|---------|
|   | Dist CaF <sub>2</sub> % | Temp °C | Dist CaF <sub>2</sub> % | Temp °C |
| Concentrate<br>(% CaF <sub>2</sub> in Conc) | 86.0<br>(96.33)         |         | 76.5<br>(95.80)         |         |
| C1 6 Tails                                  | 2.3                     | 9       | 2.9                     | 23      |
| C1 5 Tails                                  | 0.8                     | 9       | 2.2                     | 23      |
| C1 4 Tails                                  | 1.4                     | 9       | 2.8                     | 23      |
| C1 3 Tails                                  | 1.5                     | 9       | 3.3                     | 23      |
| C1 2 Tails                                  | 2.0                     | 10      | 4.3                     | 22      |
| C1 1 Tails                                  | 4.1                     | 11      | 5.6                     | 24      |
| Rougher Tails                               | 1.9                     | 20      | 2.4                     | 22      |

These figures indicate that once the promoter is settled on the fluorite by hot conditioning the actual recovery process by flotation is better accomplished at low pulp temperatures.

8) Hot Conditioning Temperature

The key to the hot conditioning process for fluorite lies in heating the pulp to near boiling after the addition of the promoter. This seems to cause an almost permanent bond of the promoter to the fluorite. To determine the optimum temperature for this "conditioning" step a series of tests was performed in which the pulps were raised to various temperatures. All other conditions were the same in each test. The products obtained are given in Table 19.

TABLE 19

Conditioning Temperature

| Test No. | Temp °C | Wt % | CaF <sub>2</sub> % | Recovery % |
|----------|---------|------|--------------------|------------|
| 29-D     | 70      | 10.5 | 90.80              | 59.0       |
| 28-D     | 80      | 14.7 | 94.84              | 83.9       |
| 27-D     | 90      | 13.6 | 98.53              | 77.4       |
| 22-D     | 96+     | 15.7 | 96.71              | 88.2       |

These results suggest that the pulp should be raised to at least 90° C, and preferably higher.

9) Summary

The tests performed using the hot conditioning technique on the mixed feed from India indicate that:

- 1) a feed of at least 85 % minus 200 mesh is necessary,
- 2) quebracho improves both recovery and grade, and when added after hot conditioning produces better cleaning action,
- 3) good recovery and grade may be obtained with a number of fatty acid promoters of which Distilled Oleic Acid is the best,
- 4) rougher density at above 30% solids may be used, but cleaning is most effective at 5 or 6% solids.
- 5) low flotation temperatures, 8 to 10° C, especially in the cleaning stages, give better cleaning action, and
- 6) hot conditioning should be at 90° C or higher.

C: Combined Normal and Hot Conditioning

Because pulp heating is costly, test work aimed at reducing the bulk to be heated was performed. In this work the rougher step was with normal conditioning and the heating step was applied to the smaller bulks of the rougher float.

1) Hot Conditioning after Initial Flotation

The group of 5 tests shown in Table 20 compares 4 attempts at hot conditioning after normal roughing to a test, 3-B, on the pre-rougher hot conditioning schedule. The test conditions are generally similar throughout; exceptions are indicated in the Table.

TABLE 20

Hot Conditioning Prior to Cleaning

| Test No.                                | 3-B                     | 9-C                     | 13-C                    | 14-C                    | 10-C                    |
|---|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|
| Hot Cond                                | Before R                | Before Cl 1             | Before Cl 1             | Before Cl 1             | Before Cl 2             |
| Soda Ash<br>(lb/ton)                    | 4.0                     | 4.0                     | 4.5*                    | 8.0*                    | 4.0                     |
| Fraction                                | Dist CaF <sub>2</sub> % | Dist CaF <sub>2</sub> % | Dist CaF <sub>2</sub> % | Dist CaF <sub>2</sub> % | Dist CaF <sub>2</sub> % |
| Conc<br>(% CaF <sub>2</sub> in<br>Conc) | 82.6<br>(96.37)         | 93.2<br>(90.90)         | 94.3<br>(78.60)         | 92.0<br>(85.50)         | 87.2<br>(87.40)         |
| Cl 5 Tails                              | 1.9                     | 0.9                     | 0.5                     | 0.9                     | 0.5                     |
| Cl 4 "                                  | 2.4                     | 1.1                     | 0.5                     | 0.7                     | 0.4                     |
| Cl 3 "                                  | 2.2                     | 0.7                     | 0.4                     | 0.7                     | 0.3                     |
| Cl 2 "                                  | 3.3                     | 0.5                     | 0.4                     | 0.6                     | 0.2                     |
| Cl 1 "                                  | 5.4                     | 0.5                     | 0.5                     | 0.6                     | 8.6                     |
| Rougher "                               | 2.2                     | 3.1                     | 3.4                     | 4.5                     | 2.8                     |

\*Additional soda ash added prior to hot conditioning.

In the tests where hot conditioning took place after the initial flotation, small losses to cleaner tails indicate loss of cleaning action, resulting in high recoveries at reduced grades.

2) Hot Conditioning of Rougher Float After Re grind

The lack of cleaning action in tests reported in Table 20 suggested that the initial promoter coating from normal-conditioned roughing would have to be dislodged prior to hot conditioning. To verify this, two trials were made with a re grind step following the rougher flotation and prior to the hot conditioning. The results are recorded in Table 21 in comparison with test 22-D, representative of the pre-rougher hot conditioning schedule. The differences in test conditions are indicated in the Table.

TABLE 21

Effect of Regrind Step

| Test No.                                    | 22-D                    | 37-E                    | 38-E                    |
|---|-------------------------|-------------------------|-------------------------|
| Initial Fineness<br>(% -200 m)              | 87.1                    | 70.0                    | 87.1                    |
| Regrind<br>Quebracho (lb/ton)               | 0.5                     | 85.0% -200 m<br>0.5     | 90.0% -325 m<br>0.125   |
| " (added)                                   | After Hot C             | Before<br>Rougher       | After Hot C             |
| Fraction                                    | Dist CaF <sub>2</sub> % | Dist CaF <sub>2</sub> % | Dist CaF <sub>2</sub> % |
| Concentrate<br>(% CaF <sub>2</sub> in Conc) | 88.2<br>(96.71)         | 59.9<br>(96.71)         | 91.4<br>(95.31)         |
| C1 6 Tails                                  | 2.2                     | 7.7                     | 1.1                     |
| C1 5 "                                      | 0.9                     | 4.4                     | 0.8                     |
| C1 4 "                                      | 0.8                     | 4.7                     | 0.8                     |
| C1 3 "                                      | 1.1                     | 3.8                     | 0.9                     |
| C1 2 "                                      | 1.4                     | 3.6                     | 1.2                     |
| C1 1 "                                      | 2.4                     | 1.5                     | 1.7                     |
| Rougher "                                   | 3.0                     | 14.4                    | 2.1                     |

The results of these tests show that the regrind step clears up the difficulty of cleaning following hot conditioning of the rougher float. They also indicate that the rougher feed must be about 85% minus 200 mesh.

3) The Effect of Quebracho

Test 38-E suggests that roughing without quebracho but adding a small amount after hot conditioning might produce satisfactory results. To verify this, a test series was performed in which the amount of this reagent, added after the hot conditioning step, was varied. For these tests the general procedure used for 38-E was employed except that the rougher float was made after a bulk addition of promoter rather than step addition. A rougher flotation time equal to that for step addition was used. All conditions for the tests in Table 22 are similar except for the quantity of quebracho.

TABLE 22

Quebracho Requirement in Re grind System

| Test No.                                   | 40-E  | 41-E  | 42-E  |
|--|-------|-------|-------|
| Quebracho (lb/ton)                         | 0.125 | 0.250 | 0.50  |
| Recovery and Grade After 6 Cleaning Stages |       |       |       |
| Recovery %                                 | 90.7  | 89.8  | 89.9  |
| % CaF <sub>2</sub>                         | 94.78 | 95.55 | 97.85 |
| Recovery and Grade After 8 Cleaning Stages |       |       |       |
| Recovery %                                 | 86.7  | 85.6  | 86.4  |
| % CaF <sub>2</sub>                         | 95.58 | 96.24 | 98.10 |

These tests make it clear that a quebracho addition of about 0.5 lb/ton is required for the best grade control.

4) Summary

Hot conditioning of the rougher float will not yield good results unless it is reground prior to the heating step. To obtain a high grade and recovery, about 0.5 lb of quebracho per ton must be added prior to cleaner flotation.

DISCUSSION

The evolution of a highly effective system for the beneficiation of comparatively low grade fluorite samples from India has been traced and summarized. The various facets involved appear to be clearly defined by the tests reported, and require little in the way of further elucidation. It may however, be of value to draw the basic points together and add one or two additional observations.

The mixed feed (containing 17.7% CaF<sub>2</sub>) may be beneficiated to acid grade with a recovery of above 90% by first grinding to at least 85% minus 200 mesh, conditioning with 4.0 lb of soda ash per ton and 1.0 lb of oleic acid per ton, floating the fluorite in a rougher operation, regrinding to about 90% minus 325 mesh, heating to above 90° C, adding 0.5 lb of quebracho per ton and passing through 3 or 4 cleaner stages. The initial conditioning and the rougher flotation may be conducted at 30% solids or more and preferably

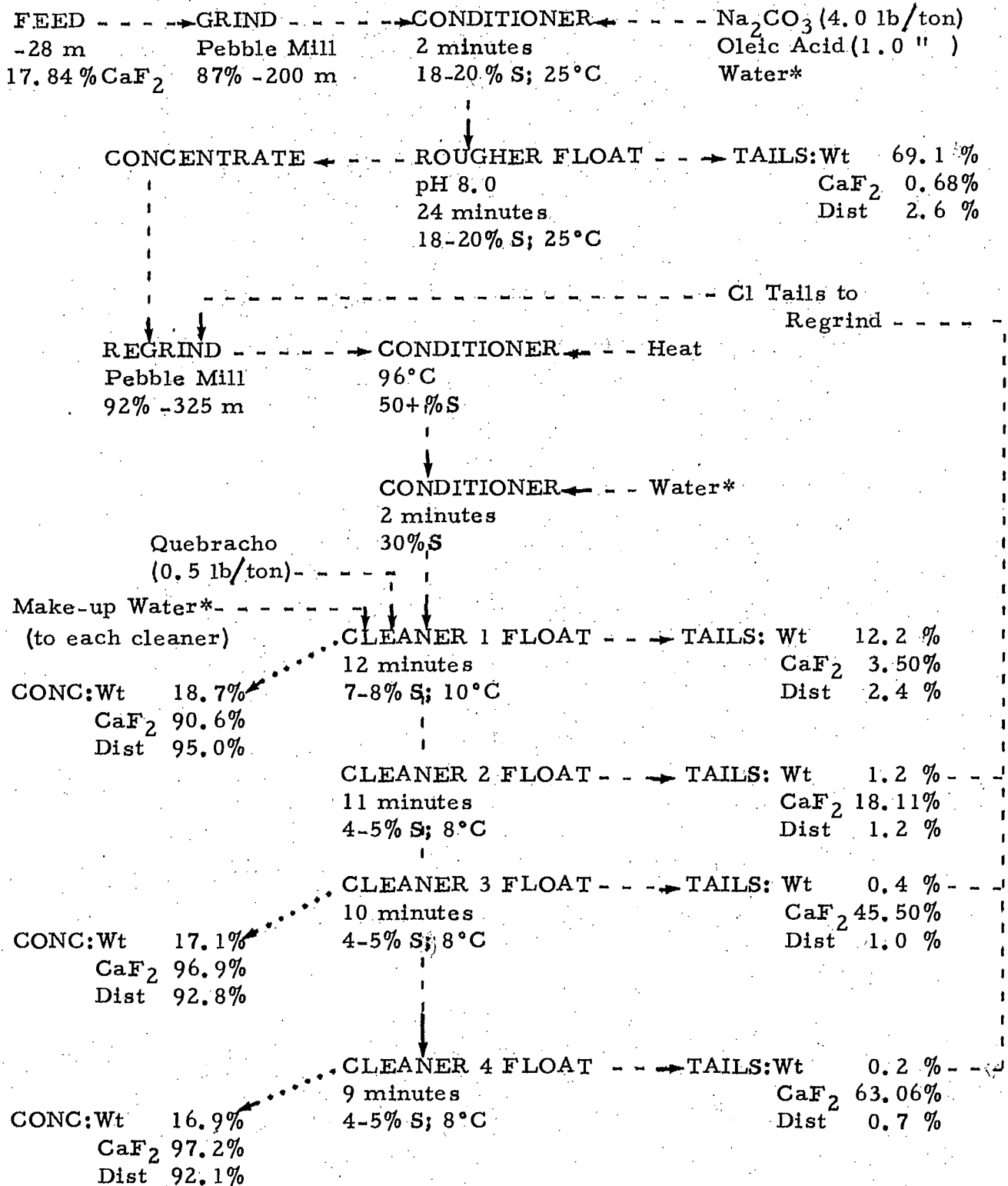
not above 25° C. Regrinding and hot conditioning may be performed at high density (at least 50% solids) with possibly some leeway on the conditioning temperature. Although the test work suggests that the closer to boiling the pulp is brought, the better the results, it might be possible in plant operation to use about 85° C and a longer exposure time than employed in the bench scale testing. There might have to be a compromise between the cost of heating and the value of the additional recovery derived. Quebracho should be added after the hot conditioning step, and prior to the cleaner flotation. The various cleaner stages should be operated at as low a density as practical (below 10 and preferably near 5% solids), and also as low a temperature as practical (preferably below 10° C). To produce metallurgical grade one cleaning stage could be used (about 95% recovery of 90% CaF<sub>2</sub> would be realized). To produce acid grade, 3 or 4 cleanings would be necessary. It is probable that, with the exception of Cleaner 1 tails, all cleaner tails could profitably be recirculated to the regrind mill to enhance recovery.

A suggested flowsheet, based on Test 42-E, as an example of this system, is given in Table 23.



TABLE 23

Suggested Flowsheet: Test 42-E



\* < 60 p.p.m. Hard

Cleaning without the hot conditioning step -- normal conditioning -- offers no real comparison in recovery and grade. There is therefore, no need for further elaboration.

It should be noted that, although metallurgical grade could be obtained by sink-float processing of the high grade material this offers little attraction either in recovery or grade in comparison to the high yield by flotation of the mixed grade.

A feature of the test work is the discovery that adding quebracho after the heating step enhances the cleaning operation. This is contrary to the usual practice where quebracho is added with the promoting agent. It is also of interest that although quebracho is normally employed as a depressant for carbonates, in this system it provides grade control, the amount used being somewhat critical.

Water plays a considerable role in the success of the flotation operation. An analysis of the water used throughout the test work is provided as Table 24. This is considered to be a comparatively soft water, with a normal hardness of less than 60 p. p. m. During the course of the test work it was being received through the city mains at from 6 to 8° C.

The tests reported in Table 16 suggest that any good grade of oleic acid would be a satisfactory promoter. The "old" Harfat was obtained approximately 2 years prior to the tests; the "new" Harfat was obtained specifically for this work.

TABLE 24

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

MINES BRANCH  
MINERAL PROCESSING DIVISION

INDUSTRIAL WATERS SECTION  
40 Lydia Street, Ottawa, Ont.

ANALYSIS OF WATER SAMPLE(S)  
(In parts per million)

|  |                              |
|--|------------------------------|
| Location   | Ottawa, Ontario              |
| Source of water  | Ottawa River                 |
| Sampling point   | Fleet Street pumping station |
| Reference  |                              |
| Laboratory number  | 7719                         |
| Date of sampling   | 2-5-68                       |
| Storage period (days)  | 28150                        |
| Temp. at sampling (°C)                                       | 10.0                         |
| Temp. at testing (°C)  | 24.3                         |
| Appearance, odour, etc.                                      |                              |
| Organic matter:  |                              |
| Oxygen consumed (KMnO <sub>4</sub> )                         |                              |
| Chem. oxygen demand (C.O.D.)                                 |                              |
| Ultra violet absorption ( mu)                                |                              |
| Carbon dioxide (CO <sub>2</sub> ), calculated                | 1.2                          |
| pH   | 7.6                          |
| Colour (Hazen units)   | 10                           |
| Turbidity (Units)  | 0                            |
| Alkalinity as (-Phenolphthalein<br>CaCO <sub>3</sub> (-Total | 0.0<br>28.9                  |
| Susp. matter, dried at 105°C                                 |                              |
| " " ignited at 550°C   |                              |
| Res. on evap., dried at 105°C                                | 92.8                         |
| Loss on ignition at 550°C                                    | 17.6                         |
| Sp. conductance, micromhos at 25°C                           | 125.9                        |
| Hardness as (Total   | 51.8                         |
| CaCO <sub>3</sub> (Non-carbonate                             | 28.9                         |
| Calcium (Ca)   | 16.1                         |
| Magnesium (Mg)   | 2.8                          |
| Iron (Fe) Total  | Trace                        |
| Dissolved  | 0.0                          |
| Aluminum (Al)  | 0.07                         |
| Manganese (Mn) Total   | 0.01                         |
| Dissolved  | 0.01                         |
| Copper (Cu)  | Trace                        |
| Zinc (Zn)  | 0.0                          |
| Sodium (Na)  | 1.6                          |
| Potassium (K)  | 0.8                          |
| Ammonia (NH <sub>3</sub> )                                   | 0.1                          |
| Carbonate (CO <sub>3</sub> )                                 | 0.0                          |
| Bicarbonate (HCO <sub>3</sub> )                              | 27.9                         |
| Sulphate (SO <sub>4</sub> )                                  | 28.6                         |
| Chloride (Cl)  | 2.6                          |
| Fluoride (F)   | 0.15                         |
| Phosphate (PO <sub>4</sub> ) Total                           | < 0.1                        |
| Dissolved  | < 0.1                        |
| Nitrate (NO <sub>3</sub> )                                   | 0.7                          |
| Silica (SiO <sub>2</sub> )                                   | 7.1                          |
| Sum of constituents  | 74.3                         |
| % Sodium   | 6.1                          |
| Saturation index at test temperature                         | -1.2                         |
| Stability index at test temperature                          | 10.0                         |
| Sodium Absorption Ratio (SAR)                                | 0.007                        |

..... Water treated with alum, chlorine and lime.

## CONCLUSIONS

- 1) Fluorite samples from India, assaying 17.7%  $\text{CaF}_2$ , can be beneficiated by normal flotation, followed by regrinding, hot conditioning and cleaning to an acid grade product of 97.2%  $\text{CaF}_2$  with a recovery of 92.1% after 4 cleaning stages, or 98.1%  $\text{CaF}_2$  with a recovery of 86.4% after 8 cleaning stages.
- 2) The same circuit, with only one cleaning stage, will produce a metallurgical grade product of 90%  $\text{CaF}_2$  with a recovery of 95%.
- 3) An initial grind of at least 85% minus 200 mesh, and a regrind to approximately 90% minus 325 mesh, are required for satisfactory results. The regrind step, following normal conditioning, is essential to an adequate cleaning operation after hot conditioning.
- 4) Reagents required are 4.0 lb of sodium carbonate per ton, 1.0 lb of oleic acid per ton and 0.5 lb of quebracho per ton.
- 5) The quantity (0.5 lb/ton) and point of addition (after hot conditioning) of the quebracho are critical, but contribute a great deal to the success of the system.
- 6) A density of above 30% solids may be used for the initial conditioning and flotation, with a temperature preferably below 25° C.
- 7) Regrinding and hot conditioning may be performed at 50% solids or higher.
- 8) Hot conditioning should be above 90° C.
- 9) Cleaning operations produce the best results when performed below 10% solids and below 10° C.
- 10) It is possible to obtain metallurgical grade fluorite (86%  $\text{CaF}_2$ ) with a recovery of 59% from the high grade (24%  $\text{CaF}_2$ ) material.

APPENDIX

Record of Tests

| Test No.             | 1     |                    |        | 2     |                    |        |
|----------------------|-------|--------------------|--------|-------|--------------------|--------|
| Conditions:          |       |                    |        |       |                    |        |
| Fineness (% -200 m)  | 69.8  |                    |        | 87.1  |                    |        |
| Soda Ash (lb/ton)    | 4.0   |                    |        | 4.0   |                    |        |
| Oleic Acid "         | 1.0*  |                    |        | 1.0*  |                    |        |
| Hot Condition Temp   | 96° C |                    |        | 96° C |                    |        |
| Quebracho (lb/ton)   | 0.5   |                    |        | 0.5   |                    |        |
| Condition Time (min) | 2     |                    |        | 2     |                    |        |
| Pulp: pH             | 8.0   |                    |        | 8.0   |                    |        |
| % Solids             | 18.0  |                    |        | 18.0  |                    |        |
| Results:             | Wt %  | CaF <sub>2</sub> % | Dist % | Wt %  | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 18.9  | 92.53              | 81.3   | 17.1  | 97.36              | 75.5   |
| Cleaner 5 Tails      |       |                    |        | 0.6   | 83.52              | 2.3    |
| " 4 "                |       |                    |        | 0.7   | 76.22              | 2.4    |
| " 3 "                | 1.3   | 55.46              | 3.3    | 0.9   | 64.27              | 2.6    |
| " 2 "                | 2.4   | 41.97              | 4.7    | 1.5   | 44.90              | 3.1    |
| " 1 "                | 7.5   | 19.31              | 6.7    | 8.3   | 26.70              | 10.0   |
| Rougher "            | 69.9  | 1.22               | 4.0    | 70.9  | 1.29               | 4.1    |
| Feed (calc'd)        | 100.0 | 21.53              | 100.0  | 100.0 | 22.10              | 100.0  |

\*Harfat 231 (old).

| Test No.             | 3-B   |                    |        | 4-B   |                    |        |
|----------------------|-------|--------------------|--------|-------|--------------------|--------|
| Conditions:          |       |                    |        |       |                    |        |
| Fineness (% -200 m)  | 87.1  |                    |        | 69.8  |                    |        |
| Soda Ash (lb/ton)    | 4.0   |                    |        | 4.0   |                    |        |
| Oleic Acid "         | 1.0*  |                    |        | 1.0*  |                    |        |
| Hot Condition Temp   | 96° C |                    |        | 96° C |                    |        |
| Quebracho (lb/ton)   | 0.5   |                    |        | 0.5   |                    |        |
| Condition Time (min) | 2     |                    |        | 2     |                    |        |
| Pulp: pH             | 8.0   |                    |        | 8.0   |                    |        |
| % Solids             | 18.0  |                    |        | 18.0  |                    |        |
| Results:             | Wt %  | CaF <sub>2</sub> % | Dist % | Wt %  | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 15.5  | 96.37              | 82.6   | 13.4  | 95.83              | 70.4   |
| Cleaner 5 Tails      | 0.5   | 69.18              | 1.9    | 1.1   | 79.70              | 4.8    |
| " 4 "                | 0.7   | 61.34              | 2.4    | 1.7   | 76.02              | 7.1    |
| " 3 "                | 0.9   | 45.84              | 2.2    | 1.9   | 60.00              | 6.2    |
| " 2 "                | 1.9   | 31.72              | 3.3    | 2.3   | 34.15              | 4.3    |
| " 1 "                | 8.3   | 11.84              | 5.4    | 7.1   | 10.22              | 4.0    |
| Rougher Tails        | 72.2  | 0.54               | 2.2    | 72.5  | 0.81               | 3.2    |
| Feed (calc'd)        | 100.0 | 18.13              | 100.0  | 100.0 | 18.28              | 100.0  |

\*Harfat 231 (old)

| Test No.             | 6-B           |                    |        | 7-B           |                  |        |
|----------------------|---------------|--------------------|--------|---------------|------------------|--------|
| <b>Conditions:</b>   |               |                    |        |               |                  |        |
| Fineness (% -200 m)  | 87.1          |                    |        | 87.1          |                  |        |
| Soda Ash (lb/ton)    | 4.0           |                    |        | 4.0           |                  |        |
| Quebracho "          | 0.5           |                    |        | 0.62**        |                  |        |
| Oleic Acid "         | 1.0*          |                    |        | 1.0*          |                  |        |
| Steps                | 7             |                    |        | 7             |                  |        |
| Condition Time (min) | 2 (each step) |                    |        | 2 (each step) |                  |        |
| Pulp: pH             | 8.0           |                    |        | 8.0           |                  |        |
| % Solids             | 18.0          |                    |        | 18.0          |                  |        |
| <b>Results:</b>      |               |                    |        |               |                  |        |
|                      | Wt %          | CaF <sub>2</sub> % | Dist % | Wt %          | CaF <sub>2</sub> | Dist % |
| Concentrate          | 16.3          | 89.18              | 80.5   | 3.4           | 98.13            | 18.2   |
| Cleaner 10 Tails     | 0.2           | 23.08              | 0.3    |               |                  |        |
| " 9 "                | 0.1           | 28.01              | 0.2    |               |                  |        |
| " 8 "                | 0.2           | 22.94              | 0.3    |               |                  |        |
| " 7 "                | 0.2           | 23.88              | 0.3    | 3.0           | 95.71            | 15.7   |
| " 6 "                | 0.3           | 19.35              | 0.3    | 3.1           | 90.98            | 15.4   |
| " 5 "                | 0.4           | 21.75              | 0.5    | 10.9          | 71.79            | 42.7** |
| " 4 "                | 0.5           | 20.79              | 0.6    | 0.7           | 15.14            | 0.6    |
| " 3 "                | 0.9           | 23.82              | 1.2    | 1.2           | 12.44            | 0.8    |
| " 2 "                | 2.2           | 21.68              | 2.6    | 2.2           | 11.18            | 1.3    |
| " 1 "                | 9.1           | 22.03              | 11.1   | 7.3           | 7.93             | 3.1    |
| Rougher Tails        | 69.6          | 0.54               | 2.1    | 68.2          | 0.59             | 2.2    |
| Feed (calc'd)        | 100.0         | 18.10              | 100.0  | 100.0         | 18.36            | 100.0  |

\*Harfat 231 (old)

\*\*An additional 0.12 lb quebracho per ton before cleaner 5

| Test No.             | 8-B           |                    |        | 9-C           |                    |        |
|----------------------|---------------|--------------------|--------|---------------|--------------------|--------|
| <b>Conditions:</b>   |               |                    |        |               |                    |        |
| Fineness (% -200 m)  | 87.1          |                    |        | 87.1          |                    |        |
| Soda Ash (lb/ton)    | 4.0           |                    |        | 4.0           |                    |        |
| Quebracho "          | 0.5           |                    |        | 0.5           |                    |        |
| Oleic Acid "         | 1.0*          |                    |        | 1.0*          |                    |        |
| Steps                | 7             |                    |        | 7             |                    |        |
| Condition Time (min) | 2 (each step) |                    |        | 2 (each step) |                    |        |
| Pulp: pH             | 8.0           |                    |        | 8.0           |                    |        |
| % Solids             | 18.0          |                    |        | 18.0          |                    |        |
| <b>Results:</b>      |               |                    |        |               |                    |        |
|                      | Wt %          | CaF <sub>2</sub> % | Dist % | Wt %          | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 11.8          | 93.63              | 61.0   | 17.3          | 90.90              | 93.2   |
| Cleaner 10 Tails     | 0.7           | 69.64              | 2.7    |               |                    |        |
| " 9 "                | 0.8           | 68.47              | 3.0    |               |                    |        |
| " 8 "                | 0.9           | 65.32              | 3.2    |               |                    |        |
| " 7 "                | 0.8           | 57.18              | 2.5    |               |                    |        |
| " 6 "                | 0.9           | 52.57              | 2.6    |               |                    |        |
| " 5 "                | 1.0           | 47.73              | 2.6    | 0.6           | 23.98              | 0.9    |
| " 4 "                | 1.1           | 41.06              | 2.5    | 1.0           | 17.88              | 1.1    |
| " 3 "                | 1.6           | 36.68              | 3.2    | 1.3           | 9.30               | 0.7    |
| " 2 "                | 2.7           | 30.75              | 4.6    | 2.1           | 4.25               | 0.5    |
| " 1 "                | 8.7           | 20.37              | 9.2    | 6.1           | 1.48               | 0.5    |
| Rougher Tails        | 69.0          | 0.77               | 2.9    | 71.6          | 0.73               | 3.1**  |
| Feed (calc'd)        | 100.0         | 18.14              | 100.0  | 100.0         | 16.83              | 100.0  |

\*Harfat 231 (old)

\*\*Rougher, normal conditioning. Hot conditioning before Cleaner 1.

| Test No.             | 10-C          |                    |        | 13-C             |                    |                   |
|----------------------|---------------|--------------------|--------|------------------|--------------------|-------------------|
| <b>Conditions:</b>   |               |                    |        |                  |                    |                   |
| Fineness (% -200 m)  | 87.1          |                    |        | 87.1             |                    |                   |
| Soda Ash (lb/ton)    | 4.0           |                    |        | 4.5 <sup>x</sup> |                    |                   |
| Quebracho "          | 0.5           |                    |        | 0.5              |                    |                   |
| Oleic Acid "         | 1.0*          |                    |        | 1.0*             |                    |                   |
| Steps                | 7             |                    |        | 7                |                    |                   |
| Condition Time (min) | 2 (each step) |                    |        | 2 (each step)    |                    |                   |
| Pulp: pH             | 8.0           |                    |        | 8.0              |                    |                   |
| % Solids             | 18.0          |                    |        | 18.0             |                    |                   |
| <b>Results:</b>      |               |                    |        |                  |                    |                   |
|                      | Wt %          | CaF <sub>2</sub> % | Dist % | Wt %             | CaF <sub>2</sub> % | Dist %            |
| Concentrate          | 16.7          | 87.40              | 87.2   | 19.7             | 78.60              | 94.3              |
| Cleaner 5 Tails      | 0.6           | 12.74              | 0.5    | 0.6              | 13.11              | 0.5               |
| " 4 "                | 0.8           | 8.66               | 0.4    | 0.7              | 12.23              | 0.5               |
| " 3 "                | 0.9           | 4.95               | 0.3    | 0.7              | 8.89               | 0.4               |
| " 2 "                | 1.5           | 2.48               | 0.2    | 1.7              | 3.51               | 0.4               |
| " 1 "                | 7.9           | 18.08              | 8.6**  | 5.4              | 1.39               | 0.5               |
| Rougher Tails        | 71.6          | 0.66               | 2.8    | 71.2             | 0.77               | 3.4 <sup>xx</sup> |
| Feed (calc'd)        | 100.0         | 16.71              | 100.0  | 100.0            | 16.33              | 100.0             |

\*Harfat 231 (old)

\*\*Rougher and cleaner 1, normal conditioning, hot conditioning before cleaner 2.

<sup>x</sup>An additional 0.5 lb of soda ash per ton added before hot conditioning (pH 8).

<sup>xx</sup>Rougher, normal conditioning, hot conditioning before cleaner 1.

| Test No.             | 14-C          |                    |                  | 16-C                |                    |        |
|----------------------|---------------|--------------------|------------------|---------------------|--------------------|--------|
| <b>Conditions:</b>   |               |                    |                  |                     |                    |        |
| Fineness (% -200 m)  | 87.1          |                    |                  | 87.1                |                    |        |
| Soda Ash (lb/ton)    | 8.0**         |                    |                  | 4.0                 |                    |        |
| Quebracho "          | 0.5           |                    |                  | 0.518 <sup>xx</sup> |                    |        |
| Oleic Acid "         | 1.0*          |                    |                  | 1.0*                |                    |        |
| Steps                | 7             |                    |                  | 7                   |                    |        |
| Condition Time (min) | 2 (each step) |                    |                  | 2 (each step)       |                    |        |
| Pulp: pH             | 8.0           |                    |                  | 8.0                 |                    |        |
| % Solids             | 18.0          |                    |                  | 18.0                |                    |        |
| <b>Results:</b>      |               |                    |                  |                     |                    |        |
|                      | Wt %          | CaF <sub>2</sub> % | Dist %           | Wt %                | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 17.9          | 85.50              | 92.0             | 7.2                 | 95.55              | 40.7   |
| Cleaner 7 Tails      |               |                    |                  | 5.1                 | 91.26              | 27.4   |
| " 6 "                |               |                    |                  | 2.1                 | 73.71              | 9.2    |
| " 5 "                | 0.9           | 16.15              | 0.9              | 1.6                 | 49.95              | 4.7    |
| " 4 "                | 0.9           | 13.79              | 0.7              | 1.5                 | 32.01              | 2.8    |
| " 3 "                | 1.0           | 12.00              | 0.7              | 1.8                 | 22.80              | 2.4    |
| " 2 "                | 1.6           | 6.28               | 0.6              | 2.4                 | 17.52              | 2.5    |
| " 1 "                | 6.3           | 1.52               | 0.6              | 9.3                 | 13.76              | 7.6    |
| Rougher Tails        | 71.4          | 1.06               | 4.5 <sup>x</sup> | 69.0                | 0.66               | 2.7    |
| Feed (calc'd)        | 100.0         | 16.63              | 100.0            | 100.0               | 16.93              | 100.0  |

\*Harfat 231 (old)

\*\*An additional 4.0 lb of soda ash per ton added before hot conditioning (pH 9).

<sup>x</sup>Rougher, normal conditioning. Hot conditioning before cleaner 1.

<sup>xx</sup>Composed of 0.5 lb/ton before rougher and 0.003 lb/ton before cleaner 2, and each subsequent cleaner.

| Test No.             | 11-C          |                    |        | 17-C               |                    |        |
|----------------------|---------------|--------------------|--------|--------------------|--------------------|--------|
| <b>Conditions:</b>   |               |                    |        |                    |                    |        |
| Fineness (% -200 m)  | 87.1          |                    |        | 87.1               |                    |        |
| Soda Ash (lb/ton)    | 4.0           |                    |        | 4.0                |                    |        |
| Quebracho "          | 0.5165**      |                    |        | 0.518 <sup>x</sup> |                    |        |
| Oleic Acid "         | 1.0*          |                    |        | 1.0*               |                    |        |
| Steps                | 7             |                    |        | 7                  |                    |        |
| Condition Time (min) | 2 (each step) |                    |        | 2 (each step)      |                    |        |
| Pulp: pH             | 8.0           |                    |        | 8.0                |                    |        |
| % Solids             | 18.0          |                    |        | 18.0               |                    |        |
| <b>Results:</b>      |               |                    |        |                    |                    |        |
|                      | Wt %          | CaF <sub>2</sub> % | Dist % | Wt %               | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 13.4          | 93.83              | 75.4   | 2.4                | 96.86              | 13.4   |
| Cleaner 10 Tails     | 1.1           | 65.28              | 4.3    |                    |                    |        |
| " 9 "                | 0.9           | 53.71              | 2.9    |                    |                    |        |
| " 8 "                | 0.3           | 33.06              | 0.6    |                    |                    |        |
| " 7 "                | 0.3           | 28.56              | 0.5    |                    |                    |        |
| " 6 "                | 0.4           | 24.61              | 0.6    |                    |                    |        |
| " 5 "                | 0.5           | 20.27              | 0.6    | 8.6                | 91.76              | 45.8   |
| " 4 "                | 0.8           | 18.11              | 0.9    | 3.9                | 71.03              | 16.0   |
| " 3 "                | 1.0           | 16.42              | 1.0    | 2.7                | 42.02              | 6.6    |
| " 2 "                | 1.8           | 13.44              | 1.5    | 3.2                | 30.44              | 5.6    |
| " 1 "                | 8.7           | 18.46              | 9.6    | 9.1                | 18.27              | 9.6    |
| Rougher Tails        | 70.8          | 0.50               | 2.1    | 70.1               | 0.74               | 3.0    |
| Feed (calc'd)        | 100.0         | 16.70              | 100.0  | 100.0              | 17.27              | 100.0  |

\*Harfat 231 (old)

\*\*Quebracho schedule: Rougher, 0.5 lb/ton; before C1 2, 0.0015 lb/ton; before C1 4, 0.0015 lb/ton; before C1 8, 0.0030 lb/ton; before C1 9, 0.0045 lb/ton; before C1 10, 0.0060 lb/ton.

<sup>x</sup>Composed of 0.5 lb/ton before rougher and 0.0045 lb/ton before C1 2 and each subsequent cleaner.

| Test No.             | 18-C  |                    |        | 19-C  |                    |        |
|----------------------|-------|--------------------|--------|-------|--------------------|--------|
| <b>Conditions:</b>   |       |                    |        |       |                    |        |
| Fineness (% -200 m)  | 87.1  |                    |        | 87.1  |                    |        |
| Soda Ash (lb/ton)    | 4.0   |                    |        | 4.0   |                    |        |
| Oleic Acid "         | 1.0*  |                    |        | 1.0*  |                    |        |
| Hot Condition Temp   | 96°C  |                    |        | 96°C  |                    |        |
| Quebracho (lb/ton)   | 0.5   |                    |        | 0.5** |                    |        |
| Condition Time (min) | 2     |                    |        | -     |                    |        |
| Pulp: pH             | 8.0   |                    |        | 8.0   |                    |        |
| % Solids             | 18.0  |                    |        | 18.0  |                    |        |
| <b>Results:</b>      |       |                    |        |       |                    |        |
|                      | Wt %  | CaF <sub>2</sub> % | Dist % | Wt %  | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 13.6  | 97.29              | 80.0   | 16.2  | 92.41              | 89.6   |
| Cleaner 6 Tails      | 0.7   | 89.60              | 3.8    | 1.0   | 51.59              | 3.1    |
| " 5 "                | 0.5   | 78.57              | 2.4    | 0.4   | 29.21              | 0.7    |
| " 4 "                | 0.6   | 70.01              | 2.5    | 0.5   | 22.90              | 0.7    |
| " 3 "                | 0.7   | 50.11              | 2.1    | 0.8   | 16.22              | 0.8    |
| " 2 "                | 1.4   | 27.18              | 2.3    | 1.5   | 10.58              | 1.0    |
| " 1 "                | 8.1   | 8.56               | 4.2    | 5.5   | 4.26               | 1.4    |
| Rougher Tails        | 74.4  | 0.60               | 2.7    | 74.1  | 0.60               | 2.7    |
| Feed (calc'd)        | 100.0 | 16.56              | 100.0  | 100.0 | 16.69              | 100.0  |

\*Harfat 231 (old)

\*\*Quebracho added before oleic acid, and before hot conditioning.



| Test No.             | 20-D     |                    |        | 25-D              |                    |        |
|----------------------|----------|--------------------|--------|-------------------|--------------------|--------|
| <b>Conditions:</b>   |          |                    |        |                   |                    |        |
| Fineness (% -200 m)  | 87.1     |                    |        | 87.1              |                    |        |
| Soda Ash (lb/ton)    | 4.0      |                    |        | 4.0               |                    |        |
| Quebracho "          | 0.5135** |                    |        | 0.5165**          |                    |        |
| Oleic Acid "         | 1.0*     |                    |        | 1.0*              |                    |        |
| Steps                | -        |                    |        | 7                 |                    |        |
| Condition Time (min) | 2        |                    |        | 2 (each step)     |                    |        |
| Pulp: pH             | 8.0      |                    |        | 8.0               |                    |        |
| % Solids             | 18.0     |                    |        | 18.0 <sup>x</sup> |                    |        |
| <b>Results:</b>      |          |                    |        |                   |                    |        |
|                      | Wt %     | CaF <sub>2</sub> % | Dist % | Wt %              | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 15.4     | 94.88              | 84.6   | 16.2              | 91.64              | 87.5   |
| Cleaner 12 Tails     |          |                    |        | 0.4               | 43.79              | 1.0    |
| " 11 "               |          |                    |        | 0.4               | 35.08              | 0.8    |
| " 10 "               | 0.4      | 51.23              | 1.2    | 0.4               | 25.61              | 0.6    |
| " 9 "                | 0.5      | 34.73              | 1.0    | 0.4               | 26.21              | 0.6    |
| " 8 "                | 0.6      | 26.11              | 0.9    | 0.5               | 24.44              | 0.7    |
| " 7 "                | 0.6      | 19.04              | 0.6    | 0.4               | 22.91              | 0.6    |
| " 6 "                | 0.5      | 17.89              | 0.5    | 0.4               | 20.47              | 0.5    |
| " 5 "                | 0.5      | 17.19              | 0.5    | 0.5               | 16.13              | 0.5    |
| " 4 "                | 0.6      | 16.65              | 0.6    | 0.8               | 15.79              | 0.7    |
| " 3 "                | 0.9      | 21.28              | 1.1    | 1.1               | 12.52              | 0.8    |
| " 2 "                | 1.6      | 14.54              | 1.3    | 2.4               | 8.54               | 1.2    |
| " 1 "                | 6.4      | 6.91               | 2.6    | 8.0               | 4.36               | 2.1    |
| Rougher Tails        | 72.0     | 1.20               | 5.1    | 68.1              | 0.60               | 2.4    |
| Feed (calc'd)        | 100.0    | 17.25              | 100.0  | 100.0             | 16.93              | 100.0  |

\*Harfat 231 (old) - bulk addition 20-D.

\*\*Quebracho Schedule: 0.5 lb/ton to rougher, 0.0015 lb/ton before cleaner 2 and each subsequent cleaner.

<sup>x</sup>Water at 25°C for all stages.

| Test No.             | 21-D  |                    |        | 22-D              |                    |        |
|----------------------|-------|--------------------|--------|-------------------|--------------------|--------|
| <b>Conditions:</b>   |       |                    |        |                   |                    |        |
| Fineness (% -200 m)  | 87.1  |                    |        | 87.1              |                    |        |
| Soda Ash (lb/ton)    | 4.0   |                    |        | 4.0               |                    |        |
| Oleic Acid "         | 1.0*  |                    |        | 1.0**             |                    |        |
| Hot Condition Temp   | 96°C  |                    |        | 96°C              |                    |        |
| Quebracho (lb/ton)   | 0.5   |                    |        | 0.5               |                    |        |
| Condition Time (min) | 2     |                    |        | 2                 |                    |        |
| Pulp                 | 8.0   |                    |        | 8.0               |                    |        |
| % Solids             | 18.0  |                    |        | 18.0 <sup>x</sup> |                    |        |
| <b>Results:</b>      |       |                    |        |                   |                    |        |
|                      | Wt %  | CaF <sub>2</sub> % | Dist % | Wt %              | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 13.7  | 95.29              | 76.2   | 15.7              | 96.71              | 88.2   |
| Cleaner 6 Tails      | 1.0   | 78.91              | 4.6    | 0.5               | 75.75              | 2.2    |
| " 5 "                | 0.7   | 66.95              | 2.7    | 0.3               | 52.36              | 0.9    |
| " 4 "                | 1.0   | 59.46              | 3.5    | 0.3               | 45.65              | 0.8    |
| " 3 "                | 1.2   | 48.49              | 3.4    | 0.6               | 32.99              | 1.1    |
| " 2 "                | 1.7   | 31.43              | 3.1    | 1.3               | 18.81              | 1.4    |
| " 1 "                | 6.7   | 9.56               | 3.7    | 6.9               | 5.92               | 2.4    |
| Rougher Tails        | 74.0  | 0.65               | 2.8    | 74.4              | 0.69               | 3.0    |
| Feed (calc'd)        | 100.0 | 17.19              | 100.0  | 100.0             | 17.24              | 100.0  |

\*Harfat 231 (new)

\*\*Distilled oleic acid.

<sup>x</sup>Density in cleaners: 6% solids in cleaner 1, decreasing 4% solids in cleaner 6.

| Test No.             | 23-D  |                    |        | 24-D  |                    |        |
|----------------------|-------|--------------------|--------|-------|--------------------|--------|
| <b>Conditions:</b>   |       |                    |        |       |                    |        |
| Fineness (% -200 m)  | 87.1  |                    |        | 87.1  |                    |        |
| Soda Ash (lb/ton)    | 4.0   |                    |        | 4.0   |                    |        |
| Oleic Acid "         | 1.0*  |                    |        | 1.0** |                    |        |
| Hot Condition Temp   | 96°C  |                    |        | 96°C  |                    |        |
| Quebracho (lb/ton)   | 0.5   |                    |        | 0.5   |                    |        |
| Condition Time (min) | 2     |                    |        | 2     |                    |        |
| Pulp: pH             | 8.0   |                    |        | 8.0   |                    |        |
| % Solids             | 18.0  |                    |        | 18.0  |                    |        |
| <b>Results:</b>      |       |                    |        |       |                    |        |
|                      | Wt %  | CaF <sub>2</sub> % | Dist % | Wt %  | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 15.7  | 95.42              | 87.6   | 14.5  | 97.20              | 81.8   |
| Cleaner 6 Tails      | 0.4   | 73.33              | 1.7    | 0.7   | 84.79              | 3.4    |
| " 5 "                | 0.3   | 56.72              | 1.0    | 0.5   | 70.35              | 2.0    |
| " 4 "                | 0.4   | 48.92              | 1.1    | 0.5   | 58.44              | 1.7    |
| " 3 "                | 0.5   | 34.92              | 1.0    | 0.7   | 42.72              | 1.7    |
| " 2 "                | 1.1   | 20.13              | 1.3    | 1.3   | 26.71              | 2.0    |
| " 1 "                | 6.5   | 6.55               | 2.5    | 6.9   | 8.96               | 3.6    |
| Rougher Tails        | 75.1  | 0.85               | 3.8    | 74.9  | 0.86               | 3.8    |
| Feed (calc'd)        | 100.0 | 17.10              | 100.0  | 100.0 | 17.24              | 100.0  |

\*Oleic Acid No. 4

\*\*Oleic Acid SS (undistilled)

| Test No.             | 26-D                |                    |        |
|----------------------|---------------------|--------------------|--------|
| <b>Conditions:</b>   |                     |                    |        |
| Fineness (% -200 m)  | 87.1                |                    |        |
| Soda Ash (lb/ton)    | 4.0                 |                    |        |
| Quebracho "          | 0.5165 <sup>x</sup> |                    |        |
| Oleic Acid "         | 1.0*                |                    |        |
| Steps                | 7                   |                    |        |
| Condition Time (min) | 2 (each step)       |                    |        |
| Pulp: pH             | 8.0                 |                    |        |
| % Solids             | 18.0**              |                    |        |
| <b>Results:</b>      |                     |                    |        |
|                      | Wt %                | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 11.4                | 97.00              | 64.4   |
| Cleaner 12 Tails     | 1.0                 | 90.18              | 5.3    |
| " 11 "               | 0.7                 | 85.09              | 3.5    |
| " 10 "               | 0.7                 | 78.89              | 3.2    |
| " 9 "                | 0.6                 | 65.92              | 2.3    |
| " 8 "                | 0.5                 | 49.09              | 1.4    |
| " 7 "                | 0.6                 | 37.17              | 1.3    |
| " 6 "                | 0.7                 | 29.43              | 1.2    |
| " 5 "                | 0.8                 | 22.16              | 1.0    |
| " 4 "                | 1.0                 | 19.58              | 1.1    |
| " 3 "                | 1.3                 | 16.96              | 1.3    |
| " 2 "                | 2.2                 | 13.99              | 1.8    |
| " 1 "                | 9.9                 | 16.08              | 9.3    |
| Rougher Tails        | 68.6                | 0.73               | 2.9    |
| Feed (calc'd)        | 100.0               | 17.16              | 100.0  |

\*Harfat 231 (old)

\*\*Water at 15°C for rougher, decreasing to 8°C for cleaners.

<sup>x</sup>Quebracho schedule: 0.5 lb/ton before rougher; 0.0015 lb/ton before cleaner 2, and each subsequent cleaner.

| Test No.             | 27-D  |                    |        | 28-D  |                    |        |
|----------------------|-------|--------------------|--------|-------|--------------------|--------|
| <b>Conditions:</b>   |       |                    |        |       |                    |        |
| Fineness (% -200 m)  | 87.1  |                    |        | 87.1  |                    |        |
| Soda Ash (lb/ton)    | 4.0   |                    |        | 4.0   |                    |        |
| Oleic Acid "         | 1.0*  |                    |        | 1.0*  |                    |        |
| Hot Condition Temp   | 90°C  |                    |        | 80°C  |                    |        |
| Quebracho (lb/ton)   | 0.5   |                    |        | 0.5   |                    |        |
| Condition Time (min) | 2     |                    |        | 2     |                    |        |
| Pulp: pH             | 8.0   |                    |        | 8.0   |                    |        |
| % Solids             | 18.0  |                    |        | 18.0  |                    |        |
| <b>Results:</b>      |       |                    |        |       |                    |        |
|                      | Wt %  | CaF <sub>2</sub> % | Dist % | Wt %  | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 13.6  | 98.53              | 77.4   | 14.7  | 94.84              | 83.9   |
| Cleaner 6 Tails      | 1.3   | 92.85              | 7.0    | 0.7   | 84.46              | 3.5    |
| " 5 "                | 0.6   | 80.50              | 2.8    | 0.5   | 70.71              | 2.1    |
| " 4 "                | 0.6   | 68.12              | 2.4    | 0.5   | 54.63              | 1.7    |
| " 3 "                | 0.7   | 46.48              | 1.9    | 0.6   | 36.66              | 1.3    |
| " 2 "                | 1.4   | 26.76              | 2.2    | 1.2   | 20.63              | 1.5    |
| " 1 "                | 6.7   | 7.49               | 2.9    | 6.9   | 6.88               | 2.9    |
| Rougher Tails        | 75.1  | 0.78               | 3.4    | 74.9  | 0.70               | 3.1    |
| Feed (calc'd)        | 100.0 | 17.29              | 100.0  | 100.0 | 16.67              | 100.0  |

\*Distilled oleic acid.

| Test No.             | 29-D  |                    |        | 30-D  |                    |        |
|----------------------|-------|--------------------|--------|-------|--------------------|--------|
| <b>Conditions:</b>   |       |                    |        |       |                    |        |
| Fineness (% -200 m)  | 87.1  |                    |        | 87.1  |                    |        |
| Soda Ash (lb/ton)    | 4.0   |                    |        | 4.0   |                    |        |
| Oleic Acid "         | 1.0*  |                    |        | 1.0*  |                    |        |
| Hot Condition Temp   | 70°C  |                    |        | 96°C  |                    |        |
| Quebracho (lb/ton)   | 0.5   |                    |        | -     |                    |        |
| Condition Time (min) | 2     |                    |        | -     |                    |        |
| Pulp: pH             | 8.0   |                    |        | 8.0   |                    |        |
| % Solids             | 18.0  |                    |        | 18.0  |                    |        |
| <b>Results:</b>      |       |                    |        |       |                    |        |
|                      | Wt %  | CaF <sub>2</sub> % | Dist % | Wt %  | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 10.5  | 90.80              | 56.8   | 15.3  | 93.53              | 87.1   |
| Cleaner 6 Tails      | 0.5   | 71.73              | 2.1    | 0.6   | 73.64              | 2.7    |
| " 5 "                | 0.6   | 68.21              | 2.4    | 0.4   | 52.29              | 1.3    |
| " 4 "                | 0.8   | 67.66              | 3.2    | 0.5   | 39.85              | 1.2    |
| " 3 "                | 1.2   | 63.23              | 4.5    | 0.8   | 27.97              | 1.3    |
| " 2 "                | 2.1   | 52.92              | 6.6    | 1.4   | 16.58              | 1.4    |
| " 1 "                | 10.9  | 33.24              | 21.6   | 6.1   | 5.40               | 2.0    |
| Rougher Tails        | 73.4  | 0.65               | 2.8    | 74.9  | 0.67               | 3.0    |
| Feed (calc'd)        | 100.0 | 17.82              | 100.0  | 100.0 | 16.44              | 100.0  |

\*Distilled oleic acid.

| Test No.             | 31-E  |                    |        | 34-E   |                    |        |
|----------------------|-------|--------------------|--------|--------|--------------------|--------|
| <b>Conditions:</b>   |       |                    |        |        |                    |        |
| Fineness (% -200 m)  | 87.1  |                    |        | 87.1   |                    |        |
| Soda Ash (lb/ton)    | 4.0   |                    |        | 4.0    |                    |        |
| Oleic Acid "         | 1.0*  |                    |        | 1.0*   |                    |        |
| Hot Condition Temp   | 96° C |                    |        | 96° C  |                    |        |
| Quebracho (lb/ton)   | -     |                    |        | 0.5    |                    |        |
| Condition Time (min) | -     |                    |        | 2      |                    |        |
| Pulp: pH             | 8.0   |                    |        | 8.0    |                    |        |
| % Solids             | 18.0  |                    |        | 18.0** |                    |        |
| <b>Results:</b>      |       |                    |        |        |                    |        |
|                      | Wt %  | CaF <sub>2</sub> % | Dist % | Wt %   | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 13.1  | 95.92              | 69.6   | 15.5   | 96.33              | 86.0   |
| Cleaner 6 Tails      | 1.4   | 87.21              | 6.8    | 0.5    | 81.28              | 2.3    |
| " 5 "                | 1.1   | 76.78              | 4.7    | 0.2    | 68.16              | 0.8    |
| " 4 "                | 1.2   | 64.17              | 4.3    | 0.4    | 61.79              | 1.4    |
| " 3 "                | 1.6   | 47.78              | 4.2    | 0.6    | 42.97              | 1.5    |
| " 2 "                | 2.6   | 29.40              | 4.2    | 1.5    | 23.73              | 2.0    |
| " 1 "                | 8.7   | 7.65               | 3.7    | 9.3    | 7.77               | 4.1    |
| Rougher Tails        | 70.3  | 0.64               | 2.5    | 72.0   | 0.46               | 1.9    |
| Feed (calc'd)        | 100.0 | 18.03              | 100.0  | 100.0  | 17.36              | 100.0  |

\*Harfat 231 (old)

\*\*Water at 20° C for rougher, decreasing to 9° C for cleaners.

| Test No.             | 35-E   |                    |        | 36-E               |                    |        |
|----------------------|--------|--------------------|--------|--------------------|--------------------|--------|
| <b>Conditions:</b>   |        |                    |        |                    |                    |        |
| Fineness (% -200 m)  | 87.1   |                    |        | 87.1               |                    |        |
| Soda Ash (lb/ton)    | 4.0    |                    |        | 4.0                |                    |        |
| Oleic Acid "         | 1.0*   |                    |        | 1.0 <sup>x</sup>   |                    |        |
| Hot Condition Temp   | 96° C  |                    |        | 96° C              |                    |        |
| Quebracho (lb/ton)   | 0.5    |                    |        | 0.5                |                    |        |
| Condition Time (min) | 2      |                    |        | 2                  |                    |        |
| Pulp: pH             | 8.0    |                    |        | 9.0                |                    |        |
| % Solids             | 18.0** |                    |        | 35.4 <sup>xx</sup> |                    |        |
| <b>Results:</b>      |        |                    |        |                    |                    |        |
|                      | Wt %   | CaF <sub>2</sub> % | Dist % | Wt %               | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 12.3   | 96.14              | 67.6   | 14.8               | 94.31              | 77.3   |
| Cleaner 8 Tails      | 0.9    | 93.88              | 4.8    |                    |                    |        |
| " 7 "                | 0.8    | 90.71              | 4.1    | 1.0                | 83.60              | 4.6    |
| " 6 "                | 0.6    | 85.35              | 2.9    | 0.8                | 74.39              | 3.3    |
| " 5 "                | 0.5    | 76.88              | 2.2    | 0.7                | 60.65              | 2.4    |
| " 4 "                | 0.7    | 69.50              | 2.8    | 0.9                | 50.23              | 2.5    |
| " 3 "                | 1.0    | 56.68              | 3.3    | 1.2                | 35.06              | 2.3    |
| " 2 "                | 2.1    | 35.75              | 4.3    | 2.6                | 18.91              | 3.3    |
| " 1 "                | 9.9    | 9.91               | 5.6    | 9.6                | 4.51               | 2.4    |
| Rougher Tails        | 71.2   | 0.58               | 2.4    | 68.4               | 0.50               | 1.9    |
| Feed (calc'd)        | 100.0  | 17.51              | 100.0  | 100.0              | 18.09              | 100.0  |

\*Harfat 231 (old)

\*\*Water to rougher at 32° C, to cleaners at 23 to 24° C

<sup>x</sup>Distilled oleic acid.

<sup>xx</sup>Rougher density 35.4 % Solids; C1 1 density 13.6 % Solids - decreasing to 8% Solids in 7th cleaner.

| Test No.             | 37-E          |                    |        | 38-E          |                    |        |
|----------------------|---------------|--------------------|--------|---------------|--------------------|--------|
| <b>Conditions:</b>   |               |                    |        |               |                    |        |
| Fineness (% -200 m)  | 69.8          |                    |        | 87.1          |                    |        |
| Soda Ash (lb/ton)    | 4.0           |                    |        | 4.0           |                    |        |
| Quebracho "          | 0.5           |                    |        | -             |                    |        |
| Oleic Acid "         | 1.0*          |                    |        | 1.0*          |                    |        |
| Steps                | 7             |                    |        | 7             |                    |        |
| Condition Time (min) | 2 (each step) |                    |        | 2 (each step) |                    |        |
| Pulp: pH             | 8.0           |                    |        | 8.0           |                    |        |
| % Solids             | 18.0          |                    |        | 18.0          |                    |        |
| Regrind Fineness     | 85.0% -200 m  |                    |        | 92.0% -325 m  |                    |        |
| Hot Condition Temp   | 96° C         |                    |        | 96° C         |                    |        |
| Quebracho (lb/ton)   | -             |                    |        | 0.125         |                    |        |
| Condition Time (min) | -             |                    |        | 2             |                    |        |
| <b>Results:</b>      |               |                    |        |               |                    |        |
|                      | Wt %          | CaF <sub>2</sub> % | Dist % | Wt %          | CaF <sub>2</sub> % | Dist % |
| Concentrate          | 10.7          | 96.71              | 59.9   | 17.4          | 95.31              | 91.4   |
| Cleaner 6 Tails      | 1.4           | 95.19              | 7.7    | 0.3           | 64.85              | 1.1    |
| " 5 "                | 0.8           | 94.08              | 4.4    | 0.3           | 47.21              | 0.8    |
| " 4 "                | 0.9           | 91.12              | 4.7    | 0.4           | 36.94              | 0.8    |
| " 3 "                | 0.8           | 82.93              | 3.8    | 0.7           | 25.07              | 0.9    |
| " 2 "                | 1.2           | 51.49              | 3.6    | 1.9           | 12.04              | 1.2    |
| " 1 "                | 4.2           | 5.94               | 1.5    | 12.0          | 2.67               | 1.7    |
| Rougher Tails        | 80.0          | 3.11               | 14.4   | 67.0          | 0.57               | 2.1    |
| Feed (calc'd)        | 100.0         | 17.31              | 100.0  | 100.0         | 18.19              | 100.0  |

\*Distilled oleic acid.

| Test No.                       | 40-E  |                    |        | 41-E  |                    |        |
|--------------------------------|-------|--------------------|--------|-------|--------------------|--------|
| <b>Conditions:</b>             |       |                    |        |       |                    |        |
| Fineness (% -200 m)            | 87.1  |                    |        | 87.1  |                    |        |
| Soda Ash (lb/ton)              | 4.0   |                    |        | 4.0   |                    |        |
| Oleic Acid "                   | 1.0*  |                    |        | 1.0*  |                    |        |
| Condition (min)                | 2     |                    |        | 2     |                    |        |
| Pulp: pH                       | 8.0   |                    |        | 8.0   |                    |        |
| % Solids                       | 18.0  |                    |        | 18.0  |                    |        |
| Regrind Fineness<br>(% -325 m) | 92.   |                    |        | 92.   |                    |        |
| Hot Condition Temp             | 96° C |                    |        | 96° C |                    |        |
| Quebracho (lb/ton)             | 0.125 |                    |        | 0.250 |                    |        |
| Condition Time (min)           | 2     |                    |        | 2     |                    |        |
| <b>Results:</b>                |       |                    |        |       |                    |        |
|                                | Wt %  | CaF <sub>2</sub> % | Dist % | Wt %  | CaF <sub>2</sub> % | Dist % |
| Concentrate                    | 16.0  | 95.58              | 86.7   | 15.8  | 96.24              | 85.6   |
| Cleaner 8 Tails                | 0.5   | 82.29              | 2.3    | 0.5   | 86.82              | 2.5    |
| " 7 "                          | 0.4   | 74.24              | 1.7    | 0.4   | 77.11              | 1.7    |
| " 6 "                          | 0.4   | 59.26              | 1.4    | 0.3   | 66.60              | 1.1    |
| " 5 "                          | 0.3   | 40.16              | 0.7    | 0.3   | 49.48              | 0.8    |
| " 4 "                          | 0.5   | 33.07              | 0.9    | 0.4   | 41.29              | 0.9    |
| " 3 "                          | 0.9   | 20.18              | 1.0    | 0.6   | 27.93              | 0.9    |
| " 2 "                          | 2.0   | 10.73              | 1.2    | 1.4   | 13.48              | 1.1    |
| " 1 "                          | 10.5  | 2.47               | 1.5    | 10.9  | 3.40               | 2.1    |
| Rougher Tails                  | 68.5  | 0.66               | 2.6    | 69.4  | 0.86               | 3.3    |
| Feed (calc'd)                  | 100.0 | 17.66              | 100.0  | 100.0 | 17.83              | 100.0  |

\*Distilled oleic acid.

| Test No.                       | 42-E  |                    |        | 39-E  |                    |        |
|--------------------------------|-------|--------------------|--------|-------|--------------------|--------|
| <b>Conditions:</b>             |       |                    |        |       |                    |        |
| Fineness (% -200 m)            | 87.1  |                    |        | 87.1  |                    |        |
| Soda Ash (lb/ton)              | 4.0   |                    |        | 4.0   |                    |        |
| Oleic Acid "                   | 1.0*  |                    |        | 1.0*  |                    |        |
| Condition (min)                | 2     |                    |        | 2     |                    |        |
| Pulp: pH                       | 8.0   |                    |        | 8.0   |                    |        |
| % Solids                       | 18.0  |                    |        | 18.0  |                    |        |
| Regrind Fineness<br>(% -325 m) | 92    |                    |        | 92    |                    |        |
| Hot Condition Temp             | 96° C |                    |        | 96° C |                    |        |
| Quebracho (lb/ton)             | 0.5   |                    |        | 0.125 |                    |        |
| Condition Time (min)           | 2     |                    |        | 2     |                    |        |
| <b>Results:</b>                |       |                    |        |       |                    |        |
|                                | Wt %  | CaF <sub>2</sub> % | Dist % | Wt %  | CaF <sub>2</sub> % | Dist % |
| Concentrate                    | 15.7  | 98.10              | 86.4   | 16.8  | 94.54              | 89.4   |
| Cleaner 8 Tails                | 0.4   | 91.01              | 2.0    |       |                    |        |
| " 7 "                          | 0.3   | 87.11              | 1.5    |       |                    |        |
| " 6 "                          | 0.3   | 81.75              | 1.4    | 0.5   | 72.41              | 2.1    |
| " 5 "                          | 0.2   | 70.45              | 0.8    | 0.4   | 49.25              | 1.1    |
| " 4 "                          | 0.2   | 63.06              | 0.7    | 0.5   | 38.24              | 1.1    |
| " 3 "                          | 0.4   | 45.50              | 1.0    | 0.9   | 21.90              | 1.1    |
| " 2 "                          | 1.2   | 18.11              | 1.2    | 2.0   | 10.35              | 1.2    |
| " 1 "                          | 12.2  | 3.50               | 2.4    | 10.9  | 2.37               | 1.4    |
| Rougher Tails                  | 69.1  | 0.68               | 2.6    | 68.0  | 0.67               | 2.6    |
| Feed (calc'd)                  | 100.0 | 17.84              | 100.0  | 100.0 | 17.77              | 100.0  |

\*Distilled oleic acid.

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FHH:RAW:cc