ER 2515

BENEFICIATION OF A MEXICAN FLUORITE ORE

by

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ER 2515

(Submittal Sheet)

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ABSTRACT

An effort was made to improve upon the performance of an operating flotation plant, treating a Mexican fluorite ore, by an extensive program of 48 batch flotation tests. A factorial test series generated a complex equation for the recovery of fluorite as a function of five variables, namely temperature, pH, quebracho, sodium silicate and oleic acid. A flowsheet based on a technique called release analysis consistently produced a product of 90 to 92 percent fluorite with 96 to 99 percent recovery. The desired "acid" grade product (97 percent CaF_2) was not achieved during this study, but high recovery of "ceramic" grade (93 to 95% CaF_2) was realized.

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INTRODUCTION

Summary and Conclusions

The following summary and conclusions are based on the samples provided by La Domincia and the work described in the following sections of the report.

1. A total of 48 batch flotation tests were carried out in the course of this investigation.
2. The ore sample consisted of approximately 300 pounds of fluorite ore. The average CaF_2 content of the ore was found to be 73.7 percent.
3. Preliminary studies showed that the liberation size was in the range of 100 to 150 mesh. Grinding at 50 percent solids for 10 minutes in a rod mill yielded a product which was 99 + percent passing 150 mesh.
4. A five-factor, two-level factorial test series was carried out to investigate the effects of temperature, pH, quebracho, sodium silicate and oleic acid on recovery of fluorite. The results of this testing gave a very complex equation to describe the recovery, indicating that other factors besides those tested were influencing the flotation response and/or that the system was in a highly curved region of response.
5. A flow sheet based on a technique called release analysis consistently produced a concentrate which assayed 90

to 92 percent CaF_2 with a recovery of 96 to 99 percent CaF_2. Although an acid grade concentrate was not produced (highest grade product was 95.7 percent CaF_2) the flow sheet seems very promising and deserves additional efforts to try and achieve the desired acid grade.

6. Iron (acquired from the grinding mill) interferes with the successful flotation of the fluorite resulting in a concentrate of much lower grade than normal.

Recommendations

The flowsheet as structured around the release analysis test is worthy of more study. Some factors which could be studied include:

1. Effect of grinding in a porcelain mill to overcome the apparent iron contamination problem.
2. Temperature effects on grade and recovery of the fluorite concentrate.
3. Effects of different reagent addition points and conditioning times.

Characterization of Fluorite

Fluorite, also known as fluorspar, is a commonly occuring mineral with a chemical formula of CaF_2. The fluorite crystal is cubic and the mineral's color varies widely from colorless to white to brown, also light green, yellow, blue and purple. It has a hardness of 4 and a specific gravity of 3.18. Fluorite is found associated with many other minerals including; calcite, dolomite, barite, quartz, sphalerite, and apatite (1,2,3).

Fluorite is widely distributed with the largest estimated reserves being in South Africa, with 157 million short tons. Other large reserves are held in the following countries (all values in short tons); United States - 117 million, Mexico - the largest producer - 68 million, United Kingdom - 32 million and Thailand, U.S.S.R., France and Italy-20-25 million each (4). Within the U.S. the largest producing area is in Southern Illinois and Kentucky with other producers in the western and southwestern states (1,4).

Fluorite has many uses, the principle ones being: 1) preparation of hydrofluoric acid, 2) as a flux in steel making, 3) in the manufacture of some types of glass and ceramics and as a fluorine source for other compounds (1,4).

To be of commercial value most fluorite ores need to be concentrated to increase the fluorite content. There are three grades of fluorite concentrate which have commercial application. These are (5):
1) Acid grade - minimum of 97 percent CaF_2 with 1.5 percent maximum

SiO_2 and 0.10 percent maximum sulfide sulfur; 2) Ceramic grade - 93 to 95 percent CaF_2 with 3.0 percent maximum SiO_2, 1.5 percent $CaCO_3$ maximum, 0.14 percent Fe_2O_3 maximum and no lead, zinc or sulfur, 3) Metallurgical grade - 60 percent minimum effective CaF_2 after subtracting 2.5 times the SiO_2 content from the CaF_2 content.

Prices for the concentrates have, in recent times, been largely determined by the policies of the Mexican government. Prices charged by the other large producers ususally follow this lead. In January, 1981, the minimum price for acid grade concentrate as set by the Mexican government was $138 per short ton. For metspar (70 percent effective CaF_2) the price was $107 to $112 per short ton. For the concentrates containing less than 97 percent CaF_2, an ad valorem tax of 13½ percent is added to the base price. The added tax imposed by the Mexican government provides an incentive for the Mexican producers to make acid grade concentrates (4).

Purpose of Study

The purpose of this study was to try and improve upon the performance of an operating flotation plant, located in northern Mexico, known as La Domincia.

At La Domincia the ore, after some hand sorting, ran 70 to 75 percent CaF_2, with the gangue minerals being calcium carbonate (∿20%) and silica (∿5%) with small and variable amounts of iron oxides and sulfide minerals.

The plant flowsheet (Fig. 1) was moderately complex involving one stage of rougher flotation followed by five stages of cleaning with recycle of the tailings to earlier stages. Also in the flowsheet was a retreatment step where the tailings from cleaners 2 and 3 were refloated. The concentrate from this step was recycled to the first cleaner and the tailing went to a desliming stage. The slime product was rejected to the final tailing and the coarse product went back to the grinding circuit (6).

The feed to the mill was 9 tons per hour of ore and the final concentrate averaged 4 tons per hour (6). Based on these numbers and assuming that the final concentrate was always of acid grade quality, the plant was achieving about 60 percent recovery.

It was thought that this recovery figure might be improved by undertaking a laboratory investigation of some of the factors which might influence the performance of the plant. Therefore, La Domincia provided an ore sample and samples of the reagents used in the plant for this study.

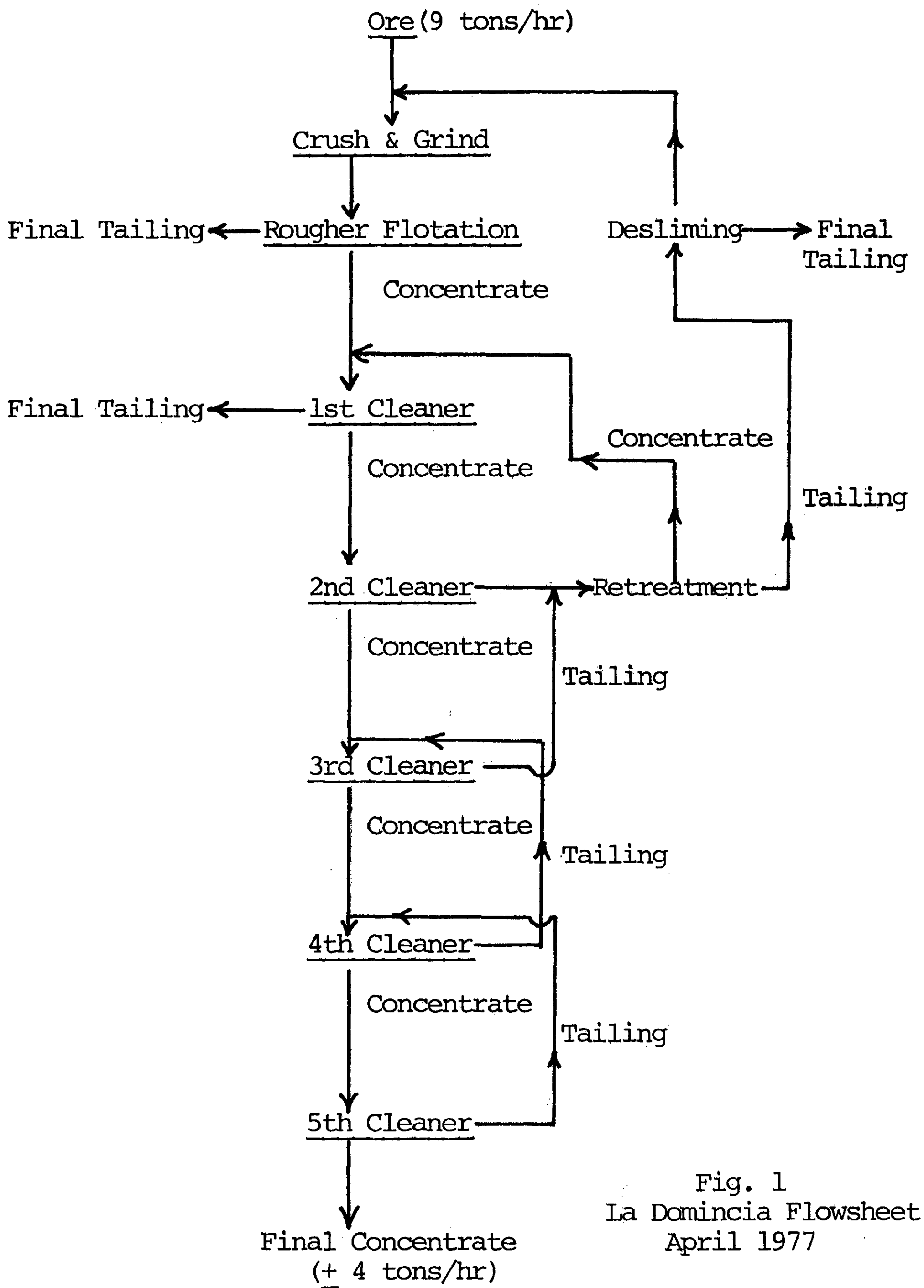

Fig. 1
La Domincia Flowsheet
April 1977

II TEST WORK AND RESULTS

Ore Description - The sample consisted of one 55 gallon barrel which contained about 300 pounds of minus 2 inch ore. The reagents provided were samples of oleic acid, quebracho and sodium carbonate used at the plant. (The sodium silicate used in this study was technical grade obtained from Van Waters and Rodgers).

Preparation of the Ore - The sample was prepared in the following manner. The ore was screened at ½ inch and the oversize material was staged crushed to minus ½ inch. The minus ½ inch ore was blended and split into one-half (retain), one-quarter (retain) and one-quarter which was crushed to nominal 10 mesh. The 10 mesh material was blended and split into one kilogram charges. Work on the project was then suspended for several months.

Upon resumption of the work, the minus ½ inch material was stage crushed to 10 mesh and all of the various splits were recombined, blended and split into halves. One half was reserved for possible future work, the other half was split into one kilogram charges.

Preliminary Studies - Preliminary studies consisted of the following steps; microscopic examination of the ore to determine liberation size, grinding studies, preliminary flotation testing and analysis of the products.

Microscopic Observations - A sample of the minus 10 mesh ore was wet and dry screened to separate the particles into size fractions. Portions of each size fraction were then examined under a microscope to determine in which size fraction the particles were liberated. This examination indicated that liberation occurred in the 100 mesh to 150 mesh fraction. Therefore, it was decided that the flotation testing should be carried out using a grind of 150 mesh.

Grinding Studies - A series of grinding and wet-and-dry screening tests were then carried out to determine the amount of grinding required to achieve the desired size. (These test results are presented in Exhibit II in the appendix.) A summary of the test results is shown below.

TABLE 1
Results of Grinding Studies

Grind Time	Percent Passing		
(min)	65 m	150 m	400 m
0	37.5	24.2	10.1
2	91.0	55.2	24.3
3	98.9	70.8	30.0
5	100.0	90.1	38.2
8	100.0	98.5	50.0
10	100.0	99.3	59.3

Flotation Testing - Two preliminary flotation tests were carried out to gain experience with the flotation characteristics of the ore and to provide some products for analysis. The five and ten minute grinding tests' products were used as feed for the flotation tests. These tests showed that the slurry formed a very stable froth which would not collapse even upon standing for long periods of time. It was also determined that the tailings could be flocculated with a 1 gpl solution of Percol 455.

Analytical Studies - Products from the preliminary flotation tests were used to develop an analytical procedure (7). (The procedure is presented in Exhibit VI in the Appendix). Results of the analysis showed that the test using the 5 minute grind yielded a concentrate of 82 percent CaF_2 with a 66.9 percent recovery and the test using the 10 minute grind yielded a concentrate of 75 percent CaF_2 with a recovery of 88 percent. Since the objective of this study was to increase the overall recovery, while achieving acid grade, it was decided to use a 10 minute grind for the subsequent test work. This would maximize recovery from which an acid grade product could then be produced.

Factorial Testing - A five-factor, two-level factorial test program was designed to test the effects and interactions of the reagents and temperature on fluorite recovery. The factors and the levels tested were: 1) temperature - $35^{\circ}C$ and $60^{\circ}C$, 2) pH - 8.5 and 10.0, 3) quebracho addition 0.2 and 0.5 lb/ton of ore, 4) sodium silicate addition 0.2 and 0.6 lb/ton and 5) oleic acid addition 0.50 and 1.50 lb/ton. The reagent addition levels were in the range of typical operating conditions (8).

These factors were chosen because they would be among the most easily adjusted factors in the operating plant. It was hoped that a slight adjustment in one or more of these factors would cause a substantial increase in the plant performance.

The purpose of the factorial test series was to generate a simple equation which would describe the response, in this case recovery of fluorite, in terms of the factors being studied. This equation could then be used for optimization of the system with respect to recovery of fluorite.

The test series and recovery results are shown in Table 2. A detailed flotation test procedure is presented in Exhibit III in the appendix.

Assay results from the 32 factorial tests showed that the average fluorite analysis was 73.7 percent. This indicated that the sample used was reasonably representative of the ore treated at the La Domincia plant.

Factorial Testing Results - The factorial tests and results in Table 2 are shown in the standard Yates order. It is possible to process the results and determine the effect of each factor and the interaction effects between combinations of factors (9,10,11). The factors and each effect and interaction effect are shown in Table 3.

The significant factors (effects) were determined by the confidence interval method. This method is described in Exhibit IV in the Appendix (12).

Those factors which were concluded to be significant were: temperature (coded x_1), temperature - pH interaction (x_1x_2), temperature - oleic acid interaction (x_1x_5), pH - sodium silicate interaction (x_2x_4), pH - oleic acid interaction (x_2x_5) and the temperature - pH - quebracho - oleic acid interaction ($x_1x_2x_3x_5$). Two other factors were found to be very nearly significant. They were pH (x_2) and quebracho (x_3).

TABLE 2

Factorial Series and Results

Test No	Response (Recovery)	Temperature x_1 60°C (+) 35°C (-)	pH x_2 10 (+) 8.5 (-)	Quebracho x_3 0.5 (+) 0.2 (-)	Na Silicate x_4 0.6 (+) 0.2 (-)	Oleic Acid x_5 1.50 (+) 0.50 (-)
1	73.9	-	-	-	-	-
2	92.8	+	-	-	-	-
3	85.5	-	+	-	-	-
4	90.0	+	+	-	-	-
5	85.7	-	-	+	-	-
6	90.4	+	-	+	-	-
7	87.3	-	+	+	-	-
8	92.8	+	+	+	-	-
9	85.3	-	-	-	+	-
10	90.9	+	-	-	+	-
11	87.2	-	+	-	+	-
12	87.2	+	+	-	+	-
13	85.6	-	-	+	+	-
14	93.3	+	-	+	+	-
15	85.8	-	+	+	+	-
16	88.4	+	+	+	+	-
17	90.1	-	-	-	-	+
18	92.3	+	-	-	-	+
19	85.8	-	+	-	-	+
20	85.8	+	+	-	-	+
21	90.5	-	-	+	-	+
22	91.5	+	-	+	-	+
23	93.5	-	+	+	-	+
24	87.6	+	+	+	-	+
25	89.3	-	-	-	+	+
26	89.3	+	-	-	+	+
27	86.3	-	+	-	+	+
28	84.5	+	+	-	+	+
29	89.4	-	-	+	+	+
30	93.7	+	-	+	+	+
31	88.8	-	+	+	+	+
32	79.6	+	+	+	+	+
33	90.8	Midpoint	Midpoint	Midpoint	Midpoint	Midpoint
34	91.5	Midpoint	Midpoint	Midpoint	Midpoint	Midpoint
35	82.0	Repeat F9 for Variance				
36	85.4	Repeat F15 for Variance				

TABLE 3
Factorial Effects and Interactions

Factor	Effect Value	Factor	Effect Value
x_1	2.51	$x_1x_2x_3$	-0.04
x_2	-1.74	$x_1x_2x_4$	-0.21
x_3	1.73	$x_1x_3x_4$	1.37
x_4	-0.68	$x_2x_3x_4$	-0.93
x_5	0.99	$x_1x_2x_5$	-0.01
		$x_1x_3x_5$	-0.11
x_1x_2	-3.04	$x_2x_3x_5$	0.67
x_1x_3	-1.17	$x_1x_4x_5$	0.86
x_1x_4	-1.36	$x_2x_4x_5$	0.53
x_1x_5	-3.68	$x_3x_4x_5$	0.28
x_2x_3	-0.29		
x_2x_4	-1.88	$x_1x_2x_3x_4$	-1.36
x_2x_5	-2.53	$x_1x_2x_3x_5$	-2.01
x_3x_4	-1.16	$x_1x_2x_4x_5$	-0.57
x_3x_5	-0.33	$x_1x_3x_4x_5$	-0.87
x_4x_5	-1.34	$x_2x_3x_4x_5$	-1.17
		$x_1x_2x_3x_4x_5$	0.48

Where x_1 = Temperature
x_2 = pH
x_3 = Quebracho
x_4 = Sodium Silicate
x_5 = Oleic Acid

Combining these factors with their coefficients to generate a response equation gave the following result. Recovery = 88.1 + a_1x_1+ a_2x_2 + a_3x_3 + $a_{12}x_1x_2$ + $a_{15}x_1x_5$ + $a_{24}x_2x_4$ + $a_{25}x_2x_5$ + $a_{1235}x_1x_2x_3x_5$ where a_1 = 1.25, a_2 = -0.87, a_3 = 0.87, a_{12} = -1.52, a_{15} = -1.84, a_{24} = -0.94, a_{25} = -1.27, a_{1235} = -1.00, x_1 = temperature value, x_2 = pH value, x_3 = quebracho value, x_4 = sodium silicate value and x_5 = oleic acid value.

An equation of this complexity indicates that there are other factors involved which influence the recovery of fluorite and/or that the system is in a highly curved region of response. Speculation on what these factors might be can lead in many directions. However, as discovered in later test work it appears that the iron obtained from the mill may have a very large adverse effect on the overall grade of the concentrate.

Since the response equation was of such a complex form, conditions for the remaining test work were chosen after close examination of the factorial series test results. The conditions of the five tests which yielded the highest recoveries were compared for similarities. If a common condition was found among the tests than it was chosen as a condition for further work. In cases where there was not a clear definition of which factor level to chose the midpoint level was compromised on. This led to a selection of the following conditions for the rest of the test work; temperature 60°C, pH 9.2, quebracho 0.5 lb/ton, sodium silicate 0.35 lb/ton and oleic acid 1.00 lb/ton. These conditions were used for all subsequent rougher flotation test work.

The final step in this work was to try and make an acid grade concentrate using the conditions chosen from the factorial testing series. The next consideration in attempting to make grade was the amount of regrinding of the concentrate required. One method of examining this parameter is a technique called release analysis. This method is discussed in the following section.

Release Analysis Testing - One method of upgrading concentrates is to regrind the concentrate to liberate the gangue minerals from the locked particles and then refloat the material, thus rejecting the liberated gangue to the tailing. This results in a final concentrate of higher grade.

Release analysis was proposed by Dell (13,14) as a means of determining how much regrinding is required to upgrade the concentrate to the desired level. Briefly, the method involves floating a slurry until the froth is barren. This separates the material into a rougher concentrate and tail. The concentrate is then cleaned several times, each time by floating the concentrate until the froth is barren. The tailings produced in each cleaning step are combined with the rougher tailing. When the amount of tailing produced from a cleaning stage becomes insignificant, the concentrate is then separated into fractions, by refloating one final time and adjusting the impeller speed. The first concentrate is collected at a low impeller speed (850 rpm). The speed is then increased (1000 rpm) and a second concentrate is collected. The impeller speed is increased once more (1200 rpm) and a third concentrate is collected and the fourth concentrate consists of the material remaining in the cell.

This method allows the highest grade particles (fastest floating) to be collected in the first concentrate, under the "gentlest" conditions where free gangue particles are least likely to be physically entrained in the froth, followed by the next highest grade in the second concentrate, etc. By analyzing the results, it can be determined how much regrinding is required, to make a desired grade, assuming recovery remains constant.

Results of the release analysis tests are summarized in Table 4 and discussed in the following section. Detailed procedures and results are presented in Exhibit V in the appendix.

The initial release analysis test (Test No. 37) yielded a combined recovery for the first three concentrates of 96.7 percent CaF_2 at a grade of 92.1 percent CaF_2. (The 1st concentrate, highest grade, had a grade of 94.4 percent CaF_2). Calculation showed that assuming constant recovery, five percent of the concentrate weight needed to be rejected to the tailing in order to make an acid grade product. However, since the recovery was already so high, constant recovery was not required and it was thought that by regrinding the concentrate and taking only the earliest floating material at least some acid grade concentrate could be obtained.

TABLE 4
Release Analysis Results

Test No.	Regrind	Depressant Addition Prior to Final Separation	Fraction	Weight %	CaF_2 % Assay	CaF_2 % Recovery	Calculated Grade Cumulative Product
37	No	No	1st Concentrate	26.1	94.4	32.0	94.4
			2nd Concentrate	29.8	94.3	36.5	94.3
			3rd Concentrate	24.9	87.0	28.2	92.1
			4th Concentrate	3.3	52.7	2.3	90.5
			Combined Tail	15.9	4.8	1.0	76.9
38	2 Minutes	No	1st Concentrate	15.2	92.2	19.7	92.2
			2nd Concentrate	34.4	89.2	43.3	90.1
			3rd Concentrate	28.1	85.9	34.0	88.6
			4th Concentrate	1.4	41.8	0.9	87.8
			Combined Tail	20.9	7.3	2.1	70.9
39	10 Minutes	No	1st Concentrate	18.4	82.6	21.1	82.6
			2nd Concentrate	46.7	82.3	53.4	82.4
			3rd Concentrate	21.6	81.2	24.3	82.1
			4th Concentrate	0.6	23.8	0.2	81.7
			Combined Tail	12.7	5.7	1.0	72.0
40	5 Minutes	No	1st Concentrate	20.4	93.6	25.5	93.6
			2nd Concentrate	44.8	92.5	55.4	92.8
			3rd Concentrate	13.6	89.2	16.3	92.2
			4th Concentrate	1.3	51.4	0.9	91.5
			Combined Tail	19.9	7.0	1.9	74.7
41	5 Minutes	Yes	1st Concentrate	35.7	94.1	44.7	94.1
			2nd Concentrate	33.1	91.6	40.4	92.9
			3rd Concentrate	12.1	83.9	13.6	91.6
			4th Concentrate	1.3	17.0	0.3	90.4
			Combined Tail	17.8	4.6	1.0	75.1
42	No	Yes	1st Concentrate	18.7	92.1	23.4	92.1
			2nd Concentrate	33.2	91.5	41.2	91.7
			3rd Concentrate	28.8	87.5	34.1	90.2
			4th Concentrate	0.8	22.7	0.2	89.5
			Combined Tail	18.5	4.6	1.1	73.8
43	60 Minutes	Yes	1st Concentrate	11.8	81.7	13.6	81.7
			2nd Concentrate	28.1	84.7	33.6	83.8
			3rd Concentrate	39.1	85.5	47.3	84.7
			4th Concentrate	4.5	70.0	4.5	83.9
			Combined Tail	16.5	4.4	1.0	70.8
44	5 Minutes	Yes Doubled	1st Concentrate	25.0	93.9	33.2	93.9
			2nd Concentrate	32.3	91.6	42.0	92.6
			3rd Concentrate	17.9	85.0	21.5	90.8
			4th Concentrate	2.1	39.4	1.2	89.4
			Combined Tail	22.7	6.5	2.1	70.6
46	No	No	1st Concentrate	28.5	95.7	38.1	95.7
			2nd Concentrate	27.5	90.7	34.8	93.3
			3rd Concentrate	5.4	76.1	5.7	91.7
			3rd Tail	7.8	71.1	7.7	89.4
			2nd Tail	8.5	52.0	6.2	85.3
			1st Tail	11.8	41.7	6.9	79.6
			Rougher Tail	10.5	4.2	0.6	71.6
47	No 5 Minute Primary Grind	No	1st Concentrate	22.8	88.1	28.2	88.1
			2nd Concentrate	36.2	82.5	41.9	84.7
			3rd Concentrate	26.8	76.9	28.9	82.2
			Combined Tail	14.2	4.5	1.0	71.2
48	No 8 Minute Primary Grind	No	1st Concentrate	17.9	93.3	23.3	93.3
			2nd Concentrate	28.9	93.2	37.5	93.2
			3rd Concentrate	31.3	85.8	37.5	90.3
			Combined Tail	21.9	5.5	1.7	71.7

The results from this test were surprising, both in terms of recovery of CaF_2 and overall grade. Therefore, a flowsheet for treating this ore was developed around this release analysis procedure. The flowsheet is shown in Figure 2.

This flowsheet in some ways is similar to the flowsheet of the La Domincia plant (fig. 1). However, it is very simplified in that there are no recycle circuits (all tailings are rejected to the final tailing) or retreatment stages. It was thought that if this treatment scheme would produce acid grade concentrate, it would be a significant improvement over the flowsheet from La Domincia. Therefore several additional tests were carried out in an effort to produce an acid grade concentrate.

Three tests (Test Nos. 38, 39, & 40) were carried out to evaluate the effects of short regrinding times, 2 minutes to 10 minutes, on the concentrate grade. Somewhat surprisingly, regrinding did not improve the overall grade of the final concentrate. In fact for the longest grind time, 10 minutes, the concentrate grade dropped dramatically, to below 83 percent.

As called for in the release analysis procedure, the only reagents which were added to the above tests, after the rougher flotation, were collector (oleic acid) and sodium carbonate to adjust the pH. Therefore it was decided to try a series of tests with short regrind periods and with quebracho and sodium silicate additions prior to the final separation step to suppress the remaining gangue. The results of these

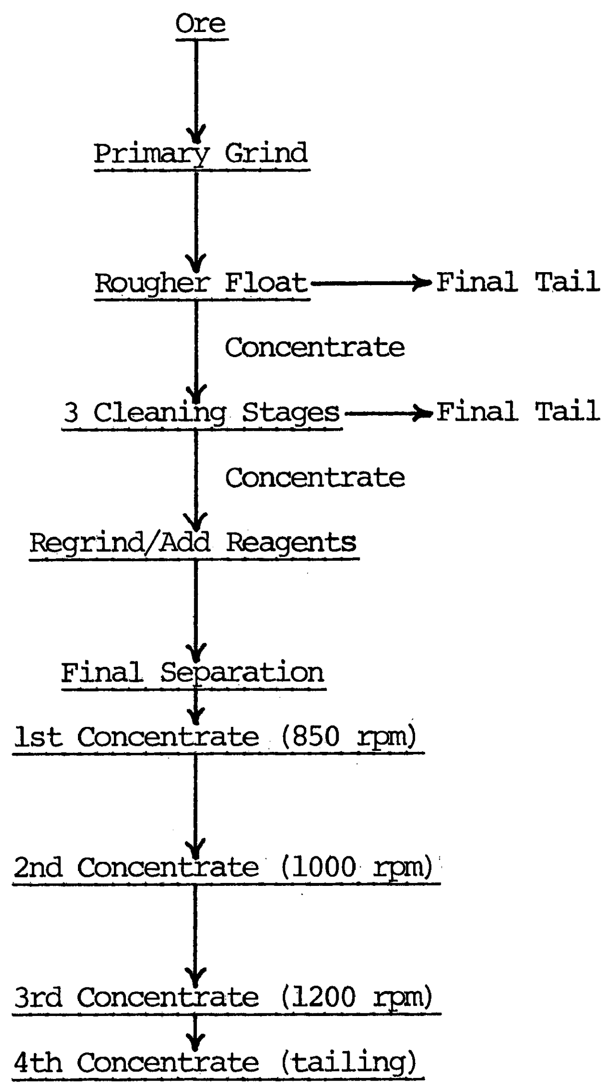

Figure 2
Release Analysis Flowsheet

tests (Test Nos. 41, 42, and 44) again showed no significant upgrading of the final concentrates.

One test (Test No. 43) was carried out to investigate the effect of a very long regrind period with the additional reagents added before the final separation. The results of this test showed that the concentrate grade once again dropped drastically, to below 85 percent.

A review of the release analysis test results showed that a concentrate of 90 to 92 percent grade and 96 to 99 percent recovery was consistently made with no regrinding or short regrinding times. Increasing the regrinding times caused the concentrate grade to drop drastically although recovery remained very high. It is thought that the iron introduced into the slurry during the long regrinding times was responsible for the dropoff in grade, and/or that even the gentlest flotation conditions still caused very fine, free gangue particles to be entrapped in the froth.

It was decided to carry out a modified release analysis test to generate cumulative grade and recovery vs flotation time data to determine if there was a definite point at which it would be advantageous to stop floating material, in order to make an acid grade concentrate.

Test No. 45 was carried out following the standard release analysis test procedure except that during the first stage of cleaning, the products were collected for specific time intervals. The products were treated in the usual manner and assayed for CaF_2 content.

The results of Test 45 are shown below:

TABLE 5
Test 45 - Concentrate Grade vs Flotation Time

Fraction	Time	Wt %	CaF_2 %
1st	30 sec	29.2	89.7
2nd	60 sec	23.0	85.1
3rd	120 sec	26.8	83.9
4th	180 sec	12.3	75.3
5th	300 sec	4.1	34.8
Tailing	--	4.6	5.1

The results showed that the higher grade material had been floated by the third minute. Therefore, it was decided to carry out the next release analysis test using cleaning stages of 2½ minutes or less instead of cleaning until the froth was barren.

Test 46 was carried out in such a manner. The first two cleaning stages were 2½ minutes long, the third cleaning stage was 2 minutes long and the concentrate was then separated into fractions in the usual manner. This technique did improve the grade of the first concentrate to 95.7 percent fluorite.

However, the overall results of the test (recovery 78.6% CaF_2, grade 91.7% CaF_2) were no better than previous tests.

Finally, it was thought that since the iron added to the slurry during regrinding had a detrimental effect on the flotation performance, perhaps the iron added during the 10 minute primary grind was also limiting the flotation performance. If this were the case, then perhaps a primary grinding time of less than 10 minutes would improve the performance by decreasing the amount of iron added to the slurry.

Therefore, the final two tests were carried out to study the effect of a primary grind of five and eight minutes. The results of these tests show that five minutes is probably too short a grinding time. The first concentrate assayed only 88 percent CaF_2 and the combined concentrate assayed 82.2 percent CaF_2 with a recovery of 99 percent. The low concentration was probably due to a liberation problem. The eight-minute primary grind gave results that were substantially the same as the earlier 10-minute grind tests. The 1st concentrate assayed 93.3 percent and the combined concentrate assayed 98.3 percent recovery at a grade of 90.3 percent.

Conclusions and Recommendations for Further Work

The response equation generated, by the factorial test series, to describe recovery of CaF_2 as a function of temperature, pH, quebracho, sodium silicate and oleic acid was very complex. This indicated that other factors besides those tested were influencing the flotation response and/or that the system was in a highly curved region of space.

A flowsheet based on a technique called release analysis consistently produced a concentrate of 90 to 92 percent CaF_2 with a recovery of 96 to 99 percent CaF_2. Although an acid grade concentrate was not produced, the flowsheet seems very promising and deserves additional efforts to try and achieve acid grade.

Factors which might be included in additional studies include: effect of grinding in a porcelain mill to overcome the apparent problem with iron, temperature effect on grade and recovery of the fluorite concentrate, and effect of different reagent addition points and conditioning times on grade and recovery of the fluorite concentrate.

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APPENDIX EXHIBIT I

Sample Description and Preparation

The ore sample and reagents were received in Golden in the spring of 1977. The ore sample consisted of one 55-gallon drum which contained approximately 300 lbs of minus 2 inch material.

The reagents consisted of 4 bottles (½ gallon) of oleic acid to be used as collector, and two 5-gallon containers, one containing sodium carbonate and the second containing quebracho. The reagents were the same as used at the La Domincia plant and were used throughout this study. The sodium silicate used in this study was technical grade obtained from Van Waters and Rodgers.

The ore sample contained roughly 70 to 75 percent fluorite, 20 percent calcite with the balance being silica, iron oxides and sulfide minerals.

The sample was prepared in the following manner. The ore was screened at ½ inch and the oversize material was stage crushed to minus ½ inch. The ½ inch ore was blended and split into one-half (retain), one-quarter (retain) and one-quarter which was then crushed to nominal 10 mesh. The 10 mesh material was blended and split into one kilogram charges. Work on the project was then suspended for several months.

Upon resumption of the work, the minus ½ inch material was stage-crushed to 10 mesh and all of the various splits were recombined, blended and split into halves. One half was further split into 1 kilogram charges and the other half was reserved for possible future work.

Appendix Exhibit II

Grinding Studies

EXHIBIT II

Screen Test No. 1

Sample: One Kilogram of Minus 10 Mesh Ore

Procedure: The sample was wet screened at 400 mesh, the two fractions were dried and the oversize was dry screened at the following sizes - 10, 28, 35, 48, 65, 100, 150, 200, 325, and 400 mesh.

Results:

Direct			Cumulative Passing	
Screen Product (Tyler) Mesh	Weight g	Weight %	(Tyler) Mesh	Weight %
+ 10	4.9	0.5	10	99.5
- 10 + 28	401.7	40.5	28	59.0
- 28 + 35	91.7	9.3	35	49.7
- 35 + 48	67.1	6.8	48	42.9
- 48 + 65	53.2	5.4	65	37.5
- 65 + 100	66.9	6.7	100	30.8
- 100 + 150	65.2	6.6	150	24.2
- 150 + 200	48.5	4.9	200	19.3
- 200 + 325	69.2	7.0	325	12.3
- 325 + 400	21.6	2.2	400	10.1
- 400	99.8	10.1		
Total	989.8	100.0		

EXHIBIT II

Screen Test No. 2

Sample: One Kilogram of Minus 10 Mesh Ore

Procedure: Sample was ground in the rod mill for 2 minutes at 50 percent solids. The sample was wet screened at 400 mesh, the two fractions were dried and the oversize was dry screened at the following sizes - 10, 28, 35, 48, 65, 100, 150, 200, 325, and 400 mesh.

Results:

Direct			Cumulative Passing	
Screen Product (Tyler) Mesh	Weight g	Weight %	(Tyler) Mesh	Weight %
+ 10			10	
- 10 + 28	0.4	-	28	
- 28 + 35	0.8	0.1	35	99.9
- 35 + 48	15.2	1.5	48	98.4
- 48 + 65	73.1	7.4	65	91.0
- 65 + 100	192.3	19.5	100	71.5
- 100 + 150	161.3	16.3	150	55.2
- 150 + 200	98.2	9.9	200	45.3
- 200 + 325	156.0	15.8	325	29.5
- 325 + 400	51.3	5.2	400	24.3
- 400	240.5	24.3		
Total	989.1	100.0		

EXHIBIT II

Screen Test No. 3

Sample: One Kilogram of Minus 10 Mesh Ore

Procedure: Sample was ground in the rod mill for 3 minutes at 50 percent solids. The sample was wet screened at 400 mesh, the two fractions were dried and the oversize was dry screened at the following sizes - 10, 28, 35, 48, 65, 100, 150, 200, 325, and 400 mesh.

Results:

Direct			Cumulative Passing	
Screen Product (Tyler) Mesh	Weight g	Weight %	(Tyler) Mesh	Weight %
+ 10		-	10	
- 10 + 28	0.1	-	28	
- 28 + 35	0.1	-	35	100.0
- 35 + 48	0.6	0.1	48	99.9
- 48 + 65	10.4	1.0	65	98.9
- 65 + 100	93.3	9.4	100	89.5
- 100 + 150	186.3	18.7	150	70.8
- 150 + 200	154.1	15.5	200	55.3
- 200 + 325	202.3	20.3	325	35.0
- 325 + 400	50.0	5.0	400	30.0
- 400	299.3	30.0		
Total	996.5	100.0		

EXHIBIT II

Screen Test No. 4

Sample: One Kilogram of Minus 10 Mesh Ore

Procedure: Sample was ground in the rod mill for 5 minutes at 50 percent solids. The sample was wet screened at 400 mesh, the two fractions were dried and the oversize was dry screened at the following sizes - 10, 28, 35, 48, 65, 100, 150, 200, 325, and 400 mesh.

Results:

Direct			Cumulative Passing	
Screen Product (Tyler) Mesh	Weight g	Weight %	(Tyler) Mesh	Weight %
+ 10		-	10	
- 10 + 28		-	28	
- 28 + 35		-	35	
- 35 + 48		-	48	
- 48 + 65	0.3	-	65	100.0
- 65 + 100	12.3	1.2	100	98.8
- 100 + 150	85.8	8.7	150	90.1
- 150 + 200	173.4	17.5	200	72.6
- 200 + 325	276.8	27.9	325	44.7
- 325 + 400	64.4	6.5	400	38.2
- 400	378.7	38.2		
Total	991.7	100.0		

EXHIBIT II

Screen Test No. 5

Sample: One Kilogram of Minus 10 Mesh Ore

Procedure: Sample was ground in the rod mill for 8 minutes at 50 percent solids. The sample was wet screened at 400 mesh, the two fractions were dried and the oversize was dry screened at the following sizes - 10, 28, 35, 48, 65, 100, 150, 200, 325, and 400 mesh.

Results:

Direct			Cumulative Passing	
Screen Product (Tyler) Mesh	Weight g	Weight %	(Tyler) Mesh	Weight %
+ 10		-	10	
- 10 + 28		-	28	
- 28 + 35		-	35	
- 35 + 48		-	48	
- 48 + 65		-	65	
- 65 + 100	1.3	0.1	100	99.9
- 100 + 150	14.4	1.4	150	98.5
- 150 + 200	84.5	8.5	200	90.0
- 200 + 325	318.3	32.0	325	58.0
- 325 + 400	79.2	8.0	400	50.0
- 400	497.1	50.0		
Total	994.8	100.0		

EXHIBIT II

Screen Test No. 6

Sample: One Kilogram of Minus 10 Mesh Ore

Procedure: Sample was ground in the rod mill for 10 minutes at 50 percent solids. The sample was wet screened at 400 mesh, the two fractions were dried and the oversize was dry screened at the following sizes - 10, 28, 35, 48, 65, 100, 150, 200, 325, and 400 mesh.

Results:

Direct			Cumulative Passing	
Screen Product (Tyler) Mesh	Weight g	Weight %	(Tyler) Mesh	Weight %
+ 10		-	10	
- 10 + 28		-	28	
- 28 + 35		-	35	
- 35 + 48		-	48	
- 48 + 65	0.2	-	65	
- 65 + 100	0.5	-	100	100.0
- 100 + 150	7.0	0.7	150	99.3
- 150 + 200	103.0	10.3	200	89.0
- 200 + 325	242.0	24.2	325	64.8
- 325 + 400	54.6	5.5	400	59.3
- 400	592.7	59.3		
Total	1000.0	100.0		

EXHIBIT II

Screen Test No. 7

Sample: One Kilogram of Minus 10 Mesh Ore

Procedure: Sample was ground in the rod mill for 12 minutes at 50 percent solids. The sample was wet screened at 400 mesh, the two fractions were dried and the oversize was dry screened at the following sizes - 10, 28, 35, 48, 65, 100, 150, 200, 325, and 400 mesh.

Results:

Direct			Cumulative Passing	
Screen Product (Tyler) Mesh	Weight g	Weight %	(Tyler) Mesh	Weight %
+ 10		-	10	
- 10 + 28		-	28	
- 28 + 35		-	35	
- 35 + 48		-	48	
- 48 + 65		-	65	
- 65 + 100	0.4	-	100	100.0
- 100 + 150	3.4	0.3	150	99.7
- 150 + 200	23.6	2.4	200	97.3
- 200 + 325	312.5	31.3	325	66.0
- 325 + 400	85.2	8.5	400	57.5
- 400	573.4	57.5		
Total	998.5	100.0		

APPENDIX EXHIBIT III
Flotation Test Procedure and Test Summaries - Factorial Series

1. A one kilogram charge of minus 10 mesh ore was ground in the rod mill for 10 minutes. The rod mill measured (inside dimensions) 7 inches in diameter by 15 inches in length. The mill was turned at 65 to 70 rpm. The rod charge consisted of the following:

	Rod Size	Number of Rods	Wt per Rod (g)	Total Wt (g)
	7/8 x 13½	3	1,300	3,900
	3/4 x 13½	1	1,025	1,025
	5/8 x 13½	15	720	10,800
	½ x 13½	10	500	5,000
Totals		28 rods		20,725 grams

2. The slurry was transferred from the mill to the 3-liter flotation cell and the cell was filled with water. The resulting slurry was about 33 percent solids.

3. A layer of insulation (1" styrofoam sheet) was placed around the cell to help retard heat loss from the heated pulp.

4. The flotation machine was started and the impeller speed was adjusted to 1500 rpm. Immersion heaters were placed in the slurry and the pulp was heated to the desired temperature.

5. When the slurry reached the desired temperature the reagents were added and the pH was adjusted to the desired value. Reagents were added as solutions in the following concentrations, oleic acid-concentrated no dilution, sodium silicate - 10 gpl, quebracho - 20 gpl and sodium carbonate - 100 gpl. The slurry was then allowed to condition for 10 minutes.

6. After conditioning, the air was turned on and flotation was carried out for five minutes. Two products were produced - a concentrate and a tail.

7. The products were flocculated with Percol 455 (1 gpl), filtered, dried and weighed.

8. The samples were then prepared for analysis.

EXHIBIT III

Flotation Test No. 1

Purpose: Test 32 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10	7.5				
Condition	10	10.0	1.5	0.6	0.5	8.0
Flotation	5	10.1				

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			74.1	
Concentrate	658.8	67.1	87.8	79.6
Tail	322.8	32.9	46.0	20.4
Total	981.6	100.0		100.0

EXHIBIT III

Flotation Test No. 2

Purpose: Test 27 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	10	1.5	0.6	0.2	5.0
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			78.8	
Concentrate	756.1	77.1	88.1	86.3
Tail	224.5	22.9	47.3	13.7
Total	980.6	100.0		100.0

EXHIBIT III

Flotation Test No. 3

Purpose: Test 9 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	8.5	0.5	0.6	0.2	2.0
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			71.6	
Concentrate	782.4	79.6	76.6	85.3
Tail	200.0	20.4	51.8	14.7
Total	982.4	100.0		100.0

EXHIBIT III

Flotation Test No. 4

Purpose: Test 17 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	8.5	1.5	0.2	0.2	2.4
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			73.3	
Concentrate	809.7	83.6	79.0	90.1
Tail	158.9	16.4	44.1	9.9
Total	968.6	100.0		100.0

EXHIBIT III

Flotation Test No. 5

Purpose: Test 4 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to $60^{o}C$	10					
Condition	10	10.1	0.5	0.2	0.2	4.4
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			72.6	
Concentrate	791.3	82.4	79.3	90.0
Tail	168.7	17.6	41.4	10.0
Total	960.0	100.0		100.0

EXHIBIT III

Flotation Test No. 6

Purpose: Test 8 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	10.2	0.5	0.2	0.5	3.2
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			77.7	
Concentrate	836.7	84.3	85.5	92.8
Tail	156.2	15.7	35.8	7.2
Total	992.9	100.0		100.0

EXHIBIT III

Flotation Test No. 7

Purpose: Test 23 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	10.0	1.5	0.2	0.5	2.5
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			73.2	
Concentrate	818.8	84.2	81.3	93.5
Tail	154.0	15.8	30.2	6.5
Total	972.8	100.0		100.0

EXHIBIT III

Flotation Test No. 8

Purpose: Test 22 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	8.5	1.5	0.2	0.5	2.4
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			71.2	
Concentrate	775.5	79.6	81.8	91.5
Tail	198.3	20.4	29.9	8.5
Total	973.8	100.0		100.0

EXHIBIT III

Flotation Test No. 9

Purpose: Test 5 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	8.5	0.5	0.2	0.5	1.4
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			73.6	
Concentrate	761.2	76.0	83.0	85.7
Tail	240.3	24.0	43.9	14.3
Total	1001.5	100.0		100.0

EXHIBIT III

Flotation Test No. 10

Purpose: Test 31 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	9.8	1.5	0.6	0.5	3.4
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			78.8	
Concentrate	780.3	81.2	86.2	88.8
Tail	180.2	18.8	47.0	11.2
Total	960.5	100.0		100.0

EXHIBIT III

Flotation Test No. 11

Purpose: Test 14 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	8.5	0.5	0.6	0.5	2.0
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			75.2	
Concentrate	852.5	84.4	83.2	93.3
Tail	158.2	15.6	32.1	6.7
Total	1010.7	100.0		100.0

EXHIBIT III

Flotation Test No. 12

Purpose: Test 10 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	8.5	0.5	0.6	0.2	2.1
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			77.2	
Concentrate	789.7	83.4	84.1	90.9
Tail	157.2	16.6	42.4	9.1
Total	946.9	100.0		100.0

EXHIBIT III

Flotation Test No. 13

Purpose: Test 16 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	10.0	0.5	0.6	0.5	2.9
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			72.8	
Concentrate	825.8	82.6	77.9	88.4
Tail	174.0	17.4	48.4	11.6
Total	999.8	100.0		100.0

EXHIBIT III

Flotation Test No. 14

Purpose: Test 6 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time	pH	Reagents, lb/ton of Ore			
	min		Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	8.4	0.5	0.2	0.5	2.3
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			72.9	
Concentrate	808.2	81.0	81.4	90.4
Tail	189.2	19.0	36.8	9.6
Total	997.4	100.0		100.0

EXHIBIT III

Flotation Test No. 15

Purpose: Test 21 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	8.4	1.5	0.2	0.5	2.0
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			74.9	
Concentrate	816.3	81.3	83.4	90.5
Tail	188.1	18.7	37.9	9.5
Total	1004.4	100.0		100.0

EXHIBIT III

Flotation Test No. 16

Purpose: Test 19 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	10.1	1.5	0.2	0.2	3.4
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			73.0	
Concentrate	762.0	79.5	78.7	85.8
Tail	196.0	20.5	50.8	14.2
Total	958.0	100.0		100.0

EXHIBIT III

Flotation Test No. 17

Purpose: Test 7 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	10.0	0.5	0.2	0.5	3.1
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			70.7	
Concentrate	805.6	81.6	75.7	87.3
Tail	181.8	18.4	48.7	12.7
Total	987.4	100.0		100.0

EXHIBIT III

Flotation Test No. 18

Purpose: Test 29 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	8.5	1.5	0.6	0.5	1.6
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			71.2	
Concentrate	779.0	81.1	78.5	89.4
Tail	181.8	18.9	39.7	10.6
Total	960.8	100.0		100.0

EXHIBIT III

Flotation Test No. 19

Purpose: Test 15 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	10.0	0.5	0.6	0.5	2.5
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			71.8	
Concentrate	777.8	79.0	78.0	85.8
Tail	206.4	21.0	48.5	14.2
Total	984.2	100.0		100.0

EXHIBIT III

Flotation Test No. 20

Purpose: Test 12 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	10.0	0.5	0.6	0.2	3.0
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			73.3	
Concentrate	763.9	76.7	83.3	87.2
Tail	231.7	23.3	40.2	12.8
Total	995.6	100.0		100.0

EXHIBIT III

Flotation Test No. 21

Purpose: Test 11 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	10.0	0.5	0.6	0.2	4.2
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			71.1	
Concentrate	794.0	82.3	75.3	87.2
Tail	171.0	17.7	51.5	12.8
Total	965.0	100.0		100.0

EXHIBIT III

Flotation Test No. 22

Purpose: Test 2 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	8.5	0.5	0.2	0.2	2.5
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			74.5	
Concentrate	834.3	84.1	82.2	92.8
Tail	158.1	15.9	33.9	7.2
Total	992.4	100.0		100.0

EXHIBIT III

Flotation Test No. 23

Purpose: Test 26 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	8.5	1.5	0.6	0.2	2.5
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			70.1	
Concentrate	791.7	79.5	78.8	89.3
Tail	204.7	20.5	36.4	10.7
Total	996.4	100.0		100.0

EXHIBIT III

Flotation Test No. 24

Purpose: Test 18 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to $60^{o}C$	10					
Condition	10	8.5	1.5	0.2	0.2	2.4
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			74.6	
Concentrate	802.7	80.6	85.4	92.3
Tail	192.9	19.4	29.6	7.7
Total	995.6	100.0		100.0

EXHIBIT III

Flotation Test No. 25

Purpose: Test 30 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	8.5	1.5	0.6	0.5	2.3
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF$_2$ %	Percent Distribution CaF$_2$
Head (Assayed)			73.7	
Head (Computed)			70.4	
Concentrate	817.4	82.5	80.0	93.7
Tail	173.5	17.5	25.2	6.3
Total	990.9	100.0		100.0

EXHIBIT III

Flotation Test No. 26

Purpose: Test 3 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	9.9	0.5	0.2	0.2	3.7
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			73.6	
Concentrate	779.2	79.7	79.0	85.5
Tail	198.5	20.3	52.6	14.5
Total	977.7	100.0		100.0

EXHIBIT III

Flotation Test No. 27

Purpose: Test 28 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	10.0	1.5	0.6	0.2	5.4
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			75.1	
Concentrate	768.2	78.1	81.3	84.5
Tail	215.1	21.9	52.9	15.5
Total	983.3	100.0		100.0

EXHIBIT III

Flotation Test No. 28

Purpose: Test 24 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time	pH	Reagents, lb/ton of Ore			
	min		Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to $60^{\circ}C$	10					
Condition	10	9.9	1.5	0.2	0.5	5.4
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			74.2	
Concentrate	746.3	76.0	85.6	87.6
Tail	235.7	24.0	38.3	12.4
Total	982.0	100.0		100.0

EXHIBIT III

Flotation Test No. 29

Purpose: Test 1 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	8.5	0.5	0.2	0.2	1.5
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			73.8	
Concentrate	698.0	70.7	77.1	73.9
Tail	289.4	29.3	65.8	24.1
Total	987.4	100.0		100.0

EXHIBIT III

Flotation Test No. 30

Purpose: Test 25 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore			
			Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	8.6	1.5	0.6	0.2	2.2
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			74.9	
Concentrate	826.3	82.7	80.9	89.3
Tail	172.8	17.3	46.2	10.7
Total	999.1	100.0		100.0

EXHIBIT III

Flotation Test No. 31

Purpose: Test 13 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	8.6	0.5	0.6	0.5	2.0
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			75.6	
Concentrate	789.2	78.8	82.1	85.6
Tail	212.8	21.2	51.4	14.4
Total	1002.0	100.0		100.0

EXHIBIT III

Flotation Test No. 32

Purpose: Test 20 from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10					
Condition	10	10.0	1.5	0.2	0.2	4.4
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			73.0	
Concentrate	736.3	75.2	83.3	85.8
Tail	243.3	24.8	41.8	14.2
Total	979.6	100.0		100.0

EXHIBIT III

Flotation Test No. 33

Purpose: Midpoint test from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 47°C	7					
Condition	10	9.3	1.0	0.4	0.35	2.3
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			72.7	
Concentrate	798.8	81.4	81.1	90.8
Tail	182.6	18.6	36.0	9.2
Total	981.4	100.0		100.0

EXHIBIT III

Flotation Test No. 34

Purpose: Midpoint test from Factorial Series

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 47°C	7					
Condition	10	9.3	1.0	0.4	0.35	2.5
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			71.5	
Concentrate	822.7	82.9	78.9	91.5
Tail	170.2	17.1	35.6	8.5
Total	992.9	100.0		100.0

EXHIBIT III

Flotation Test No. 35

Purpose: Test 9 from Factorial Series - Repeat for Variance

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35^{o}C	5					
Condition	10	8.5	0.5	0.2	0.5	1.2
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			73.3	
Concentrate	755.1	75.9	79.1	82.0
Tail	239.3	24.1	55.0	18.0
Total	994.4	100.0		100.0

EXHIBIT III

Flotation Test No. 36

Purpose: Test 15 from Factorial Series - Repeat for Variance

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 35°C	5					
Condition	10	8.5	0.54	0.6	0.5	1.6
Flotation	5					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			75.5	
Concentrate	824.0	83.2	77.5	85.4
Tail	166.3	16.8	44.4	14.6
Total	990.3	100.0		100.0

APPENDIX EXHIBIT IV
Factorial Tests - Calculation of Effect Values

The effect values are calculated from the individual test results (response = recovery) in the following manner. The response values are added together according to the algebraic sign associated with each response. Thus for each effect or interaction effect there are 16 positive responses and 16 negative responses. This sum is known as the effect total. By dividing the effect total by one half of the number of tests in the design, the effect value is obtained. The results and calculations are summarized in Tables 6 and 7. (9,10,11,12)

One method of determining which effects are significant is known as the confidence interval method. An estimate of response error can be made from replicated tests. For the case of duplicated tests an estimate of s, the standard deviation, is given by:

$$S = \sqrt{(Y_1 - Y_2)^2/2}$$

Where Y_1 and Y_2 are the response values for the two tests. For several duplicated tests a pooled estimate of s can be made:

$$S^2 = \Sigma(r_i - 1)\ S_i^2 / \ (r_i - 1)$$

Where S_i is the estimated error from each replicated test and r_i is the number of replications of each test. (In this case $r_i = 2$).

In this study three tests were replicated. They were: 1) the midpoint test, 2) Test 9 and 3) Test 15. The results of the replicated tests are summarized below:

TABLE 6
Factorial Series and Results

Test No	Response (Recovery)	Temperature x_1 60°C (+) 35°C (-)	pH x_2 10 (+) 8.5 (-)	Quebracho x_3 0.5 (+) 0.2 (-)	Na Silicate x_4 0.6 (+) 0.2 (-)	Oleic Acid x_5 1.50 (+) 0.50 (-)
1	73.9	-	-	-	-	-
2	92.8	+	-	-	-	-
3	85.5	-	+	-	-	-
4	90.0	+	+	-	-	-
5	85.7	-	-	+	-	-
6	90.4	+	-	+	-	-
7	87.3	-	+	+	-	-
8	92.8	+	+	+	-	-
9	85.3	-	-	-	+	-
10	90.9	+	-	-	+	-
11	87.2	-	+	-	+	-
12	87.2	+	+	-	+	-
13	85.6	-	-	+	+	-
14	93.3	+	-	+	+	-
15	85.8	-	+	+	+	-
16	88.4	+	+	+	+	-
17	90.1	-	-	-	-	+
18	92.3	+	-	-	-	+
19	85.8	-	+	-	-	+
20	85.8	+	+	-	-	+
21	90.5	-	-	+	-	+
22	91.5	+	-	+	-	+
23	93.5	-	+	+	-	+
24	87.6	+	+	+	-	+
25	89.3	-	-	-	+	+
26	89.3	+	-	-	+	+
27	86.3	-	+	-	+	+
28	84.5	+	+	-	+	+
29	89.4	-	-	+	+	+
30	93.7	+	-	+	+	+
31	88.8	-	+	+	+	+
32	79.6	+	+	+	+	+
33	90.8	Midpoint	Midpoint	Midpoint	Midpoint	Midpoint
34	91.5	Midpoint	Midpoint	Midpoint	Midpoint	Midpoint
35	82.0	Repeat F9 for Variance				
36	85.4	Repeat F15 for Variance				

TABLE 7
Factorial Summary

Factor[1]	Sum of Positive Responses	Sum of Negative Responses	Effect Total	Effect Value	Significance Test 95% Confidence Interval
* x_1	1430.1	1390.0	40.1	2.51	0.58–4.44
* x_2	1396.1	1424.0	-27.9	-1.74	-3.67–0.19
* x_3	1423.9	1396.2	27.7	1.73	-0.20–3.66
x_4	1404.6	1415.5	-10.9	-0.68	-2.61–1.25
x_5	1418.0	1402.1	15.9	0.99	-0.94–2.92
* x_1x_2	1385.7	1434.4	-48.7	-3.04	-4.97–-1.11
x_1x_3	1400.7	1419.4	-18.7	-1.17	-3.10–0.76
x_1x_4	1399.2	1420.9	-21.7	-1.36	-3.29–0.57
* x_1x_5	1380.6	1439.5	-58.9	-3.68	-5.61–-1.75
x_2x_3	1407.7	1412.4	- 4.7	-0.29	-2.22–1.64
* x_2x_4	1395.0	1425.1	-30.1	-1.88	-3.81–0.05
* x_2x_5	1389.8	1430.3	-40.5	-2.53	-4.46–-0.60
x_3x_4	1400.8	1419.3	-18.5	-1.16	-3.09–0.77
x_3x_5	1407.4	1412.7	- 5.3	-0.33	-2.26–1.60
x_4x_5	1399.3	1420.8	-21.5	-1.34	-3.27–0.59
$x_1x_2x_3$	1409.7	1410.4	- 0.7	-0.04	-1.97–1.89
$x_1x_2x_4$	1408.4	1411.7	- 3.3	-0.21	-2.14–1.72
$x_1x_3x_4$	1421.0	1399.1	21.9	1.37	-0.56–3.30
$x_2x_3x_4$	1402.6	1417.5	-14.9	-0.93	-2.86–1.00
$x_1x_2x_5$	1410.0	1410.1	- 0.1	-0.01	-1.94–1.92
$x_1x_3x_5$	1409.2	1410.9	- 1.7	-0.11	-2.04–1.82
$x_2x_3x_5$	1415.4	1404.7	10.7	0.67	-1.26–2.60
$x_1x_4x_5$	1416.9	1403.2	13.7	0.86	-1.07–2.79
$x_2x_4x_5$	1414.3	1405.8	8.5	0.53	-1.40–2.46
$x_3x_4x_5$	1412.3	1407.8	4.5	0.28	-1.65–2.21
$x_1x_2x_3x_4$	1399.2	1420.9	-21.7	-1.36	-3.29–0.57
* $x_1x_2x_3x_5$	1394.0	1426.1	-32.1	-2.01	-3.94–-0.08
$x_1x_2x_4x_5$	1405.5	1414.6	- 9.1	-0.57	-2.50–1.36
$x_1x_3x_4x_5$	1403.1	1417.0	-13.9	-0.87	-2.80–1.06
$x_2x_3x_4x_5$	1400.7	1419.4	-18.7	-1.17	-3.10–0.76
$x_1x_2x_3x_4x_5$	1413.9	1406.2	7.7	0.48	-1.45–2.41

[1] x_1 - Temperature
x_2 - pH
x_3 - Quebracho
x_4 - Sodium Silicate
x_5 - Oleic Acid

* Significant Effect or Interaction

Test	Response (recovery)
Midpoint	90.8
Midpoint	91.5
Test 9	85.3
Test 9	82.0
Test 15	85.8
Test 15	85.4

Inserting these results into the equations above a pooled value for S^2 is obtained:

$$S^2 = 1.9233$$

With this value of S^2 an estimate of the confidence interval can be made with the following formula:

$$\text{Confidence interval} = (\text{Main effect estimate} \pm ts/\sqrt{N/4})$$

Where S is the response error, t is the student's t with the number of degrees of freedom associated with S, and N is the total number of tests in the factorial series. (For a duplicated test the number of degrees of freedom is equal to 1, i.e. (r-1) where r is 2. For a pooled estimate of S the degrees of freedom are also pooled. Therefore for 3 duplicated tests the number of degrees of freedom is equal to 3, i.e. 3 x 1.)

For a 95% confidence limit, the value of t is 3.181. Inserting the necessary values into the confidence interval equation, the result is:

$$\text{Confidence Interval} = (\text{effect estimate}) \pm 1.93$$

For any effect estimate whose interval does not include zero, it is concluded that the effect is significantly different than zero. Using this criterion on the data developed from the factorial series, the following factors (effects) and interaction effects were found to be significant: Temperature (coded X_1), temperature -pH interaction (x_1x_2), temperature - Oleic Acid interaction (x_1x_5), pH-sodium silicate interaction (x_2x_4), pH-Oleic acid interaction (x_2x_5) and temperature - pH - quebracho - oleic acid interaction ($x_1x_2x_3x_5$). In addition pH (x_2) and quebracho (x_3) are very nearly significant and might be included in any equation generated to describe the system.

To generate such an equation the linear coefficients are equal to one half of the effect value. The linear coefficients and their corresponding factors are combined with the average response of all the factorial tests. This results in the following equation:

$$\text{Response (\% Recovery)} = 88.1 + a_1x_1 + a_2x_2 + a_3x_3 + a_{12}x_1x_2 + a_{15}x_1x_5 + a_{24}x_2x_4 + a_{25}x_2x_5 + a_{1235}x_1x_2x_3x_5$$

Where $a_1 = 1.25$
$a_2 = -0.87$
$a_3 = 0.87$
$a_{12} = -1.52$
$a_{15} = -1.84$
$a_{24} = -0.94$
$a_{25} = -1.27$
$a_{1235} = -1.00$

and x_1 = temperature value
x_2 = pH value
x_3 = quebracho value
x_4 = sodium silicate value
x_5 = oleic acid value

(For a detailed description of the methods and procedures used in this analysis refer to references 9-12 as shown in the References Cited listing).

APPENDIX EXHIBIT V
Flotation Test Procedure and Test Summaries - Release Analysis Tests

The flotation test procedure for the release analysis tests was identical to the procedure for the factorial series through the conditioning step. After the conditioning step the procedure was as follows:

1. After conditioning, the air was turned on and flotation was carried out until the froth was barren. About half way through this test preheated make up water was added to the cell.

2. After the rougher flotation the unfloated material was flocculated with Percol 455 filtered and dried. The rougher concentrate was cleaned three times, in the same manner as the rougher flotation was carried out. This allowed the mechanically trapped gangue to report to the tailing. To each recleaning stage 0.1 lbs/ton of oleic acid was added and the pH was adjusted to 9.2.

3. The tailing from each stage of cleaning was also flocculated, filtered and dried.

4. After three stages of cleaning the concentrate was ready for the final separation process. Sometimes, prior to the final separation the concentrate was reground. The final separation consisted of floating fractions from the slurry by adjusting the impeller speed. The first concentrate was collected at 850 rpm, the second at 1000 rpm and the third at 1200 rpm. The fourth concentrate was the unfloated material.

5. Five products were made for analysis, the four concentrates as mentioned above and a combined tail product.

EXHIBIT V

Flotation Test No. 37

Purpose: Release Analysis Test

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	4.0
Rougher Flotation	25	1500					
1st Cleaning stage	35	1500	9.2	0.1			0.2
2nd Cleaning stage	35	1500	9.2	0.1			0.8
3rd Cleaning stage	35	1500	9.2	0.1			0.4
1st Concentrate	10	850	9.2				0.4
2nd Concentrate	10	1000					
3rd Concentrate	10	1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			76.9	
1st Concentrate	255.1	26.1	94.4	32.0
2nd Concentrate	291.2	29.8	94.3	36.5
3rd Concentrate	243.9	24.9	87.0	28.2
4th Concentrate	32.5	3.3	52.7	2.3
Combined Tail	155.7	15.9	4.8	1.0
Total	978.4	100.0		100.0

EXHIBIT V

Flotation Test No. 38

Purpose: Release Analysis Test - Two minute regrind prior to separating the concentrates

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore			
				Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	4.0
Rougher Flotation		1500					
1st Cleaning stage		1500	9.2	0.1			0.4
2nd Cleaning stage		1500	9.2	0.1			1.0
3rd Cleaning stage		1500	9.2	0.1			0.8
1st Concentrate		850	9.2				1.4
2nd Concentrate		1000					
3rd Concentrate		1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			70.9	
1st Concentrate	145.0	15.2	92.2	19.7
2nd Concentrate	329.0	34.4	89.2	43.3
3rd Concentrate	268.3	28.1	85.9	34.0
4th Concentrate	13.8	1.4	41.8	0.9
Combined Tail	200.4	20.9	7.3	2.1
Total	956.5	100.0		100.0

EXHIBIT V

Flotation Test No. 39

Purpose: Release Analysis Test - Ten minute regrind prior to separating the concentrates

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	4.2
Rougher Flotation		1500					
1st Cleaning stage		1500	9.2	0.1			0.6
2nd Cleaning stage		1500	9.2	0.1			0.6
3rd Cleaning stage		1500	9.2	0.1			0.5
1st Concentrate		850	9.2	0.1			1.2
2nd Concentrate		1000					
3rd Concentrate		1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			72.0	
1st Concentrate	177.6	18.4	82.6	21.1
2nd Concentrate	452.2	46.7	82.3	53.4
3rd Concentrate	208.8	21.6	81.2	24.3
4th Concentrate	5.6	0.6	23.8	0.2
Combined Tail	123.5	12.7	5.7	1.0
Total	967.7	100.0		100.0

EXHIBIT V

Flotation Test No. 40

Purpose: Release Analysis Test - Five minute regrind prior to separating the concentrates

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	4.0
Rougher Flotation		1500					
1st Cleaning stage		1500	9.2	0.1			0.6
2nd Cleaning stage		1500	9.2	0.1			1.2
3rd Cleaning stage		1500	9.2	0.1			0.6
1st Concentrate		850	9.2	0.1			1.4
2nd Concentrate		1000					
3rd Concentrate		1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			74.7	
1st Concentrate	196.6	20.4	93.6	25.5
2nd Concentrate	431.6	44.8	92.5	55.4
3rd Concentrate	131.3	13.6	89.2	16.3
4th Concentrate	12.6	1.3	51.4	0.9
Combined Tail	191.7	19.9	7.0	1.9
Total	963.8	100.0		100.0

EXHIBIT V

Flotation Test No. 41

Purpose: Release Analysis Test - Five minute regrind followed by additional reagents prior to separating the concentrates

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	3.0
Rougher Flotation		1500					
1st Cleaning stage		1500	9.2	0.1			0.2
2nd Cleaning stage		1500	9.2	0.1			0.2
3rd Cleaning stage		1500	9.2	0.1			0.2
1st Concentrate		850	9.2	0.2	0.1	0.1	0.7
2nd Concentrate		1000					
3rd Concentrate		1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			75.1	
1st Concentrate	349.1	35.7	94.1	44.7
2nd Concentrate	324.4	33.1	91.6	40.4
3rd Concentrate	118.9	12.1	83.9	13.6
4th Concentrate	13.0	1.3	17.0	0.3
Combined Tail	174.1	17.8	4.6	1.0
Total	979.5	100.0		100.0

EXHIBIT V

Flotation Test No. 42

Purpose: Release Analysis Test - Additional reagents prior to separating the concentrates

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	2.6
Rougher Flotation		1500					
1st Cleaning stage		1500	9.2	0.1			0.2
2nd Cleaning stage		1500	9.2	0.1			0.2
3rd Cleaning stage		1500	9.2	0.1			0.2
1st Concentrate		850	9.2	0.2	0.1	0.1	0.4
2nd Concentrate		1000					
3rd Concentrate		1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			73.8	
1st Concentrate	183.4	18.7	92.1	23.4
2nd Concentrate	325.1	33.2	91.5	41.2
3rd Concentrate	281.5	28.8	87.5	34.1
4th Concentrate	8.0	0.8	22.7	0.2
Combined Tail	180.9	18.5	4.6	1.1
Total	978.9	100.0		100.0

EXHIBIT V

Flotation Test No. 43

Purpose: Release Analysis Test - Sixty minutes regrind followed by additional reagents prior to separating the concentrates.

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	3.0
Rougher Flotation		1500					
1st Cleaning stage		1500	9.2				0.3
2nd Cleaning stage		1500	9.2				0.2
3rd Cleaning stage		1500	9.2				0.3
1st Concentrate		850	9.2	0.2	0.1	0.1	0.7
2nd Concentrate		1000					
3rd Concentrate		1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			70.8	
1st Concentrate	117.2	11.8	81.7	13.6
2nd Concentrate	278.6	28.1	84.7	33.6
3rd Concentrate	388.5	39.1	85.5	47.3
4th Concentrate	45.2	4.5	70.0	4.5
Combined Tail	163.2	16.5	4.4	1.0
Total	992.7	100.0		100.0

EXHIBIT V

Flotation Test No. 44

Purpose: Release Analysis Test - Five minute regrind followed by double the reagent addition prior to separating the concentrates

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	3.1
Rougher Flotation		1500					
1st Cleaning stage		1500	9.2	0.1			0.3
2nd Cleaning stage		1500	9.2	0.1			0.2
3rd Cleaning stage		1500	9.2	0.1			0.2
1st Concentrate		850	9.2	0.2	0.2	0.2	0.4
2nd Concentrate		1000					
3rd Concentrate		1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			70.6	
1st Concentrate	239.7	25.0	93.9	33.2
2nd Concentrate	310.4	32.3	91.6	42.0
3rd Concentrate	171.5	17.9	85.0	21.5
4th Concentrate	20.2	2.1	39.4	1.2
Combined Tail	218.6	22.7	6.5	2.1
Total	960.4	100.0		100.0

EXHIBIT V

Flotation Test No. 45

Purpose: To develop data for a recovery curve

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	2.8
Rougher Flotation		1500					
1st Concentrate	0.5	1500	9.2	0.1			0.3
2nd Concentrate	0.5	1500					
3rd Concentrate	1.0	1500					
4th Concentrate	1.0	1500					
5th Concentrate	2.0	1500					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			71.1	
1st Concentrate	255.1	26.0	89.7	32.8
2nd Concentrate	200.6	20.5	85.1	24.5
3rd Concentrate	234.2	23.9	83.9	28.2
4th Concentrate	107.5	11.0	75.3	11.6
5th Concentrate	35.6	3.6	34.8	1.8
Cleaner Tail	40.2	4.1	5.1	0.3
Rougher Tail	106.5	10.9	5.0	0.8
Total	979.7	100.0		100.0

EXHIBIT V

Flotation Test No. 46

Purpose: Resease Analysis Test - Shortened Cleaning Stages

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for 10 minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	2.8
Rougher Flotation		1500					
1st Cleaning stage	2.5	1500	9.2	0.1			0.3
2nd Cleaning stage	2.5	1500	9.2	0.1			0.2
3rd Cleaning stage	2.0	1500	9.2	0.1			0.2
1st Concentrate		850	9.2	0.1			0.2
2nd Concentrate		1000					
3rd Concentrate		1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			71.6	
1st Concentrate	276.1	28.5	95.7	38.1
2nd Concentrate	265.8	27.5	90.7	34.8
3rd Concentrate	52.3	5.4	76.1	5.7
3rd Stage Tail	75.1	7.8	71.1	7.7
2nd Stage Tail	82.1	8.5	52.0	6.2
1st Stage Tail	114.6	11.8	41.7	6.9
Rougher Tail	102.0	10.5	4.2	0.6
Total	968.0	100.0		100.0

EXHIBIT V

Flotation Test No. 47

Purpose: Release Analysis Test

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for five minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore: Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	3.0
Rougher Flotation		1500					
1st Cleaning stage		1500	9.2	0.1			0.2
2nd Cleaning stage		1500	9.2	0.1			0.2
3rd Cleaning stage		1500	9.2	0.1			0.2
1st Concentrate		850	9.2	0.1			0.2
2nd Concentrate		1000					
3rd Concentrate		1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			71.2	
1st Concentrate	220.2	22.8	88.1	28.2
2nd Concentrate	349.0	36.2	82.5	41.9
3rd Concentrate	258.5	26.8	76.9	28.9
Combined Tail	137.2	14.2	4.5	1.0
Total	964.9	100.0		100.0

EXHIBIT V

Flotation Test No. 48

Purpose: Resease Analysis Test

Sample: One Kilogram of minus 10 mesh ore, ground in the rod mill for eight minutes at 50% solids

Test Conditions:

	Time min	Impeller Speed (RPM)	pH	Reagents, lb/ton of Ore Oleic Acid	Sodium Silicate	Quebracho	Sodium Carbonate
Heat to 60°C	10						
Condition	10	1500	9.2	1.0	0.4	0.5	3.0
Rougher Flotation		1500					
1st Cleaning stage		1500	9.2	0.1			0.2
2nd Cleaning stage		1500	9.2	0.1			0.2
3rd Cleaning stage		1500	9.2	0.1			0.2
1st Concentrate		850	9.2	0.1			0.2
2nd Concentrate		1000					
3rd Concentrate		1200					

Results:

Product	Weight g	Weight %	Chemical Analysis CaF_2 %	Percent Distribution CaF_2
Head (Assayed)			73.7	
Head (Computed)			71.7	
1st Concentrate	177.5	17.9	93.3	23.3
2nd Concentrate	286.7	28.9	93.2	37.5
3rd Concentrate	311.2	31.3	85.8	37.5
Combined Tail	217.3	21.9	5.5	1.7
Total	992.7	100.0		100.0

APPENDIX EXHIBIT VI
Determination of Fluorine Content - Analytical Procedure (7)

Equipment Used:

1. Leeds and Nothrup Model 7410 pH - Specific ion meter
2. Orion Model 94-09 Fluoride Ion Selective Electrode
3. Orion Model 94-01 Reference Electrode
4. Magnetic Stirrer and Stirring Bars
5. Several 200 ml and 250 ml volumetric flasks
6. Several 250 ml beakers
7. Several 25 ml pipettes
8. Several Nickel Crucibles (30 to 50 ml volumes)
9. Analytical Balance
10. Filter funnels
11. Filter paper
12. Spatulas

Reagents Used:

1. Dilute Hydrochloric Acid (1 to 3)
2. Sodium Citrate
3. Sodium Carbonate
4. Potassium Carbonate
5. Sodium Fluoride
6. Distilled or Deionized water

Preparation of Stock Reagent Solutions

1. Dilute Hydrochloric acid (1:3) - prepared by mixing one volume of concentrated acid to three volumes of water. This solution is used to adjust the pH of the sample solutions.

2. Sodium Citrate Buffer - prepared by making a one molar solution of sodium citrate and carefully acidifying the solution to pH 6 with hydrochloric acid. This solution provides a constant pH and ionic strength for the analysis and prevents the formation of fluoride ion complexes.

3. Dilute Sodium Carbonate (10 gpl) - prepared by dissolving 10 grams of sodium carbonate in one liter of water. Used for washing residues during filtration.

4. Mixed Sodium and Potassium Carbonate solution (40 gpl of each) - prepared by mixing 40 grams of each carbonate in one liter of water. Used to provide the proper matrix for the standard solutions.

5. Sodium Fluoride Stock Solution (1000 ppm) - prepared by dissolving 2.0756 grams of sodium fluoride in one liter of water. Used for preparing standard solutions to calibrate the pH-specific ion meter.

6. Mixed Sodium and Potassium Carbonates (solid reagents) - prepared by mixing together equivalent weights of each of the carbonates as dry reagents. This mixture is used in the fusion step.

Calibration - Preparation of Standards

In order to properly perform this procedure for fluorine analysis, it is necessary to calibrate the pH - specific ion meter using standard solutions with known fluoride ion concentrations.

The standard solutions were prepared by first making a stock solution of a 1000 ppm fluoride. Aliquots of this solution were diluted to make the standard solutions. Dilute hydrochloric acid and a solution of sodium and potassium carbonates were also added to the standards to approximate the matrix of the unknown samples. The amounts of each component are shown in the table below:

Standard Solution (ppm F)	Volume of 1000 ppm solution (ml)	Volume of Dilute HCl (ml)	Volume of Sodium/Potassium Carbonates Solution (ml)	Total Volume (ml)
20	5	35	50	250
50	10	28	40	200
100	25	35	50	250
150	30	28	40	200
200	50	35	50	250
250	50	28	40	200

These solutions were diluted with deionized water to the total volume indicated.

A 25 ml volume of each standard solution was transferred to a beaker which contained 25 ml of the sodium citrate buffer solution.

The solution was stirred on the magnetic stirrer and the electrodes were placed in the solution and the pH - specific ion meter was activated. After allowing the electrodes to stabilize for several minutes the millivolt reading on the meter was recorded and the next standard solution was measured. Typical readings are shown below:

Standard Solution	mV Reading
20 ppm	120.0
50 ppm	96.7
100 ppm	78.8
150 ppm	68.2
200 ppm	61.0
250 ppm	55.0

The meter was adjusted to read 120 millivolts for the 20 ppm solution on the expanded (200 millivolt) scale.

These results were then plotted on semilog paper, millivolt reading vs log of the standard solution concentration. This plot results in a straight line with a slope of about -59. The plot is then used to determine the values of the unknown solutions.

Analytical Procedure

1. A small amount of sample to be analyzed (usually less than 0.1000 grams) was accurately weighed and then mixed with 4 grams of the sodium and potassium carbonate (1 to 1 fusion mixture) in a nickel crucible.

2. The mixture in the crucible was heated to melting (fused) over a bunsen burner and then allowed to cool.

3. The resulting fused mass was removed from the crucible by placing the crucible on its side in a 250 ml beaker containing 100 ml of water. The water was heated to boiling and then the beaker and its contents were allowed to cool. Any of the fused mass which remained in the crucible was removed to the solution with a spatula.

4. The resulting solution (water and fused materials) was reheated to boiling and then allowed to cool.

5. The solution was then filtered into a 250 ml flask containing 35 ml of the dilute hydrochloric acid solution. During filtration the residue was washed with portions of the 10 gpl sodium carbonate solution.

6. After filtering the solutions were gently agitated to expel the carbon dioxide which formed. The solutions were then allowed to stand for several hours (12-18).

7. After standing the solutions were made up to volume (250 ml) by diluting with deionized water.

8. A 25 ml volume of the unknown solution was transferred to a beaker which contained 25 ml of the sodium citrate buffer solution. The solution was stirred on the magnetic stirrer and the electrodes were placed in the solution and the pH - specific ion meter was activated. After allowing the electrodes to stabilize for several minutes the millivolt reading on the meter was recorded and the fluoride ion concentration was determined from the plot of the standard solution results.