Publication No. 02-082-105

CHARACTERIZATION OF FUTURE FLORIDA PHOSPHATE RESOURCES

Prepared by

Hassan El-Shall & Mike Bogan

for

FIPR
Florida Institute of
Phosphate Research

July 1994

The Florida Institute of Phosphate Research was created in 1978 by the Florida Legislature (Chapter 378.101, Florida Statutes) and empowered to conduct research supportive to the responsible development of the state's phosphate resources. The Institute has targeted areas of research responsibility. These are: reclamation alternatives in mining and processing, including wetlands reclamation, phosphogypsum storage areas and phosphatic clay containment areas; methods for more efficient, economical and environmentally balanced phosphate recovery and processing; disposal and utilization of phosphatic clay; and environmental effects involving the health and welfare of the people, including those effects related to radiation and water consumption.

FIPR is located in Polk County, in the heart of the central Florida phosphate district. The Institute seeks to serve as an information center on phosphate-related topics and welcomes information requests made in person, by mail, or by telephone.

Research Staff

Executive Director
Richard F. McFarlin

Research Directors

G. Michael Lloyd Jr.	**-Chemical Processing**
Jinrong P. Zhang	**-Mining & Beneficiation**
Steven G. Richardson	**-Reclamation**
Gordon D. Nifong	**-Environmental Services**

Florida Institute of Phosphate Research
1855 West Main Street
Bartow, Florida 33830
(863) 534-7160
Fax:(863) 534-7165

CHARACTERIZATION OF FUTURE FLORIDA PHOSPHATE RESOURCES

Final Report
(FIPR Contract No. 89-02-082R)

BY

Hassan El-Shall and Michael Bogan

Prepared for the
FLORIDA INSTITUTE OF PHOSPHATE RESEARCH
1855 West Main Street
Bartow, FL 33830

July 1994

DISCLAIMER

PERSPECTIVE

By Patrick Zhang
Director of Beneficiation Research

Florida has two major phosphate deposits: the Bone Valley Formation and the Hawthorn Formation. The high grade, easy-to-process Bone Valley deposit is being depleted rapidly, and the mining industry is moving south/southwest to the Southern Extension of Bone Valley formation and the Hawthorn Formation. FIPR initiated this in-house study in November, 1989 to characterize in considerable detail the mining and beneficiation challenges the industry will face as it proceeds southward. We also anticipated that this study would highlight research directions that FIPR and the industry should take to address these technical problems.

Several phosphate mining companies supplied FIPR with core samples collected from both the upper and lower zone of the Southern Extension. These samples were subjected to the following analyses: chemical properties, mineralogical compositions, beneficiation behaviors, rheology of the matrix, consolidation characteristics of the fine slimes and heavy minerals distribution.

As a general comparison with the Bone Valley deposit, this study showed that these future ore bodies are higher in dolomite, clay and silica (insoluble), but lower in P_2O_5 and aluminum phosphate content.

SIGNIFICANT FINDINGS

DOLOMITE Although the increased dolomite content in the future deposits has long been recognized, this study has underscored the seriousness of dolomite contamination, particularly in the Lower Zone. While the concentrates do contain over 1% MgO, the pebble fractions analyze up to 6% MgO. Obviously, the traditional washing followed by the "double flotation" will not produce an acceptable product.

LOSS OF PHOSPHATE VALUES IN THE SLIMES The investigators showed that the potential of P_2O_5 loss with the -150 mesh fraction (slimes) is still very significant: 20% in the upper matrix zone and 11% in the lower zone.

HEAVY MINERALS Because the phosphate deposits are associated with small quantities of heavy minerals (rutile-ilmenite, staurolite, sillimanite, zircon, etc.), there arises periodically interest in recovering them economically. Data from this study are not very encouraging: the upper zone would yield only 110 tons of heavy minerals per million tons of matrix, rendering just 4.5 tons of Rutile-Ilmenite, 5.3 tons of Staurolite, and 5.3 tons of Sillimanite in the amine tail.

Table of Contents

Table of Contents
Appendix I

Table of Contents
Appendix II

Table of Contents
Appendix III

Table of Contents
Appendix IV

List of Tables

Matrix

Pebble

Feed

Concentrate

Empirical Calculations

Heavy Minerals

Trace Elements

Phosphatic Clays

Summary

EXECUTIVE SUMMARY

The available high-grade phosphate reserves, principally those in the Central Florida Phosphate District, are being depleted. Potentially economic resources and reserves are located in the southern extension of the central district. In this report, the southern extension of the Central Florida Phosphate District is considered to be south of Hillsborough and Polk Counties. The phosphate matrices in these deposits have different characteristics, particularly lower P_2O_5 and higher MgO contents. Other important differences may include variations in the mineral composition and the amount of -150 mesh phosphatic clay, variations in gangue minerals and their intergrowths with the phosphate mineral, thicker overburden, etc. Current mining, beneficiation, reclamation and process technologies may have to be modified and/or new technologies developed to solve such problems so that the phosphate resources and reserves in the southern extension can be utilized in an economically and environmentally sound manner.

For these reasons a detailed characterization of their physical, chemical, and mineralogical properties is necessary prior to any development work on these deposits. The major objectives of this project were to conduct a laboratory investigation on core samples from different areas in the southern extension. This investigation is summarized as the :

(1) Determination of the quantity and quality of matrix and its sized products.

(2) Evaluation of the phosphate mineralogy and its relationship to flotation behavior.

(3) Examination of francolite/dolomite association and liberation characteristics.

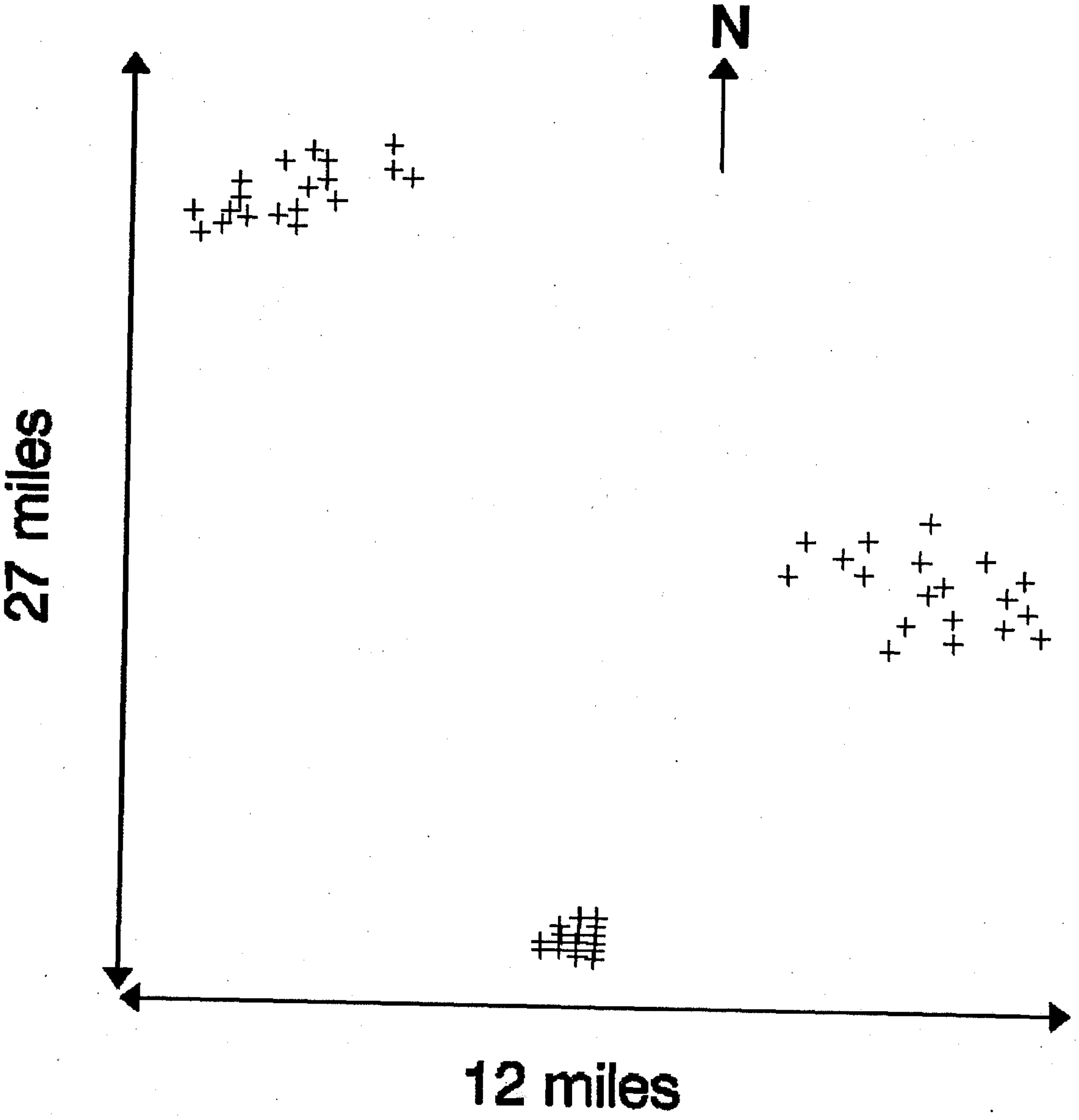
(4) Evaluation of the phosphatic clays chemistry and mineralogy and its effect on the settling and consolidation rates.

In order to achieve the above objectives, core samples were obtained from the northern (area A), central (area B), and southern (area C) parts of the southern extension of the Central Florida Phosphate District. Map 1 depicts the spacial distribution of the core sites. The study area lies south of Hillsborough and Polk counties and is 27 miles (45 kilometers) north to south and 12 miles (20 kilometers) west to east. About 80 splits from 20 core holes were obtained from each of the three participating phosphate companies. From each of these 80 splits about 15 splits were selected for more detailed studies. The “selected splits” were chosen to be somewhat representative of the area in regard to chemistry, mineralogy, proportions of matrix size fractions, and dolomite content. The splits with little or no dolomite are generally in the Upper Zone (UZ). Those splits containing higher dolomite are generally in the Lower Zone (LZ).

Different studies have been conducted depending on the type of sample (e.g. matrix, pebble, feed, etc.) as explained below:

MAP 1

Map of Locations



(1) Matrix. Perhaps the most important properties of the matrix include the weight distribution of its constituents (e.g. pebble, feed and phosphatic clays). Also, the fluidity of the pulped matrix is of prime importance to its pumping characteristics. Therefore, the following characteristics were determined: (a) % solids by weight, (b) weight distribution of different constituents, and (c) rheology as a function of % solids.

(2) Pebble Fraction (1/4" x 14 mesh):

(a) Mineralogical analysis using XRD and optical microscope
(b) Chemical analyses
(c) Size analyses
(d) Liberation studies using staining techniques

(3) Feed Fraction (14/150 mesh) Studies Included:

(a) Chemical analysis of head samples as well as different size fractions
(b) Size analyses
(c) Mineralogical analyses of head samples using XRD and optical Microscopy
(d) Concentration techniques such as froth flotation.

(4) Concentrate:

(a) Chemical and mineralogical analyses
(b) Empirical formulas

(5) Other Studies:

(a) Heavy mineral separations
(b) Trace element determinations

(4) Phosphatic Clays (-150 mesh) fraction:

(a) Chemical analyses
(b) Mineralogical analyses
(c) Centrifugal consolidation

RESULTS AND DISCUSSION

MATRIX

The average pebble and feed products in the Upper Zone consist primarily of francolite and quartz with little or no dolomite while the phosphatic clay contains minor dolomite. In the Lower Zone, dolomite is present in major amount in the pebble and phosphatic clay fractions, with only moderate amounts in the feed fractions. The only phosphate mineral observed in the three areas examined was francolite; no aluminum phosphate minerals were found.

The average weight percents and phosphate content for the sized matrix products from the Upper and Lower Zones in areas A, B, and C are shown in Table M-1.

Table M-1
Sized Matrix - Upper and Lower Zones

		Pebble			Feed			Phosphatic Clay		
		WT%	P_2O_5	BPL	WT%	P_2O_5	BPL	WT%	P_2O_5	BPL
Upper Zone										
Area	A	10	27	59	68	5	9	22	12	26
	B	8	28	63	70	8	17	22	10	22
	C	15	28	63	70	7	15	15	6	13
AVG		11	28	62	69	7	13	20	9	20
Lower Zone										
Area	A	10	16	39	51	8	17	39	2	4
	B	5	19	45	66	7	15	29	2	4
	C	9	16	35	57	7	15	34	2	4
AVG		8	17	40	58	7	15	34	2	4

Overall, the Upper Zone pebble constitutes 11% of the matrix and assays 28 % P_2O_5, while the Lower Zone pebble amounts to 8% of the matrix and analyzes 18% P_2O_5. In area B (central area), both zones contain less pebble product than the other areas.

The Upper Zone feed averages about 69% of the matrix and assays 6% P_2O_5. The Lower Zone feed averages about 58% of the matrix and analyzes 7% P_2O_5.

The phosphatic clay fraction of the matrix amounts to 20% in the Upper Zone and 34% in the Lower Zone. The Upper Zone phosphatic clay contains more phosphate, assaying about 9% P_2O_5 compared to only 2 % P_2O_5 in the Lower Zone phosphatic clay fractions.

Tests were made on slurries to simulate pumping characteristics of matrix samples. The torque exerted on an impeller rotating in slurries of matrix was determined. Torque measurements were made on samples with various percent solids from Upper and Lower splits representing each area. Generally, the power consumption (torque) increased exponentially with increases in percent solids content. However, the percent fines and clay minerals composition influence the power requirements.

PEBBLE

The three cooperating companies classified pebble according to their interpretation of the character of the matrix. All companies consider the pebble product to be coarser than about one millimeter (1mm) or about 14 mesh. The top size of the pebble fraction varied from 1/4 to l/2 inch. The oversize fraction, which is a very small percentage of the matrix, is considered as a waste product by all companies because of the high dolomite content. No further analyses were made on this oversize fraction.

In general, the Upper Zone (UZ) and Lower Zone (LZ) were differentiated by the amount of dolomite present in the pebble, expressed as %MgO. A pebble product containing more than 1% MgO was usually derived from the LZ; if it contained less than 1% MgO, it was nearly always derived from the UZ. If a pebble product assayed over 29% P_2O_5 and less than 0.5% MgO, there was essentially no free dolomite present; if the % MgO was greater, free dolomite was present.

Mineralogical and chemical analyses were made on all of the pebble products received from the three areas. More detailed analyses were made on pebble from selected splits to determine the quality of a product from which all of the free dolomite and non-francolite particles were removed by staining and hand sorting.

Description

The pebble from the Upper Zone is generally tan to brown to gray and is composed predominantly of francolite containing included, fine sand (mainly quartz). Some grains may contain minor amounts of dolomite as smears and/or inclusions. The pebble from a few splits did contain a minor amount of free, sugary dolomite aggregates plus quartz grains and some clay pellets.

The pebble product from the Lower Zone matrix is darker in color, usually brown and gray to black. Locked, fine sand and light-colored dolomite are common. Considerable dolomite is present as free, light-colored, sugary aggregates, often containing fine quartz. Some of the dolomite is gray, compact and fine grained and resembles phosphate pellets. Locally, free calcite and clay pellets are present.

Chemical Analyses

Table P-l lists the chemical analyses of the pebble fractions for the Upper and Lower Zones from each of the three areas. The major differences between the UZ and LZ pebble product are (1) considerably more MgO content (i.e. more dolomite), and (2) higher organic carbon content (contributing to a darker product color) in the LZ pebble. The UZ product averages 28% **P_2O_5**, 12% Insol, and 0.5 % MgO. This is equivalent to about 87% francolite, 12% quartz (minor feldspar), and minor amounts of dolomite. The LZ pebble averages 17% P_2O_5, 14% insol, and 6.2% MgO. This corresponds to about 60% francolite, 14% quartz (including minor amounts of feldspar, clay, and organic matter), and about 26% dolomite.

Ungraded Pebble Product

Samples of selected UZ and LZ pebble products were treated with Titan Yellow dye solution which imparts a red color to dolomite. This facilitated the manual removal of free dolomite grains and other visually identifiable non-francolite grains. The resulting upgraded pebble products were chemically analyzed with the results reported in Table P-2. A comparison can be made with corresponding analyses shown in Table P-1.

Table P-1

Average Chemical Analyses - Pebble Fractions from All Splits

	UPPER ZONE				LOWER ZONE			
	Area A	Area B	Area C	Avg	Area A	Area B	Area C	Avg
WT % Matrix	10	8	15	11	10	5	9	8
P_2O_5	27.0	28.0	28.5	27.8	16.0	19.0	16.1	17.0
CaO	40.0	42.0	43.4	41.8	37.0	36.0	34.5	35.8
MgO	0.45	0.48	0.58	0.52	6.39	4.24	7.04	6.19
Fe_2O_3	1.00	1.10	1.20	1.10	1.00	1.10	1.80	1.30
Al_2O_3	0.87	0.91	1.11	0.99	0.63	0.78	1.01	0.80
Na_2O	0.34	0.58	0.72	0.57	0.28	0.57	0.53	0.43
F	3.20	3.60	3.60	3.50	2.00	2.70	2.00	2.10
CO_2	NA*	4.50	4.90	---	NA	11.60	14.80	---
Org C	NA	0.92	0.71	---	NA	1.18	0.95	---
LOI	NA	6.80	7.40	---	NA	14.50	18.10	---
Insol	17.6	11.8	8.50	12.0	12.00	15.70	15.10	13.90
CaO/P_2O_5	1.48	1.50	1.52	1.50	2.31	1.89	2.14	2.11

Not Analyzed

Table P-2
Chemical Analyses - Selected Fractions
(Free Non-Francolite Particles Removed

	UPPER ZONE				LOWER ZONE			
	Area A	Area B	Area C	Avg	Area A	Area B	Area C	Avg
P_2O_5	30.3	29.8	29.9	30.0	26.9	25.0	26.7	26.2
CaO	45.2	45.9	45.7	45.6	42.7	40.7	42.5	42.0
MgO	0.52	0.43	0.47	0.47	1.20	1.20	1.40	1.30
Fe_2O_3	1.00	1.40	1.30	1.20	1.40	1.50	1.50	1.50
Al_2O_3	0.86	0.90	0.80	0.85	0.76	0.72	0.86	0.78
F	NA*	3.50	3.60	---	NA	3.20	3.20	---
CO_2	NA	4.10	4.90	---	NA	5.70	6.60	---
Org C	NA	1.05	0.84	---	NA	1.27	0.98	---
LOI	NA	NA	7.60		NA	NA	9.40	---
Insol	8.40	8.00	7.00	7.80	9.30	11.90	8.60	9.90
CaO/P_2O_5	1.49	1.54	1.53	1.52	1.59	1.63	1.59	1.60

* Not Analyzed

It can be seen that the average P_2O_5 content of the UZ pebble increased from 28% to 30%, while the insol dropped from 12% to 7.8%. The MgO content decreased slightly from 0.52% to 0.47%. A more dramatic change is shown in the results on the pebble from the Lower Zone matrix. The P_2O_5 increased from 17% to 26% while the insol dropped from 14% to about 10%. The MgO content dropped significantly from about 6% to about 1.3%. This remaining MgO reflects the locked dolomite in the Lower Zone pebble grains.

FEEDS

Flotation feed fractions prepared from all of the splits received were analyzed chemically and results are shown in Table F-1. These results are separately tabulated by area for the Upper and Lower Zones. For all the splits received, these data show that the Upper and Lower Zone matrix feeds are similar in phosphate content. The Lower Zone matrix, however, contains more dolomite as reflected in the higher MgO content and a higher CaO/P_2O_5 ratio.

Table F-1

Average Chemical Analyses - All Feeds

UPPER ZONE	Area A	Area B	Area C	Avg
WT% of Matrix	68	70	70	69
P_2O_5	4.60 %	7.60 %	6.80 %	6.30 %
CaO	7.00	12.60	10.20	9.90
MgO	0.10	0.13	0.14	0.12
Fe_2O_3	0.21	0.35	0.69	0.42
Al_2O_3	0.15	0.32	0.29	0.25
Insol	77.90	73.20	76.50	75.90
CaO/P_2O_5	1.52	1.66	1.50	1.57
LOWER ZONE				
WT% of Matrix	51	66	57	58
P_2O_5	8.20	6.60	6.70	7.20
CaO	14.30	12.20	11.40	12.60
MgO	0.75	0.48	0.79	0.67
Fe_2O_3	0.42	0.39	1.08	0.63
Al_2O_3	0.24	0.25	0.24	0.24
Insol	61.90	73.10	72.80	69.30
CaO/P_2O_5	1.74	1.85	1.70	1.75

Size distributions were obtained on all feed samples received from the three areas. The results are shown in Table F-2.

Table F-2
Size Distribution - Average of All Feeds

	Area A		Area B		Area C	
	Upper	Lower	Upper	Lower	Upper	Lower
16 x 28	7%	7%	9%	7%	5%	5%
28 x 35	10	10	9	8	10	11
35 x 48	27	23	20	22	20	20
48 x 65	31	26	26	33	28	28
65 x 100	9	11	24	22	26	24
- 100	16	23	12	8	11	12
	100	100	100	100	100	100

The average size distributions for the Upper and Lower matrix for area B (central) are similar; the same is true of area C (southern). The feed from the Lower matrix of area A (northern), however, tends to be slightly finer than that from the area A Upper matrix.

Size versus assay analyses were determined on selected feed samples from Upper and Lower Zones of all three areas. These are shown in Table F-3. It shows that the phosphate and dolomite content tend to be higher in the coarser size fractions of both the Upper and Lower matrix zones in all three areas.

Table F-3
Size vs. Assay Analyses - Feeds from Selected Holes

AREA A

	Upper Zone					Lower Zone				
	WT%	P_2O_5	CaO	MgO	Insol	WT%	P_2O_5	CaO	MgO	Insol
+ 28	8	15.1	22.3	0.22	46.1	9	15.6	30.1	1.67	26.7
28 x 35	11	8.2	12.3	0.13	69.4	14	12.3	22.5	1.09	46.0
35 x 48	26	4.9	6.6	0.06	75.5	26	10.3	17.8	0.67	59.4
48 x 65	28	4.2	5.2	0.03	80.5	20	7.5	18.1	0.51	65.8
65 x 100	10	3.1	4.8	0.04	81.5	7	6.4	11.3	0.43	57.6
- 100	17	3.8	5.4	0.04	80.1	23	8.3	14.0	0.53	62.8
	100	5.5	7.7	0.07	75.3	99	9.6	17.1	0.72	55.9

Table F-3 (continued)

Size vs. Assay Analyses - Feeds from Selected Holes

AREA B

	Upper Zone					Lower Zone				
	WT%	P_2O_5	CaO	MgO	Insol	WT%	P_2O_5	CaO	MgO	Insol
+ 28	9	23.6	36.0	0.36	25.1	6	18.8	30.9	1.18	31.9
28 X 35	9	17.4	26.0	0.20	53.8	9	11.5	18.4	0.51	58.8
35 X 48	17	10.2	15.9	0.13	69.6	25	8.0	13.4	0.34	69.9
48 X 65	27	7.0	11.0	0.10	79.3	32	6.5	10.8	0.21	75.9
65 X 100	26	5.5	8.5	0.08	82.1	21	6.9	10.9	0.20	75.4
- 100	12	6.3	9.3	0.10	79.3	7	5.4	9.1	0.23	79.1
	100	9.5	14.6	0.13	71.2	100	8.1	13.3	0.33	70.3

AREA C

	Upper Zone					Lower Zone				
	WT%	P_2O_5	CaO	MgO	Insol	WT%	P_2O_5	CaO	MgO	Insol
+ 28	6	18.1	27.6	0.39	39.4	3	16.4	27.5	2.07	36.1
28 X 35	13	12.9	19.9	0.27	57.0	7	10.5	17.4	1.09	58.7
35 X 48	27	7.3	11.2	0.14	75.4	17	7.4	12.0	0.58	71.6
48 X 65	26	5.9	9.0	0.12	79.8	26	5.6	9.0	0.37	79.2
65 X 100	20	4.2	6.4	0.09	85.6	31	4.3	6.8	0.26	84.0
- 100	8	3.0	4.6	0.08	89.2	16	2.7	4.7	0.44	88.1
	100	7.4	11.3	0.15	75.1	100	5.7	9.3	0.48	78.0

CONCENTRATES

Concentrates from the three area were prepared by a standard fatty acid fuel oil froth flotation process followed by an amine cleaner flotation process. Average chemical analyses for all the concentrates received from the three companies are on Table C-1. The area C, Upper Zone, is higher in MgO, Fe_2O_3, and Al_2O_3, and slightly lower in P_2O_5 than in areas A and B. In the Lower Zone, area C is higher in MgO (dolomite) and lower in P_2O_5 (francolite). The average MgO, CO_2, and LOI are all higher in the Lower Zone which indicates more dolomite, The LZ also contains less P_2O_5, indicating less francolite.

Table C-1
Concentrates - Chemical Analyses
Average Upper and Lower Zones

	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	F	CO_2	Insol	LOI	CaO/ P_2O_5
Upper											
A	32.2	47.5	0.37	1.3	0.79	0.62	3.7	ND	3.8	6.1	1.48
B	32.0	47.2	0.38	1.2	0.83	0.61	3.7	4.1	3.7	6.4	1.48
C	31.5	45.2	0.54	1.6	0.94	0.62	3.8	4.3	3.2	6.8	1.43
Avg	31.9	46.6	0.43	1.4	0.85	0.62	3.7	4.2	3.6	6.5	1.46
Lower											
A	28.5	46.4	1.11	1.3	0.64	0.67	3.6	ND	5.1	8.3	1.63
B	29.5	45.6	0.87	1.4	0.70	0.76	3.4	5.0	4.1	7.7	1.55
C	27.8	42.4	1.64	2.1	0.76	0.64	3.2	6.5	5.2	8.7	1.53
Avg	28.6	44.8	1.21	1.6	0.70	0.69	3.4	5.7	4.8	8.2	1.57

To put all the P_2O_5 analyses on a common basis, the average mineral content for the Upper and Lower Zones are listed, along with the calculated P_2O_5 contents of the concentrates at zero insol with zero dolomite.

Table C-2
Concentrate Mineralogy

	Actual Mineral Composition			P_2O_5	
Area	Francolite	Insol	Dolomite	Analyzed	Calc Zero Insol & Dolomite
Upper					
A	96%	4%	- %	32.2%	33.5%
B	96	4	-	32.0	33.2
C	97	3	Tr	31.5	32.5
Avg	96	4	Tr	31.9	33.1
Lower					
A	92	5	3	28.5	31.0
B	94	4	2	29.5	31.4
C	90	5	5	27.8	30.9
Avg	92	5	3	28.6	31.1

The P_2O_5 content of the Upper Zone concentrates is higher than the Lower Zone material at zero insol & dolomite. There may be a slightly lower P_2O_5 content in the Upper Zone area C concentrate than the area A & B samples. The P_2O_5 content at calculated zero insol and zero dolomite for concentrates from the Lower Zones for all three areas are similar.

The ratio of MgO/P_2O_5 in the concentrate is always lower than the MgO/P_2O_5 ratio in the feed (see table C-2 below). Table C-2 shows the rejection of MgO as a result of flotation. The last column of the table is the MgO concentration that would have been in the concentrate if no dolomite. rejection occurred during the beneficiation process. Comparison of the MgO content of the concentrate produced with the MgO expected from no loss during flotation indicates that in the flotation process, some dolomite is being removed to the tails and/or to the - 150 mesh fines. The average concentrate contained 1.21% MgO compared to the 2.66% MgO expected if no dolomite rejection occurred.

Table C-3

Effect of Beneficiation on Lower Zone

	FEEDS			CONCENTRATES			Calculated Conc. MgO
Area	P_2O_5	MgO	MgO/P_2O_5	P_2O_5	MgO	MgO/P_2O_5	(@ no rejection)
A	8.2	0.75	0.09	28.5	1.11	0.04	2.61
B	6.6	0.48	0.07	29.5	0.87	0.03	2.14
C	6.7	0.79	0.12	27.8	1.64	0.06	3.28
AVG	7.2	0.67	0.09	28.6	1.21	0.04	2.66

Empirical Formulas

Empirical formulas of the fluorapatite found in the three study areas were calculated from the results of the chemical analyses. Calculations were performed by the scheme described by Whippo and Murowchick, 1967. This scheme requires that ten cations are included in the francolite formula. Consequently the number of cations are normalized to 10. Concomitantly the number of anions required for the francolite formula is six anions plus two fluorine atoms. Anions were also normalized to meet this requirement. Table E-1 is a tabulation of the normalized numbers of cations and anions which were measured in the material from the three areas. Differences among the three areas are minor and are considered insignificant. Differences between the upper and lower matrix are also minor. It can be concluded from the data in table E-1 below that the crystallites of francolite found in the three study areas have very similar composition and probably the same diagenetic origins. The formula for the upper zone material has a lower PO_4 content than the formulas reported by Whippo and Murowchick, 1967 and also reported by Lehr. Lower PO, content and lower CaO content are accompanied by increases in the ions substituting for them. Magnesium substitution in the crystal lattice seems be higher in the three study areas. Lehr did not report a sulfate contribution to the anion balance and this may explain part of the difference in PO_4 between these results and his.

Table E-1
EMPIRICAL FORMULAS

	Number of Atoms in formula							Charges		Total Cat	Total An
	Ca	Mg	Na	PO_4	CO_3	SO_4	F	+	-	ions	ions
Upper											
A	9.68	0.10	0.22	4.89	0.95	0.16	2.00	19.8	19.0	10	8.0
B	9.62	0.08	0.30	4.71	1.09	0.20	1.90	19.7	18.6	10	7.9
C	9.58	0.15	0.27	4.80	1.10	0.10	2.19	19.7	19.0	10	8.2
Average	9.63	0.11	0.26	4.80	1.05	0.15	2.03	19.7	18.9	10	8.0

	Number of Atoms in formula							Charges		Total Cat	Total An
	Ca	Mg	Na	PO_4	CO_3	SO_4	F	+	-	ions	ions
Lower											
A	9.61	0.13	0.25	4.67	1.10	0.23	2.06	19.8	18.8	10	8.1
B	9.59	0.08	0.33	4.70	1.07	0.20	1.87	19.7	18.5	10	7.8
C	9.58	0.16	0.28	4.84	1.02	0.14	1.83	19.7	18.3	10	7.8
Average	9.59	0.12	0.29	4.74	1.09	0.19	1.92	19.7	18.5	10	7.9

Average Francolite Formula in Upper Zone

$$Ca_{9.63}\,Mg_{0.11}\,Na_{0.26}\,(PO_4)_{4.80}\,(CO_3)_{1.05}\,(SO_4)_{0.15}\,F_{2.03}$$

Average Francolite Formula in Lower Zone

$$Ca_{9.59}\,Mg_{0.12}\,Na_{0.29}\,(PO_4)_{4.74}\,(CO_3)_{1.09}\,(SO_4)_{0.19}\,F_{1.92}$$

Heavy Liquid Separations

Heavy liquid separations at specific gravity of 2.75 were made in an attempt to separate the dolomite from the francolite in concentrates from the Lower Zone of area A and in pebble splits of area B. Separations were poor. The sink fractions had excessive dolomite and the floats relatively high francolite content. These results reflect the fact that many grains consist of a phosphatic dolomite of varying composition and the response of the individual grains to the heavy liquid represented the relative composition of each grain. Additional tests were made on the pebble using specific gravity of 2.60. These separations were also poor because of the phophatic dolomite. Tests were not made on products from area C as further work did not seem warranted.

OTHER STUDIES

Heavy Mineral Separations

Heavy mineral separations were made at Sp. Gr. 2.95 on composite feeds, amine tails (AT) rougher tails (RT), and concentrates from the Upper and Lower Zones. Results are listed below.

Table H-1
Distribution of Heavy Minerals

	UPPER				LOWER			
	Feed	Conc.	AT	RT	Feed	Conc	AT	RT
% of Feed	100	20	4	76	100	27	5	68
% HM in Fraction	0.18	0.31	2.2	0.03	0.13	0.31	0.60	0.03
% Distr. HM	100	34	53	13	100	64	20	16
% Valuable HM								
Ilmenite-Rutile		20	10			30	20	
Zircon		10	5			5	5	
Total		30	15			35	25	

In both the Upper and Lower Zones, the heavy minerals are recovered in the amine tailings and concentrates. The percentage of heavy minerals in the feed and rougher tailings preclude consideration of recovery from these fractions.

Based on a recovery plant producing one million tons of concentrate there would be about 200,000 tons of amine tailings available for recovery of heavy minerals. Calculations for the amount of heavies available from the 200,000 tons of amine tailings and 1,000,000 tons of concentrates are listed for the Upper and Lower Zones.

The tonnages of total heavy minerals and valuable heavy minerals in the amine tailings is relatively low, and these are listed at 100% recovery. The separation and recovery of the heavy minerals from the amine tailings is not economically feasible at this time.

Recovery of heavy minerals from concentrates might offer some potential if the heavy minerals could be recovered from a wet process plant without gypsum or other diluents. This product would be relatively high in heavy minerals and should contain little francolite which confounds the recovery of the heavy minerals by density separation techniques.

Table H-2
Potential Heavy Mineral Recovery

	Amine Tailings				
				Valuable HM	
	Tons	% HM	Tons HM	%	Tons
Upper	200,000	2.2	4400	15	660
Lower	200,000	0.6	1200	25	300
	Concentrates				
Upper	1,000,000	0.3	3000	30	900
Lower	1,000,000	0.3	3000	35	1050

Trace Elements

For comparison with other studies of the central florida phosphate region trace. metals were determined in material from the three study areas. Composites of pebble and flotation concentrate from the upper zone and the lower zone were made for each of the study areas. Analysis for thirteen trace elements were performed, and the results are tabulated in table T-1.

Table T-1 reveals the similarity of the trace element composition of the products from the three areas. Other published data on trace elements found in phosphate products from the central florida phosphate region and north florida can be compared. These materials are all similar to the products from the southern extension described here.

TABLE T-1
CHEMICAL ANALYSIS OF COMPOSITES

Weight Percent

	Area A				Area B				Area C			
	Pebble		Conc.		Pebble		Conc.		Pebble		Conc.	
	U	L	U	L	U	L	U	L	U	L	U	L
AR Insol	13.7	11.3	2.4	8.6	17.1	17.3	6.0	5.0	7.0	17.2	5.0	7.0
P_2O_5	29.0	19.1	32.6	27.9	26.7	21.6	30.8	29.7	32.4	20.1	33.2	30.8
CaO	41.9	38.3	47.4	43.2	41.2	37.3	45.1	44.9	54.9	37.3	49.5	49.5
MgO	0.45	5.4	0.37	1.0	1.2	2.9	0.55	0.76	0.81	4.1	0.74	1.1

AR Insol = Aqua Regia insoluble

TABLE T-1
CHEMICAL ANALYSIS OF COMPOSITES

Values below are in PPM

	Area A				Area B				Area C			
	Pebble		Conc.		Pebble		Conc.		Pebble		Conc.	
	U	L	U	L	U	L	U	L	U	L	U	L
U_3O_8	133	115	113	103	136	103	121	100	83	71	78	66
CdO	14	14	11	11	16	14	20	16	2	2	3	3
As_2O_3	10	39	17	19	10	54	10	46	5	38	21	37
Y	50	36	148	146	32	34	128	148	24	22	108	138
Gd	8	6	16	22	8	8	22	18	4	6	18	22
La	30	24	96	90	24	26	80	92	-	-	6	8
Ce	42	30	150	128	34	32	126	134	26	28	140	162
Cr	38	30	56	46	42	28	56	44	40	42	48	54
Zn	42	30	32	48	34	44	42	54	20	22	30	32
Cu	18	8	22	2	21	28	18	26	11	9	10	7
Ag	below detection limit of 1 PPM											
Pb	46	44	50	44	46	40	46	50	60	50	60	80
Ba	106	1400	2200	1840	1460	780	168	1640				

PHOSPHATIC CLAY

Mineralogical and chemical analyses were made on the -150 mesh phosphatic clay samples from the Upper and Lower Zones. The amount of -150 mesh phosphatic clay in the matrix splits from the three areas varied from 15 to 22 % in the Upper Zone and from 29 to 39 %

in the Lower Zone samples. These phosphatic clay contents are similar to those reported in U.S.G.S Bulletin 1914 from matrix mined within the central Bone Valley region. Table PC-1 presents the overall chemical analyses and weight % of matrix based on the average analyses of all splits from each of the three areas. These data show higher P_2O_5 and lower MgO contents in the Upper Zone than is found in the Lower Zone clay fines. $\mathbf{P_2O_5}$ values in the phosphatic clay fractions are slightly lower than those reported in the past because of higher dolomite content.

The clay minerals in the -150 mesh fraction occur primarily in the - 2 micron fraction. For this reason, the -150 mesh fines were further sized by differential settling at 2 microns. The +2 and -2 micron fractions were analyzed with chemical results shown in Table PC-2. The mineral composition of these fractions is shown in Table PC-3, which are based on chemical, x-ray diffraction and optical microscopy.

In this report the mineral group name smectite is used. It includes the clay mineral montmorillonite. For practical purposes montmorillonite and smectite are essentially the same for Central Florida phosphatic clays. Palygorskite is used instead of attapulgite; mineralogically they are similar.

Upper Zone

The +2 micron fraction consists mainly of francolite and quartz with minor dolomite in the fractions from areas A and C, and major dolomite in the fraction from area B. Trace amounts of kaolinite and illite/mica may be present. This fraction averages 40% by weight of the -150 mesh material, and 8% of the Upper Zone matrix.

The -2 micron fraction contains major to moderate amounts of francolite and smectite, moderate to minor amounts of quartz; kaolinite and illite/mica, and minor amounts of dolomite. Illite/mica increases from trace amounts in the north area to moderate amounts in the south. This fraction averages 60% of the -150 mesh phosphatic clay fines, and 12% of the Upper Zone matrix.

Lower Zone

In the +2 micron fraction, dolomite is the dominant mineral present in all three areas. Moderate to major amounts of quartz, and minor to moderate amounts of francolite are also present. Minor amounts of palygorskite and a trace of smectite were noted in area B. The +2 micron fraction averages 66% by weight of the -150 mesh fines, and 22% of the matrix in the Lower Zone.

The -2 micron fraction contains minor to moderate amounts of francolite and quartz in all three areas. Dolomite is a major constituent in the south (area C) and is minor in areas A and B. Smectite is a major constituent in all three areas and is the major clay mineral present. Sepiolite and illite/mica were present in moderate amounts in area C, but occur only as traces in areas A and B. Palygorskite is present in major to moderate amounts in the -2 micron fraction in areas A and B, but only minor amounts were present in the southern area examined. Minor amounts of kaolinite are present in area C, and traces were noted in areas A and B. The -2 micron fraction averaged 34% by weight of the phosphatic clay fines and 12 % of the matrix of the Lower Zone.

In summary, francolite is the major component of the Upper Zone - 150 phosphatic clay fraction. Quartz, smectite, and kaolinite are present in moderate amounts. Dolomite and illite/mica contents are minor. The -150 mesh fraction averages 20% of the matrix weight in the Upper Zone.

The dominant component in the Lower Zone is dolomite. Moderate amounts of quartz, smectite, and palygorskite are present. Minor amounts of francolite, illite/mica, and sepiolite are locally present. The -150 mesh fraction averages 34% by weight of the matrix in the Lower Zone.

Table PC-1

Chemical Analyses - Phosphatic Clays (-150)

All Splits - All Areas

		WT% of Matrix	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	LOI	Insol
Upper	A	22	11.8	18.4	1.9	1.2	3.5	N.A.	42.3
	B	22	8.3	14.2	2.1	1.4	4.0	16.6	44.7
	C	15	6.1	13.2	1.7	1.9	5.6	16.7	51.4
Avg		20	8.7	15.3	1.9	1.5	4.4	16.7	46.1
Lower	A	39	2.5	21.8	11.5	0.6	1.0	N.A.	25.2
	B	29	2.5	17.4	10.0	1.1	1.6	29.7	31.6
	C	34	1.7	20.3	13.0	1.3	1.5	29.9	30.6
Avg		34	2.2	19.8	11.5	1.0	1.4	29.8	29.1

Table PC-2

Chemical Analyses - Selected Samples Phosphatic Clays
All Three Areas

		WT% of -150 mesh	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	CO_2	LOI	Insol*
+ 2 μm										
Upper	A	54	9.5	14.0	1.0	1.6	5.9	1.7	16.7	45.5
	B	29	11.8	24.5	5.3	1.3	2.6	13.3	16.7	33.1
	C	36	11.3	19.2	2.4	1.5	2.9	N.A.	12.0	45.3
Avg		40	10.9	19.2	3.0	1.5	3.8	7.5	15.1	41.3
Lower	A	78	3.0	N.A.	4.2	N.A.	N.A.	N.A.	22.8	54.2
	B	46	2.1	22.9	14.5	1.7	1.1	31.7	33.7	20.6
	C	74	0.6	26.6	19.7	1.2	0.5	39.7	39.7	12.4
Avg		66	1.9	24.8	12.8	1.5	0.8	35.7	32.1	29.1
- 2 μm										
Upper	A	46	9.6	13.5	1.6	2.5	14.0	N.A.	N.A.	27.3
	B	71	7.4	11.2	3.6	4.2	14.8	0.8	17.6	38.7
	C	64	3.3	4.7	1.9	3.0	19.7	1.6	19.0	41.9
Avg		60	6.8	9.8	2.4	3.2	16.2	1.2	18.3	36.0
Lower	A	22	2.9	5.2	4.5	3.0	12.1	N.A.	N.A.	36.0
	B	54	2.0	5.4	9.9	3.7	10.2	1.0	21.7	48.0
	C	26	1.4	2.3	3.7	3.4	16.9	1.6	20.7	46.6
Avg		34	2.1	4.3	6.0	3.4	13.1	1.3	21.2	43.5

* Determined by aqua regia digestion

Table PC-3
Mineral Composition - Selected Samples - Phosphatic Clays
All Three Areas

			WT% of -150 mesh	Francolite	Dolomite	Quartz	Smectite	Palygorskite	Sepiolite	Illite/ Mica	Kaolinite
UPPER											
A	+	2 μm	54	Maj	Min	Maj					
	-	2 μm	46	Maj	Min	Min	Mod	Tr		Tr	Mod
		Avg	100	Maj	Min	Maj	Mod	Tr		Tr	Mod
B	+	2 μm	29	Maj	Maj	Maj	Tr				
	-	2 μm	71	Maj	Min	Min	Maj	Min	Tr	Min	Min
		Avg	100	Maj	Mod	Mod	Mod	Min	Tr	Min	Min
C	+	2 μm	36	Maj	Min	Maj	Min			Min	Min
	-	2 μm	64	Mod	Min	Mod	Mod		Tr	Mod	Mod
		Avg	100	Mod	Min	Mod	Mod		Tr	Min	Mod
Avg	+	2 μm	40	Maj	Mod	Maj	Tr			Tr	Tr
	-	2 μm	60	Maj	Min	Mod	Mod	Tr	Tr	Min	Mod
			100	Maj	Min	Mod	Mod	Tr	Tr	Min	Mod

Table PC-3

Mineral Composition - Selected Samples - Phosphatic Clays
All Three Areas

			WT% of -150 mesh	Francolite	Dolomite	Quartz	Smectite	Palygorskite	Sepiolite	Illite/ Mica	Kaolinite
LOWER											
A	+	2 μm	78	Min	Dom	Maj					
	-	2 μm	22	Mod	Min	Min	Maj	Mod	Tr	Tr	Tr
		Avg	100	Min	Dom	Maj	Mod	Min	Tr	Tr	Tr
B	+	2 μm	46	Mod	Dom	Mod	Tr	Min			
	-	2 μm	54	Mod	Min	Mod	Maj	Maj	Tr	Tr	Tr
		Avg	100	Mod	Maj	Mod	Mod	Mod	Tr	Tr	Tr
C	+	2 μm	74	Min	Dom	Mod					
	-	2 μm	26	Min	Maj	Min	Maj	Min	Mod	Mod	Min
		Avg	100	Min	Dom	Mod	Mod	Min	Min	Min	Min
Avg	+	2 μm	66	Min	Dom	Mod	Tr	Tr			
	-	2 μm	34	Mod	Mod	Mod	Maj	Mod	Min	Min	Min
		Avg	100	Min	Dom	Mod	Mod	Mod	Min	Min	Tr

Trace (Tr)	< 1%
Minor (Min)	0-5
Moderate (Mod)	5-20
Major (Maj)	15-60
Dominant (Dom)	+50

CONSOLIDATION BEHAVIOR

Laboratory centrifuge tests were made on the -150 mesh fraction of the matrix in order to predict settling characteristics. These centrifuge tests were made on Upper and Lower Zone samples, from all three areas.

Models of the following form are developed to represent the consolidation behavior:

$$e_t = e_{in} \exp\text{-}kt + e_{in}{*}\ A\sigma^{-B}[1\text{-}\exp(\text{-}kt)]$$

$$k = F{*}N{*}e_{in}{}^{G}$$

$$k = CNe^{D}$$

where:

e_t = void ratio at time t

e_{in} = initial void ratio

t = elapsed time in centrifuge, minutes

k = permeability, ft/day

It is worthy to mention that:

(a) The consolidation test can be carried out in less than 4.0 hours (conventional techniques need between 3-4 weeks to complete a test).

(b) The parameters A,B,C and D have comparable values to that developed by conventional techniques.

(c) In the centrifuge up to six samples can be tested at the same time.

Using the void ratio model for an initial height of 1.0 ft. (30.5 cm), e_{in} = 20.0 and N = 30g, plots of height of clays vs. centrifuge time are generated. The data indicate that there is no consistent behavior of clays. In other words, some lower matrix phosphatic clays (clays) consolidate faster than the top cores. In order to explain these results, the mineralogical content of each of the studied splits are compared. These preliminary data indicate that slower rates of consolidation are attributable to a higher content of smectite and/or palygorskite clay. In addition, phosphatic clays containing palygorskite are slower settling than the ones containing other clay minerals.

Using the permeability models for a pond 30 feet high (corresponding to 1.0 ft. model height and N = 30g), the consolidation behavior (% solids and height) is calculated as a function of time in years. It is important to note that these data correlate well with the plots from the centrifugal times.

Generally, the slowest settling (least compaction) occurred in samples containing palygorskite; those with smectite (montmorillonite) were also slow settlers. The fastest settling occurred in samples high in dolomite. Mixtures of dolomite with palygorskite and/or smectite gave mixed results.

SUMMARIZING REMARKS

The available high grade phosphate reserves in the Central Florida region are being depleted. Core samples from the northern (Area A), central (Area B), and southern (Area C) parts of the southern extension of the Central Florida district were studied to determine differences in the ore character that will affect the mining and processing of these potentially valuable deposits.

The matrix in the areas studied can be divided into an Upper Zone and a Lower Zone, based upon the MgO content of the pebble. The Upper Zone pebble contains less than 1% MgO and where present, overlies the Lower Zone, where the pebble contains more than 1% MgO. A summary of the data is shown in Table S-1.

Upper Zone

The Upper Zone matrix was present in all of the cores of Area A, whereas in areas B and C, the Upper Zone matrix was present in the extent of 50% and 58% of the respective cores. Overall, the Upper Zone overburden thickness averages 21 feet, and matrix thickness 14 feet. The product composition of the Upper Zone matrix averages about 11% pebble, 69 % feed, and 20% -150 mesh phosphatic fines. Concentrate content averaged 14%. Tonnage of the pebble product averaged 2900 TPA (tons per acre) or 210 TPAF (tons per acre-foot). Grade was 62% BPL, 9% Insol, and 0.5% MgO. Concentrate averaged 3600 TPA (260 TPAF) with a grade of 70% BPL, 4% Insol, and 0.4% MgO. The combined product consists primarily of the phosphate mineral francolite, containing between 5 and 10% acid insoluble, essentially all quartz, plus traces of dolomite. No wavellite or other typical leached zone minerals were noted.

The -150 mesh phosphatic clay fraction consists mainly of francolite and clay minerals (montmorillonite, smectite, kaolin and traces of palygorskite) with moderate amounts of quartz. Minor quantities of dolomite and mica/illite are present. Locally in the southern extensions, a phosphate-bearing unit containing dolomite and/or shell fragments overlies the matrix.

Lower Zone

The Lower Zone matrix was present in most of the core holes drilled in the Southern Extension. The top of the Lower zone matrix occurs at an average depth of about 33 feet, and the average thickness of the Lower Zone matrix is 27 feet. The matrix thickness varied from 34 feet in the northern area, and 32 feet in the southern area, to only 16 feet in the central part of the Southern Extension. The Lower Matrix averages 9% pebble, 56% feed, and 35% -150 mesh phosphatic fines. Pebble tonnage averaged 4900 TPA (180 TPAF) at a grade of 39 % BPL, 6.1 % MgO, and 9% insol. Concentrate tonnage averaged 6800 TPA (250 TPAF) with a grade of 62% BPL. 1.2% MgO, and 4% Insol.

The pebble product contains about 30% dolomite and cannot be utilized with current technology which requires an MgO content of less than 1% . The concentrate product which is primarily francolite, contains about 4% insol (essentially all quartz) and about 3% dolomite.

Minor amounts of calcite are locally present in the Lower Zone matrix.

In general, the pebble and concentrate products from the Upper Zone matrix are suitable for commercial use. In the Lower Zone matrix, however, the dolomite content in the pebble product must be reduced to less than 5% before it can be considered commercial. The Lower Zone concentrate is marginal as far as MgO content is concerned. It might be utilized commercially after further reduction of dolomite by mildly acidic attrition scrubbing, or other simple technology.

TABLE S-1

		UPPER ZONE				LOWER ZONE			
		Area A	Area B	Area C	Avg	Area A	Area B	Area C	Avg
Core Hole Data	A*	19	20	19	58	19	20	19	58
	B*	19	10	11	40	17	18	16	51
Depth to Top of Matrix (ft)		23	15	25	21	34	36	30	33
Matrix Thickness (ft)		11	17	13	14	34	16	32	27
Pebble	WT%	10	8	15	11	10	5	9	9
	TPA	2600	2300	3800	2900	7800	1600	5400	4900
	TPAF	240	130	290	210	230	100	170	180
	BPL%	59	63	63	62	39	45	35	39
	P_2O_5%	27	29	29	28	18	21	16	18
	MgO%	0.5	0.5	0.6	0.5	6.4	4.2	7.0	6.1
Feed	WT%	68	70	70	69	51	66	57	56
	BPL%	9	17	15	14	17	15	15	16
	P_2O_5%	4	8	7	7	8	7	7	7
-150 Mesh Fines (%)		22	22	15	20	39	29	34	35
Conc.	WT%	7	19	12	14	14	16	11	13
	TPA	1400	6300	3000	3600	8800	5000	7200	6800
	TPAF	130	370	230	260	260	300	220	250
	BPL%	70	70	69	70	63	66	61	62
	P_2O_5%	32	32	31	32	29	30	28	29
	MgO%	0.4	0.4	0.5	0.4	1.1	0.9	1.6	1.2

A* - Number of holes drilled
B* - Number of holes in which matrix was encountered

APPENDIX I

METHODS and TECHNIQUES

The following examinations were made on the samples received as indicated:

1. MATRIX - Rheology on Unground Selected Splits
   - A. Size analysis, P_2O_5, and tons per acre foot
   - B. Rheology on unground selected splits
2. PEBBLE (-¼" x 14 mesh)
   - A. Unground Selected Splits
     - 1) Mineralogy on as-received samples
     - 2) Liberation by staining and hand sorting
     - 3) Chemical and X-ray diffraction analysis on 1) and 2)
   - B. Ground - Chemical and X-ray Diffraction Analysis on all splits received
3. FEED (14 x 150 mesh)
   - A. Unground
     - 1) Mineralogy on selected splits
     - 2) Size analysis on all splits received
     - 3) Chemical analysis on sized fractions of selected splits
   - B. Ground - Chemical and X-ray Diffraction Analysis on all splits received
4. CONCENTRATES
   - A. Unground selected splits
     - 1) Mineralogy
     - 2) Flotation
   - B. Ground Splits
     - 1) Chemical and X-ray diffraction analysis on all splits
     - 2) Detailed chemical analysis on selected splits
5. HEAVY -MINERAL STUDIES - Separation and mineral analysis of heavy mineral fraction from composite feeds of Upper and Lower Zones.
6. PHOSPHATIC CLAYS t-150 mesh)
   - A. From Unground Matrix Selected Splits
     - 1) Mineralogical, chemical, and X-ray diffraction analysis on fractions sized at 100 x 44, 44 x 2, and minus 2 microns.
     - 2) Rheology
   - B. Ground - Chemical and X-ray diffraction analysis on all splits

TECHNIQUES AND METHODS

Several different means of analysis were used in the study of these samples. These include conventional techniques such as chemical analysis, X-ray diffraction, infrared spectroscopy, optical microscopy, and upgrading the feed by froth flotation to remove both sand and dolomite. The non-conventional techniques used were rheology measurements on matrix and centrifugal consolidation tests on phosphatic clays.

The next few paragraphs list the standard and/or conventional techniques. More details, however, are given to the non-conventional methods.

A. Standard and/or Conventional Techniques

(1) CHEMICAL ANALYSES

Chemical analyses were performed with methods described in the Association of Florida Phosphate Chemists manual. Modifications were made to accommodate instrumentation available in the analytical chemistry laboratory at the Florida Institute of Phosphate Research. Reagents were ACS reagent grade and volumetric glassware was class A as required. Analytical results were monitored by analyzing AFPC certified Check 20, Check 21, and NBS 120b standards along with the project samples. Spikes were incorporated to monitor recovery of cations by the Inductively Coupled Argon Plasma (ICP) Spectrophotometer. Wet chemical methods were also used to monitor some of the results from the ICP.

Acid insoluble material was measured in two ways. An aqua regia insoluble material was determined as described in the AFPC manual. Aqua regia insoluble material includes only material that is not oxidizable such as clay, quartz, and feldspar. The second acid insoluble material was the material which was not soluble in HCl and includes the organic matter that was not readily hydrolyzable. Clays and feldspars were slightly soluble in the aqua regia and contributed to the soluble Al_2O_3 and silica.

Sulfur was determined as total S and as, sulfate S. Total sulfur was determined by Curie point pyrolysis and oxidation with oxygen then measurement with an iodometric titration (LECO sulfur analyzer). Sulfate was determined by $BaCl_2$ precipitation after hydrochloric acid digestion (from AFPC method). Both sulfur values were calculated to be reported as SO_3. Pyrite sulfur is the difference between the total value and the sulfate value.

Carbon from organic plus carbonate minerals was determined by pyrolysis and absorption of the evolved carbon dioxide on ascarite (LECO total carbon analyzer). Carbon dioxide in carbonate minerals was measured by ascarite absorption of the carbon dioxide evolved from 4% hydrochloric acid digestion of the sample. Organic carbon is determined by difference.

(2) X-RAY DIFFRACTION ANALYSIS

X-ray diffraction analysis was performed with a Phillips Electronics system employing APD 3720 software. Samples were ground to pass a 150 mesh sieve and random powder mounts were prepared by front filling aluminum sample holders then compressing the ground material with a glass microscope slide. Diffraction profiles were collected over the range of two to sixty degrees two theta. Analytical data were fitted to JCPDS PDF standard reference data. The most important concern was to correlate the analytical data from other sources to the mineralogy determined by x-ray diffraction.

Discrepancies were resolved by matching PDFs for the best statistical fit of the data. Mineralogy from the x-ray diffraction data and chemical data were compared and evaluated to determine the significance of differences. Most differences between the two methods are attributable to non-random crystal orientation contributions in the diffraction data. Dolomite and quartz had preferred orientations and cleavage which persisted after grinding. Quantification of these minerals from diffractiondata was not straight forward.

Phosphatic clay samples were taken from as received slurries. After drying an aliquot in a forced air oven at thirty degrees Celsius, the solids were ground by hand. Random powder mounts for X-ray diffraction were prepared by front filling an aluminum sample holder and compressing the material with a glass microscope slide. Reflections were collected over the range of two to sixty degrees two theta. Oriented mounts were prepared from sized clay obtained from the original phosphatic clay slurry. Sized clay was prepared from solids which had been dispersed with sodium hexametaphosphate then sized by Stokes settling in a column. A +44 micron fraction and a -44 micron fraction were obtained. Further settling of the -44 micron material yielded a +2 micron sample and a -2 micron sample. Oriented mounts were prepared by filtering each size fraction onto a 0.2 micron membrane filter then the clay was transferred from the membrane to a glass plate which was mounted in an aluminum sample holder. Reflections over the range of two to thirty degrees two theta were collected. After air drying, oriented mounts were glycolated before rerunning them to determine the expanded basal spacings. A separate oriented mount was used to determine the effect of heating at 350C, 550C on the basal spacings of the clays.

These data were used to identify the specific clay minerals Present in the original phosphatic clay.

(3) INFRARED SPECTROSCOPY

Diffuse reflectance with Kebelka-Munk corrections have been collected. Data for francolite. from the float concentrate product was collected as reference material. Comparison of the infrared data for samples collected from various depths was correlated with chemistry and x-ray diffraction data. Data collected with the classical KBr transmission technique was compared with the diffuse reflectance data to generate a library of spectra.

(4) OPTICAL MICROSCOPY

The pebble, feed, and concentrate fractions of the selected samples were examined using binocular and petrographic microscopy. Refractive index oils and birefringence were employed to dicriminate among the many minerals which comprise the mineral suite accompanying the francolite. The characteristic high birefringence of the carbonate minerals allowed dolomite to be detected at very low concentrations in the presence of the other minerals. Mineral stains, such as Titan Yellow for dolomite and Alizarin Red S for calcite, were used to discriminate the dolomite from francolite and aid in liberation studies. X-ray diffraction, chemical analyses, and optical microscopy were all used to confirm the identification of specific minerals and their quantities to arrive at a mineralogy of the selected samples.

(5) UPGRADING AND DOLOMITE REMOVAL

Froth Flotation - conventional double stage flotation was used in an attempt to upgrade the feed by removal of most of the unbound sand insol and some of the dolomite. The flotation results obtained by the FIPR lab were similar to results reported by the cooperating companies but obtained by the supplier of area C were better than FIPR was able to attain. The poorer results obtained by FIPR is attributed to the fact that the feeds had aged, and had lost some of their surface charges. Chemical analyses for all samples, including the special samples, were made from the ground concentrates obtained from the supplier of Area C material.

B. Non-conventional Techniques

(1) RHEOLOGY MEASUREMENTS

For this purpose, the torque exerted on a 4" propeller (30° pitched) in a cylindrical stainless steel tank (11.74 cm. diam. x 15 cm. high), was measured in a matrix pulp starting at the highest % solids followed by required dilutions. The propeller was activated at 1.5cm. off the tank's bottom. Readings were recorded at three different speeds (350, 400 and 450 rpms) respectively. The corresponding linear speeds are (1.86, 2.13, and 2.39 meter/sec) respectively. The obtained readings were then divided by the corresponding volume of the slurry to obtain torque/unit volume.

(2) CENTRIFUGAL CONSOLIDATION OF PHOSPHATIC CLAYS

Centrifugal force is used to enhance the settling and consolidation of phosphatic clays. Samples at solid contents corresponding to the compression zone (after the free settling zone) are transferred to plastic bottles (5.8 cm diameter, 12.5 cm high).

(a) The initial height is measured. The bottles are centrifuged at a predetermined speed for a known period of time. Then the height of interface (mud line between clay and the supernatant) is measured. Bottles are centrifuged again at the same speed for equal periods. This procedure is repeated five times.

(b) Then the speed is increased to a higher and predetermined level to give required level (Ng) where Ng equivalent number of gravity acceleration. The same previous procedure (a) is repeated at this level of Ng.

(c) The speed is increased again to a higher Ng and the procedure in (a) is repeated.

(d) The collected data will be height (H, cm) at different times, minutes, at different Ng. Initial % solids is also determined.

(e) Mathematical manipulation of the data:

i - Calculate e_t, (void ratio at any time t) using the equation:

$$e_t = (H_t/H_o)(1 + e_{in}) - 1 \qquad (1)$$

where:

H_t = height of interface at any time

H_o = height of interface at t = o

e_{in} = initial void ratio

ii - Considering the system as a chemical reaction of two components, namely water (voids) and solids. Thus, descending interface means voids or water is recovered from the system. Therefore, a first-order rate kinetics of water recovery may be considered.

This leads to the equation:

$$R = R_x [l - \exp - kt] \qquad (2)$$

where:

R = recovery of water (voids) = $\frac{e_{in} - e_t}{e_{in}}$ at time t

R_∞ = infinite recovery of water (voids) at $\frac{e_{in} - e_\infty}{e_{in}}$

R = rate constant

t = elapsed centrifugal time

iii - Methods to calculate the kinetic parameters:

- Plot R vs. t
- Interpolate values of R_i, R_{i+1}, R_{i+2} corresponding to t_i, t_{i+1}, t_{i+2} in arithmetic progression $t_{i+1} = t_i + t$ where t = constant increment and greater than 0.0
- Interpolation can be avoided if the centrifugation times are chosen as equal intervals say t_1 = 2 min, t_2 = 4 min, t_3 = 6 min, etc.
- Draw a unit slope line from the origin (45°)
- Plot R_i vs. R_{i+1}
- Fit the experimental data by using the least square method
- Extrapolate the fitted line to intercept the slope unit line at $R_i = R_{i+1} = R_\infty$
- Calculate $k = \frac{\ln \text{slope}}{\Delta t}$

Note that all the above procedures can be done using the pocket calculator which has statistical analysis programs.

iv - From R calculate e

v - For every loading (Ng) calculate σ = effective

stress and regress σ vs. e_∞ to obtain equation of the form

$$e_\infty = A_1{*}e^{-B} \qquad (3)$$

vi - Calculate $A = A_1/e_{in}$

Substitute in equation No. 3 to get $e_\infty = A{*}e_{in}{*}\text{-}B$ (4)

vii - For every loading (Ng) regress ${}^{k}/_{N}$ vs. e_{in} to get:

$$k = F * N * e^{G}_{in} \qquad (5)$$

viii - Calculate K (permeability) using the change in height for every change in time $= \dfrac{\Delta H * 47.2}{\Delta t * N}$

ΔH = cm
Δt = min. = $t_{i+1} - t_i$
N = number of g's
47.2 = factor for conversion to ft/day

ix - Regress K/N vs. e to get:

$$K = CNe^{D} \text{ ft/day} \qquad (6)$$

x - Substituting $R = \dfrac{e_{in} - e_t}{e_{in}}$, & $R_\infty = \dfrac{e_{in} - e_\infty}{e_{in}}$

in the equation (1):

$$R = R\infty\ [1 - \exp{-kt}] \text{ to get:}$$

$$e_t = e_{in} \exp{-kt} + e_\infty\ [1-\exp{-kt}] \qquad (7)$$

using equation (4) to substitute for

$$e = A\ {*}e_{in}\sigma^{-B} \text{ to get:}$$

$$e_t = e_{in} \exp{-kt} + e_{in}\ A\sigma^{-B}\ [1-\exp{-kt}] \qquad (8)$$

Data Handling (Data Base).

The mineralogy database is a collection of files using standard spreadsheet and database formats. The spreadsheets are used for initial data entry and manipulation, mass balances and ratios. Data is then transferred to data tables that render the data accessible to be tabulated, graphed, and further used. There are currently 21 data tables and about 25 spreadsheets. The data while in data tables has been used or can be used in the following ways:

(1) 2D or 3D graphs using one or more data tables

(2) Statistical calculations using one or more data tables

(3) Reports on raw or manipulated data

(4) Tabular data reports with or without summaries.

The computer software we are using is Knowledge Man. This software is one of the most powerful that is available and appears to have been designed for the scientist and engineer. The limits on the data and associated files are as follows:

(1)	Tables open at once	240
(2)	Records per table	2,137,483,647
(3)	Characters per record	65,535
(4)	Fields per record	255
(5)	Characters per field	65,534
(6)	Cells per spreadsheet	65,025
(7)	Observations per graph	Unlimited
(8)	Graphs per screen	15

These limits along with the smooth integration of spreadsheet and data table make this software extremely powerful. The actual limits to this database is the constraints the computer hardware places on us, <u>not</u> the software. FIPR has acquired an IBM Model 70-386 computer with an 120 MB hard disk to increase the amount of data capable of being handled. Our current data tables have on average 75 records per table with 20 fields per table. The spreadsheets have a similar status.

The software allows for menu driven data entry and requires some programming to retrieve data in any form other than tabular output. Some of the features have not been put to full use as of this data. Programming is continuing to increase the capability of the menu-driven portion of this database. There are presently 56 program files in use to extract this data. Not all of the program files are currently in the menu system.

Appendix II

Data for Area A

RESULTS AND DISCUSSIONS

Matrix

Product Content

Data obtained for the product content, e.g. pebble (+16 mesh), feed (16/150 mesh), and phosphatic clays (-150 mesh) expressed as tons/acre foot are shown in Appendix A. The data represent different splits from 17 drill holes. The % P_2O_5 content in these products is also given. The statistical averages of these data can be summarized as:

Product	Tons/acre foot	BPL%	Weight%
Pebble	92 (21-163)	47 (36-59)	6 (1-11)
Feed	1016 (778-1254)	15 (12-18)	67 (51-83)
Phosphatic clays	417 (253-581)	8 (1-16)	27 (16-38)

Detailed chemistry and mineralogy of each of these constituents are given in later sections of this report.

Rheology

Pumping of slurry depends on its rheological characteristics such as viscosity or fluidity which in turn depends on the solids content as well as on the mineralogical and physical properties of the solids.

For the purpose of investigating the difference in rheology of matrix slurries from the upper and lower zone material, the torque exerted on an impeller rotating in such slurries was measured. The obtained data for matrix slurries from different splits of several drill holes are given in Figures 1-6. The size distribution of the tested samples are also given for comparison.

Generally, the data indicate that energy consumption increases exponentially with the increase in the % solids content.

Results shown in Figure 1 suggest that more energy is needed to pump slurry from split

#5 than pumping slurry from split #1 . This could be attributed to the presence of more fines (-150 mesh) in split #5 as shown by the weight distribution of different size fractions. It is also worthy to mention that more smectite and palygorskite clays are found in the matrix as compared to the matrix from split #1.

The same argument can be applied to the matrix from holes #W as shown in Figure 2. On the other hand, more energy is needed to pump matrix of split #1 in hole G as compared to split #4 even though the weight distribution indicates presence of more fines in the lower matrix (#4) (see Figure 3). Examining the mineralogical data of the -150 mesh fraction suggests the presence of a larger quantity of montmorillonite in the upper matrix. In addition, the feed size distribution (16/150 mesh) tends to be finer than in the lower matrix. These two reasons could be responsible for the observed increase in energy. The same argument can be used to explain the data in Figure 5 for the matrix from hole #S.

Pebble

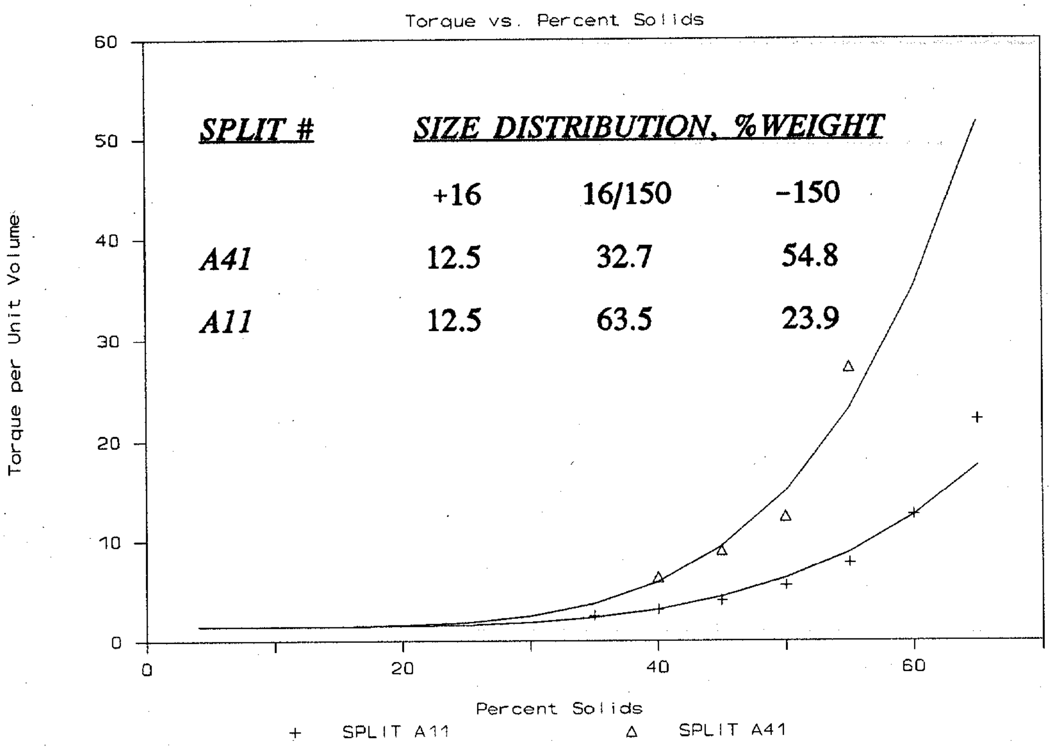
+3/8" Pebble Fractions

In the seven cores selected for examination, three samples of +3/8" pebble from the upper matrix and six samples of +3/8" pebble from the lower matrix were available for analysis.

The +3/8" pebble from the lower matrix splits was composed almost entirely of dolomite with minor included phosphate. Only two samples of upper matrix +3/8" pebble contained francolite. The +3/8" fraction from AC-1 consisted primarily of phosphatic pebble and minor free quartz. Only traces of dolomite were noted. The +3/8" fraction from AD-1 consisted of 50 % dolomite aggregates.

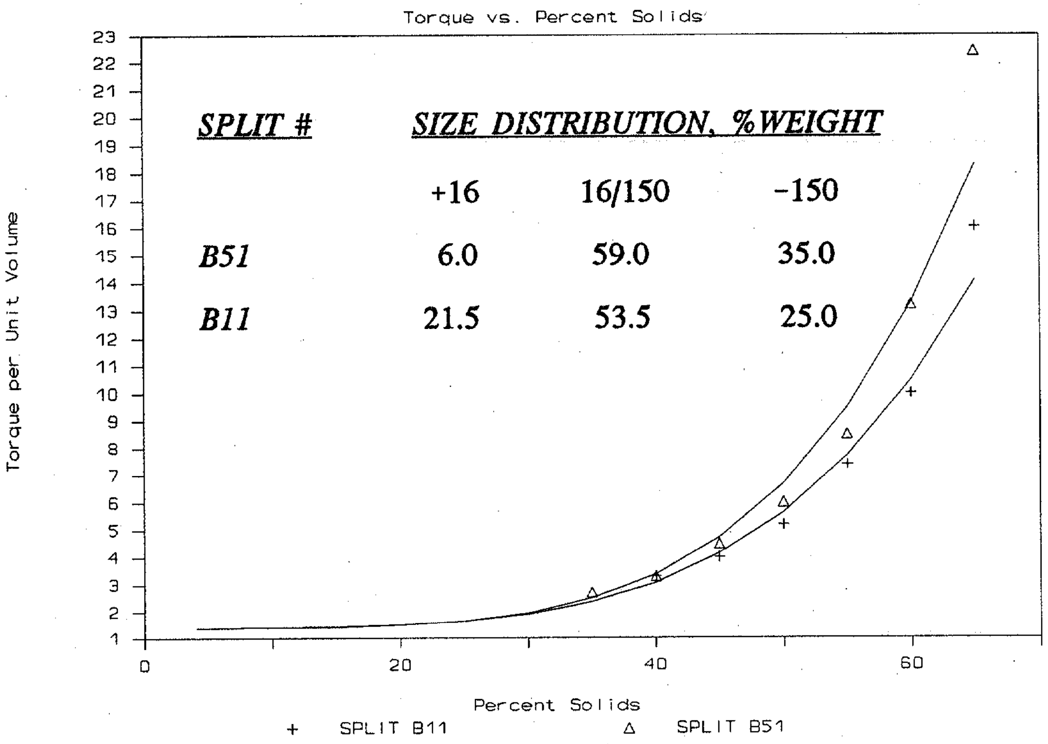
Chemical data are shown in Table P-I.

Figure 1



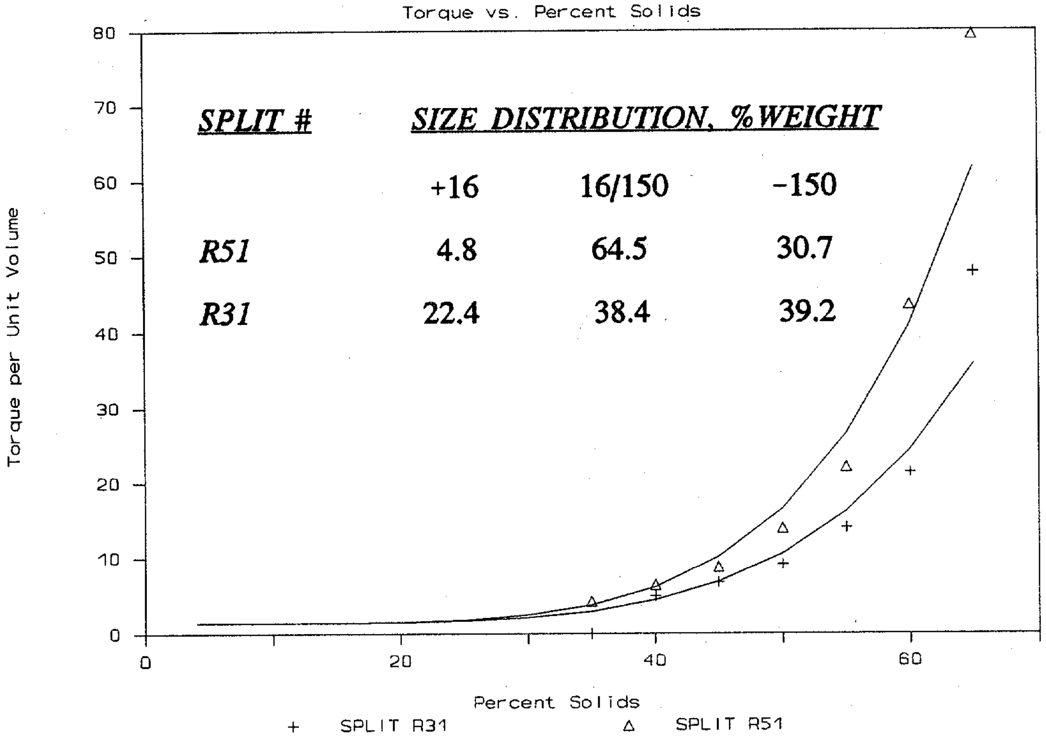
SPLIT #	***SIZE DISTRIBUTION, %WEIGHT***		
	+16	**16/150**	**-150**
A41	**12.5**	**32.7**	**54.8**
A11	**12.5**	**63.5**	**23.9**

Figure 2



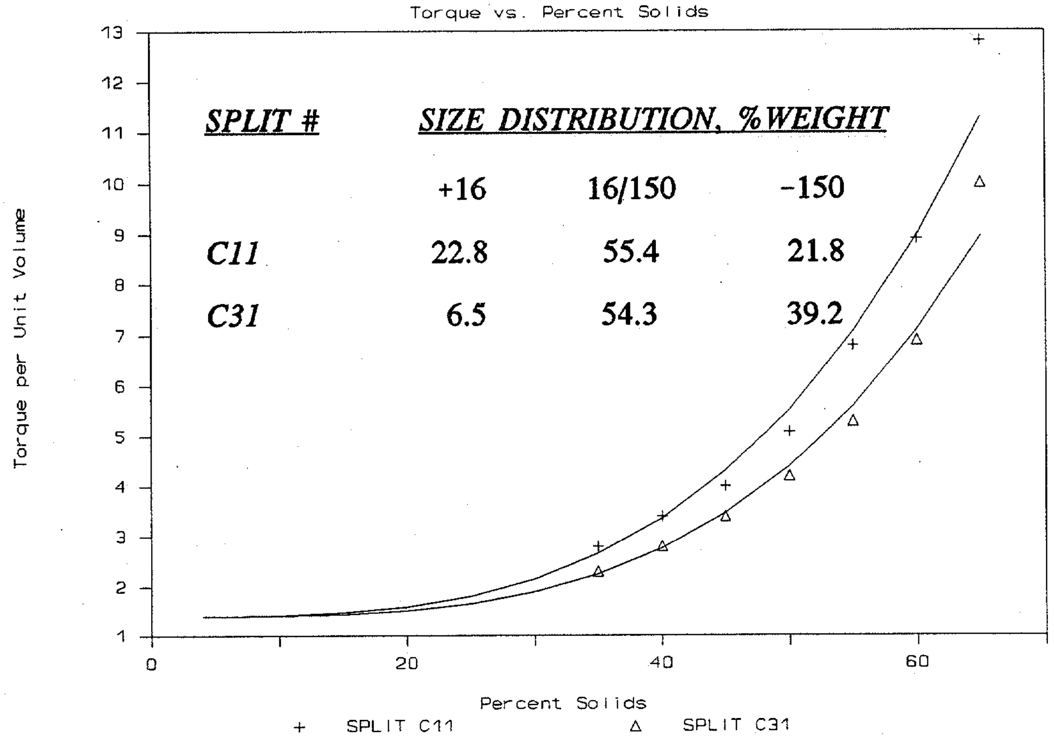
SPLIT #	***SIZE DISTRIBUTION, %WEIGHT***		
	+16	**16/150**	**-150**
B51	**6.0**	**59.0**	**35.0**
B11	**21.5**	**53.5**	**25.0**

Figure 3



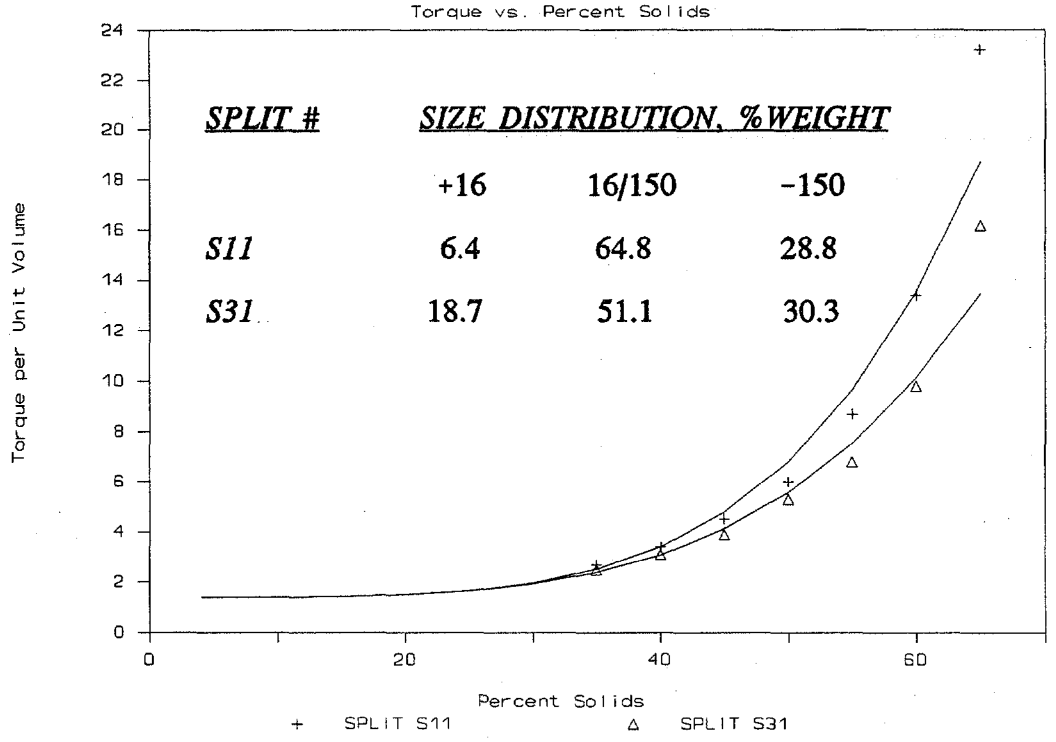
SPLIT #	SIZE DISTRIBUTION, %WEIGHT		
	+16	16/150	-150
R51	4.8	64.5	30.7
R31	22.4	38.4	39.2

Figure 4



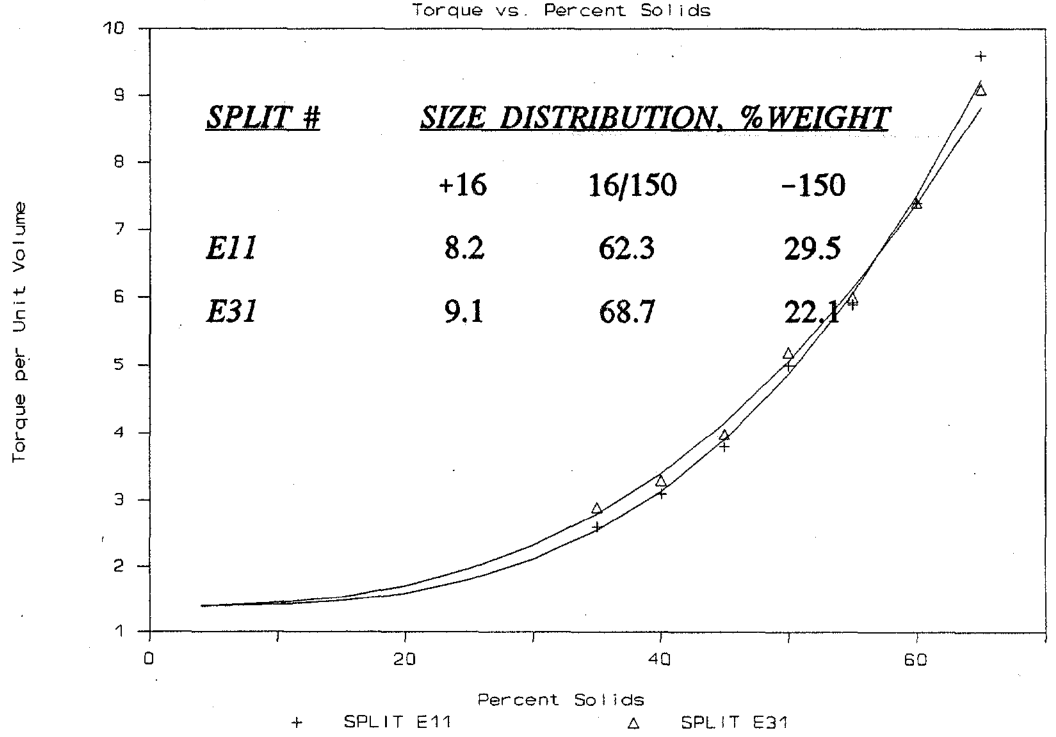
SPLIT #	SIZE DISTRIBUTION, %WEIGHT		
	+16	16/150	-150
C11	22.8	55.4	21.8
C31	6.5	54.3	39.2

Figure 5


Torque vs. Percent Solids

SPLIT #	SIZE DISTRIBUTION, %WEIGHT		
	+16	16/150	-150
S11	6.4	64.8	28.8
S31	18.7	51.1	30.3

Figure 6


Torque vs. Percent Solids

SPLIT #	SIZE DISTRIBUTION, %WEIGHT		
	+16	16/150	-150
E11	8.2	62.3	29.5
E31	9.1	68.7	22.1

Table P-I
Chemical Analysis of +3/8" Pebble Fraction

	P_2O_5	A.R. CaO	MgO	Fe_2O_3	Al_2O_3	Insol
AA4	3.28	26.12	14.56	0.24	0.34	12.44
AB3	1.54	28.05	16.72	0.44	0.27	1.68
AC1	23.62	36.77	1.36	0.72	0.70	16.94
AC5	2.81	25.89	14.33	0.36	0.53	15.38
AD1	12.45	29.73	7.63	0.66	0.76	18.64
AE3	6.00	32.50	11.21	0.67	0.47	7.74
AR3	2.98	27.45	13.86	0.54	0.38	11.58
AS-1	5.48	30.59	12.84	0.50	0.40	8.50

-3/8" Pebble Fractions

The mineral composition of this fraction, as determined by optical microscopy from the selected core splits, is shown in Table P-II. The listed percent phosphatic pebble consists mainly of francolite containing included quartz, organic matter, and perhaps some dolomite.

It can be seen that the pebble fraction from all of the upper splits contain little, if any, dolomite. Practically all of the pebble product from the lower splits contain free dolomite aggregates, amounting to about 10% in split AD-5 to 65%, in split AC-3. Furthermore, the phosphatic pebble in the lower splits often contains dolomite, ranging from a trace in split AD-5 to 14% in split AR-3. Calcite is present in the lower splits of AA-4, AD-5, and AR-3, as yellowish, transparent crystals and crystal fragments. Calcite is also present in split AR-5 as the cement in quartz-francolite aggregates. Complete mineralogical analyses as determined by XRD is given in the first annual report.

Non-phosphatic particles were removed from samples of each of the splits examined. This was accomplished by using a Titan Yellow staining method which colors dolomite red. The free quartz, feldspar, and calcite grains in the pebble fraction were easily discernible at low magnification and were also removed.

Table P-II
Mineral Composition of -3/8" Pebble Fraction

Upper Matrix

	Phosphatic Pebble	Free Dolomite	Free Calcite	Free Quartz and Feldspar	Clay
AA-1	90				10
AB-1	90		Trace		10
AC-1	100				
AD-1	92		Trace		8
AE-1	80				20
AS-1	100		Trace		Trace

Lower Matrix

	Phosphatic Pebble	Free Dolomite	Free Calcite	Free Quartz and Feldspar	Clay
AA-4	64	25	7	3	1
AB-3	85	15			
AC-3	32	65		3	
AD-5	72	10	13	5	
AE-3	85	14		1	
AR-3	79	17	3	1	
AR-5	38	37	25*	Trace	
AS-3	67	31		2	

* Sandstone (quartz-francolite sand with calcitic cement.)

Mineralogical data of these splits as obtained by XRD are given in the first annual report.

The cleaned-up phosphatic pebble for each split was further sized to give a coarse product (-3/8" +8 mesh) and a fine product (-8 + 16 mesh). The locked dolomite content, estimated visually and from chemical analysis, is shown in Table P-III. Chemical analysis of the of the hand picked phosphatic pebble products are shown in Table P-IV for the upper matrix splits and in Table P-V for the lower matrix splits. Note in Table P-IV that the calculated composite analysis for the upper matrix pebble product is as follows:

BPL	65.86
Insol	9.0
MgO	0.49
CaO/P_2O_5	1.49

The analysis in Table P-V, for the lower matrix composite product obtained, is as follows:

BPL	58.02
Insol	8.7
MgO	1.69
CaO/P_2O_5	1.77

Table P-III

Estimated Visual Locked Dolomite Content in Hand-picked Phosphatic Pebble

Upper Matrix

	3/8" x 8 Mesh (Coarse)	-8 + 16 Mesh (Fine)
AA-1	0	0
AB-1	0	0
AC-1	1	0
AD-1	2	Trace
AE-1	0	0
AS-1	1	3
Lower Matrix		
AA-4	3	3
AB-3	17	7
AC-3	3	2
AD-5	3	Trace
AE-3	1	Trace
AR-3	4	3
AR-5	10	4
AS-3	25	10

Table P-IV

Chemical Analysis of Hand-Picked Phosphatic Pebble from Upper Matrix Splits

	Wt%	P_2O_5	BPL	Insol	Fe_2O_3	Al_2O_3	CaO	MgO
AA-1 Coarse	25	29.86	65.24	10.5	0.87	0.83	44.38	0.32
Fine	75	30.05	65.66	9.5	0.90	0.89	45.28	0.35
-3/8"+16		30.00	65.53	9.8	0.89	0.87	45.06	0.34
AB-1 Coarse	30	29.67	64.82	8.3	1.10	0.68	46.26	0.45
Fine	70	30.61	66.88	7.6	1.12	0.86	45.05	0.38
-3/8"+16		30.33	66.13	7.8	1.11	0.80	45.42	.41
AC-1 Coarse	50	31.23	68.24	6.0	0.61	0.89	46.10	0.61
Fine	50	30.41	66.24	8.5	0.73	0.91	46.35	0.35
-3/8"+16		30.82	67.24	7.2	0.67	0.90	46.22	0.48
AD-1 Coarse	40	29.10	63.58	8.6	1.19	0.93	44.80	0.91
Fine	60	30.81	67.32	7.8	1.02	0.95	44.98	0.40
-3/8"+16		30.13	65.80	8.1	1.09	0.94	44.91	0.60
AE-1 Coarse	30	31.67	69.20	8.2	0.77	0.93	44.72	0.25
Fine	70	30.11	65.79	9.9	1.26	0.96	43.76	0.30
-3/8"+16		30.58	66.78	9.4	1.11	0.95	44.05	0.28
AS-1 Coarse	45	30.25	66.10	8.9	1.31	0.93	44.88	0.60
Fine	55	28.10	61.40	14.0	1.26	0.85	42.29	1.08
-3/8"+16		29.07	63.48	11.7	1.28	0.89	43.46	0.86
Avg. Composition		30.16	65.86	9.0	1.02	0.89	44.85	0.49

$CaO/P_2O_5 = 1.49$

Table P-V

Chemical Analysis of Hand-Picked Phosphatic Pebble from Lower Matrix Splits

	Wt%	P_2O_5	BPL	Insol	Fe_2O_3	Al_2O_3	CaO	MgO
AA-4 Coarse	20	26.15	57.14	10.1	1.43	0.70	41.94	1.01
Fine	80	26.86	58.69	10.5	1.53	0.71	41.69	1.18
-3/8"+16		26.72	58.35	10.4	1.51	0.71	41.89	1.15
AB-3 Coarse	35	23.21	50.71	7.3	0.97	0.62	42.66	3.92
Fine	65	27.00	60.00	8.0	1.12	0.72	43.83	1.86
-3/8"+16		25.67	56.07	7.8	1.07	0.68	43.42	2.58
AC-3 Coarse	40	29.83	65.18	4.1	1.26	0.55	45.85	1.10
Fine	60	26.97	58.93	10.2	1.94	0.98	41.73	0.98
-3/8"+16		28.11	61.40	7.7	1.67	0.81	43.38	1.03
AD-5 Coarse	40	26.80	58.56	10.6	1.59	1.02	40.78	1.34
Fine	60	26.88	58.73	10.5	1.57	1.04	40.89	1.11
-3/8"+16		26.85	58.64	10.5	1.58	1.03	40.85	1.20
AE-3 Coarse	50	29.55	64.57	7.2	0.76	0.93	44.67	0.75
Fine	50	26.85	58.67	12.6	1.29	0.76	42.07	0.68
-3/8"+16		28.20	61.62	9.9	1.02	0.84	43.37	0.72
AR-3 Coarse	45	26.70	58.33	10.7	1.56	0.85	41.99	1.21
Fine	55	26.08	56.98	10.3	1.66	0.87	41.64	1.06
-3/8"+16		26.36	57.57	10.5	1.62	0.86	41.80	1.13
AR-5 Coarse	25	24.71	53.99	9.3	0.62	0.40	42.67	2.36
Fine	75	27.93	61.02	5.9	1.03	0.49	45.00	0.97
-3/8"+16		27.12	59.24	6.8	0.93	0.47	44.42	1.32
AS-3 Coarse	60	21.42	46.41	5.4	1.02	0.57	41.90	5.69
Fine	40	26.62	58.16	7.0	1.00	0.66	41.11	2.47
-3/8"+16		23.50	51.32	6.0	1.01	0.61	41.58	4.40
Avg. Composition		26.57	58.02	8.7	1.30	0.75	42.59	1.69

Summary of the Pebble Data

(1) The +3/8" fraction is a poor quality product (very low P_2O_5 and very high MgO content.

(2) Generally, MgO content increases with hole depth, while P_2O_5, Aluminum and Iron decrease with depth.

(3) Chemical analyses of as received -3/8" pebble are summarized below:

P_2O_5	18.1 ± 7.6
CaO	37.3 ± 5.6
F	2.2 ± 0.9
MgO	5.1 ± 3.9
Al_2O_3	0.7 ± 0.4
Fe_2O_3	0.5 ± 0.4
Na_2O	0.3 ± 0.2
K2O	0.1 ± 0.1
A.I.	13.1 ± 4.6
L.O.I.	16.2 ± 9.6
CaO/P_2O_5	2.6 ± 2.2
F/P_2O_5	0.1 ± 0.0

(4) Physical separation may be used to upgrade the -3/8" fraction. The quality of product could be approximated by:

(a) Upper Zone Material

BPL %	65.90
Insol %	9.00
MgO%	0.49
CaO/P_2O_5	1.49

The insoluble material is mostly locked silicas. Thus, grinding may be a necessity for upgrading .

(b) Lower Zone Material

BPL %	58.00
Insol %	8.70
MgO%	1.70
CaO/P_2O_5	1.77

In this case dolomite and silica are also unliberated inclusions which may require grinding for liberation before further upgrading is used.

Feed (16/150 mesh)

Head samples from different splits of various drill cores were analyzed for P_2O_5, CaO, MgO, Al_2O_3 and Fe_2O_3. The obtained data are given in the first annual report. The data indicate, generally, that P_2O_5 content is dependent on the hole location. However, it is apparent that in most of the cases, such content does not depend on the hole depth. On the other hand, elemental concentration, e.g. Ca, Mg, Al, and Fe increase with depth. The ranges of the obtained data can be summarized as:

P_2O_5	7.4 ± 6.4
CaO	12.6 ± 5.3
MgO	0.6 ± 0.5
Al_2O_3	0.2 ± 0.1
Fe_2O_3	0.3 ± 0.2
A.I.	65.5 ± 11.2
L.O.I.	16.2 ± 9.6
CaO/P_2O_5	1.7 ± 0.4
MgO/P_2O_5	0.1 ± 0.1

Chemistry of size fractions from selected holes was determined as shown in Table F-I.

The data clearly show higher concentrations of P_2O_5 and other elements in the coarser sizes (16-35 mesh) as compared to finer material (-35 mesh). It should be mentioned here that the weight distribution of these size fractions indicate the fine fractions (-35 mesh) are higher than the coarse sizes. The size distribution of feed samples from selected splits are given in the first annual report. The data indicate also that feeds from bottom splits tend to be finer than that from top splits.

Generally, it can be stated that the size distributions are normal with an average size around 48 mesh. This average shifts toward 65 or 100 mesh for the lower zone material (bottom splits).

Flotation concentrate

Chemical data for flotation concentrates are given in the first annual report. Considering all data (from all holes and all splits) the following summary may be applied to describe the quality of the flotation concentrate.

Summary of the Characteristics of Flotation Concentrate

	WT %
P_2O_5	29.1 ± 3.4
CaO	46.1 ± 4.9
MgO	1.0 ± 0.6
Fe_2O_3	1.3 ± 0.4
Al_2O_3	0.7 ± 0.2
Na_2O	0.6 ± 0.2
K2O	0.2 ± 0.1
F	3.5 ± 0.6
A.I.	4.7 ± 3.4
L.O.I.	7.8 ± 2.9
P_2O_5 Recovery	73.1 ± 22.9
CaO/P_2O_5, Ratio	1.6 ± 0.4

Table F-I

Chemical Data by Split
Feed Size Chemistry,
Percent

Size Fraction	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3
Sample No.AA1					
16/20	20.40	28.89	0.24	0.78	0.68
20/28	15.84	23.01	0.21	0.60	0.49
28/35	8.00	11.34	0.13	0.34	0.22
35/48	3.60	5.23	0.05	0.17	0.11
48/65	3.12	4.22	0.03	0.14	0.11
65/100	2.96	3.91	0.03	0.12	0.13
100/150	3.28	4.39	0.05	0.14	0.18
Sample No.AA4					
16/20	17.20	33.21	2.72	1.05	0.56
20/28	14.80	29.99	1.79	1.01	0.51
28/35	11.04	19.47	0.63	0.72	0.34
35/48	7.72	13.20	0.34	0.45	0.22
48/65	6.44	10.41	0.19	0.32	0.17
65/100	7.48	12.62	0.26	0.35	0.22
100/150	5.36	8.95	0.33	0.28	0.28

The above results indicate a wide distribution of the data as suggested by higher standard deviations. This can be attributed to the difference in the flotation behavior between samples from top splits (upper zone) and that from bottom splits (lower zone). Table F-II shows the difference in the quality of the products from the top and bottom splits of the selected cores.

If data from the first split of all holes are grouped together to represent the upper zone, the following summary may describe the average characteristics of concentrates from the upper zone.

Summary of the Characteristics of Flotation Concentrates
Upper Zone Matrix

P_2O_5	32.1	±	1.0
CaO	47.0	±	2.6
MgO	0.4	±	0.0
Fe_2O_3	1.4	±	0.3
Al_2O_3	0.8	±	0.2
Na_2O	0.6	±	0.0
K2O	0.1	±	0.0
F	3.7	±	0.2
A.I.	3.8	±	1.9
L.O.I.	6.1	±	1.6
P_2O_5 Recovery	68.3	±	22.7
CaO/P_2O_5, Ratio	1.5	±	0.1

Also, the characteristics of the flotation concentrates from the lower zone matrix can be summarized as follows:

Summary of the Characteristics of Flotation Concentrates
Lower Zone Matrix

P_2O_5	28.1	±	4.7
CaO	45.3	±	7.5
MgO	1.1	±	0.6
Fe_2O_3	1.2	±	0.5
Al_2O_3	0.6	±	0.2
Na_2O	0.6	±	0.2
K2O	0.2	±	0.0
F	3.5	±	0.7
A.I.	4.9	±	3.6
L.O.I.	8.2	±	3.0
P_2O_5 Recovery	74.2	±	22.9
CaO/P_2O_5, Ratio	1.7	±	0.4

The above data suggest that higher recoveries and concentrates of poorer qualities are obtained from the lower zone matrix as compared to the upper zone matrix.

Optical microscopy as well a XRD were used to characterize the mineralogical content of these concentrates as shown in Table F-III. The results indicate presence of dolomite and calcite as in the concentrates from the lower splits. This could be the reason for the low P_2O_5 content in these concentrates. Visual observation of the presence of clay chips may explain some of the observed low recoveries. Examination of the size distribution of the feed material indicates a correlation between the flotation recovery and the size distribution. Higher recoveries are obtained from feeds of normal size distributions. However, lower recoveries were also noticed with feeds of skewed distributions either toward the coarse or the fine sizes.

Table F-II

Approximate Compositions of Selected Flotation Concentrates as Determined by Optical Microscopy

	Francolite	Dolomite	Calcite	Quartz & Others
AA-1	1			4
AA-4	1	4	4	4
AB-1	1			4
AB-3	1	4		4
AC-1	1	5		4
AC-5	1	4	4	4
AD-1	1			3
AD-5	1	4		4
AE-3	1	4		4
AE-5	1			
AR-3	1	3	3	4
AR-5	1	3	3	4
AS-1	1			4
AS-3	1	3	5	4

Major	1
Medium	2
Minor	3
V. Minor	4
Trace	5

Heavy Liquid Separations

Most of the concentrates below the #1 split contain free dolomite and/or free calcite. In order to determine the maximum grade concentrate obtainable, and to calculate the structural formulas for the lower split francolites, samples of "essentially dolomite - calcite free" concentrates need to be produced. Heavy liquid separations were made in an attempt to produce a "pure francolite" from lower splits. The francolite has an apparent gravity ranging from about 2.65 to 2.95, depending on porosity and locked impurities. Most of the dolomite is porous and will have an apparent gravity of less than 2.75; however, some of the dolomite is dense and will have a gravity approaching 2.86. The calcite is relatively pure and will have a sp. gr. of 2.71, and the quartz 2.66. Heavy liquid separations were made at a sp. gr. of 2.90, the sink fraction was relatively "pure francolite. " The float fraction at 2.90 sp. gr. was again separated at a sp. gr. of 2.75. In all, there were three heavy liquid fractions from each of the lower splits:

Sink - sp. gr. 2.90
Middlings - Float 2.90, sink 2.75
Float - Float at 2.75

Five samples of concentrates from lower splits were separated using heavy liquids. Combinations of tetrabromethane (sp. gr. 2.96) and methanol (sp. gr. 0.79) were mixed to produce the desired specific gravity.

Some of the sinks and middlings contained fine dolomite, most of which was removed by screening at 65 mesh. The five concentrates separated by heavy liquids along with weight percentages are listed below:

Concentrates	AA48	AC38	AE38	AR38	AS38
Sink +65 mesh	43%	26%	27%	20%	55%
-65 mesh		3			
Middlings +65	23	39	20	44	29
-65		5		7	
Float	34	27	53	29	16
	100%	100%	100%	100%	100%

Chemical analyses were run on the sink and middling (entire or +65 mesh) fractions. They are listed in Table X. Comparison of the sink fraction to the middlings indicated %BPL, %CaO and %Al_2O_3 are higher in the sink. Percent MgO and % Na_2O are higher in middlings.

X-ray diffraction patterns were run on these same fractions. Results are listed below:

Table F-III

Chemistry of Heavy Liquid Seperation Products

	HCl	Insol A.R.	L.O.I.	BPL	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	SO_3	F	CaO/ P_2O_5	MgO/ P_2O_5	$P2O_5$ /F
AA4M	4.64	3.30	8.69	63.85	29.22	43.82	1.01	1.67	0.70	0.74	0.18	3.41	3.51	1.50	0.03	8.32
AA4S	3.13	1.62	5.56	71.14	32.56	46.87	0.45	1.30	0.66	0.53	0.20	0.78	3.54	1.44	0.01	9.20
AC3M	3.79	2.26	6.13	68.78	31.48	45.84	0.55	1.84	0.77	0.59	0.18	0.86	3.48	1.46	0.02	9.05
AC3S	3.39	1.98	5.58	70.44	32.24	47.14	0.43	1.67	0.72	0.50	0.17	0.53	3.42	1.46	0.01	9.43
AE3M	3.86	2.86	8.99	64.89	29.70	45.59	0.71	0.76	0.96	0.74	0.19	0.72	4.56	1.54	0.02	6.51
AE3S	2.61	1.82	5.83	70.71	32.36	47.18	0.45	1.09	0.76	0.55	0.17	0.64	3.48	1.46	0.01	9.30
AR3M	4.79	4.02	8.88	63.12	28.89	43.91	0.88	1.87	0.81	0.85	0.19	1.13	3.36	1.52	0.03	8.60
AR3S	4.09	2.22	5.72	67.17	30.74	45.88	0.58	1.52	0.66	0.53	0.19	0.85	3.36	1.49	0.02	9.15
AS3M	3.40	2.80	11.21	63.76	29.18	45.07	1.66	1.00	0.68	0.73	0.15	0.75	3.12	1.54	0.06	9.35
AS3S	3.28	1.44	6.29	67.74	31.00	47.25	0.51	1.17	0.62	0.62	0.18	0.71	3.54	1.52	0.02	8.75

M = Middle
S = Sink

	Francolite	Dolomite	Calcite	Quartz
AA48				
Sink	Dominant	Trace	?	Minor
Middlings	Dominant	Minor	Minor	Minor
AC38				
Sink +65	Dominant	N.D.	N.D.	Trace ?
Middlings +65	Dominant	N.D.	N.D.	Trace
AE38				
Sink	Dominant	Trace?	N.D.	Trace
Middlings	Dominant	Trace	N.D.	Minor
AR38				
Sink +65	Dominant	Trace	N.D.	Trace
Middlings	Dominant	Minor	Trace	Minor
AS38				
Sink	Dominant	Trace	N.D.	Trace
Middlings	Dominant	Major	N.D.	Major

(N.D. = None Detected)

The fractions, obtained by heavy liquid separation and screening at ±65 mesh, were examined using a binocular microscope. Descriptions are:

<u>AA48</u>

Sink - Francolite, mainly black, minor white and tan; good separation, no free quartz or carbonates noted.

Middling - Francolite, mainly dark with some white and brown, some locked quartz; trace fine free dolomite.

Tails - Francolite, light to dark gray; some free calcite, some free white porous dolomite; free and locked quartz.

AC38

Sink	+65	Francolite, dark gray; trace white dolomite.
	- 65	Francolite, dark gray; some white dolomite.
Middling	+65	Francolite, gray.
	- 65	Francolite, gray; considerable free white dolomite.
Float		Francolite, gray; some free white dolomite; minor free quartz.

AE38

Sink		Francolite, black and tan; good separation.
Middling		Francolite, tan; good separation.
Float		Francolite, tan; minor free quartz; trace white dolomite.

AR38

Sink		Francolite, dark; trace white dolomite.
Middling	+65	Francolite, gray, minor tan and white; trace white dolomite.
	- 65	Francolite; some white dolomite.
Float		Francolite, gray; free calcite; free quartz; free white porous dolomite.

AS38

Sink		Francolite, tan replacements and gray; trace white dolomite.
Middlings		Francolite, tan replacements and gray; considerable white compact dolomite (graded).
Float		Francolite, tan; much free white dolomite; some free quartz.

A concentrate containing 65-68% BPL (30-31% P_2O_5) can be produced from feeds in the lower splits if all the free calcite, dolomite, and quartz can be removed.

This confirms the results obtained by flotation as well as the following calculation of the chemical formula of the phosphatic mineral (francolite) contained in the matrix.

Chemical analysis of the concentrate product was used to estimate the empirical formula of the phosphate mineral (Francolite) in the feed. Weight percent of each element's oxide (corrected to be free of insoluble material) was converted to weight percent of the element. Elemental weight percents were divided by the atomic weight of the element to convert the values to moles. Multiplying the moles of each element by ten gives the number of atoms of each element. Ionic charge times the number of atoms or ions is the charge contribution from the ion of the element.

Oxide Wt. % x conversion factor = Elemental Wt. %

Elemental Wt. %/Atomic Wt. = Moles of Element

Moles of Element x 10 = Number of Atoms of the Element

Number of Atoms x Charge per Atom = Charge Contribution

After the number of atoms was calculated, cations were summed and anions were summed. Charges contributed by cations and anions were summed separately. Ionic charge difference was determined by subtracting the total anion charge from the total cation charge. The result ‘is a positive number if the anion charge is larger.

Magnesium and calcium were adjusted to reflect the contribution from dolomite in the concentrate product. Splits with calcite were not used in the calculation of the overall Francolite formula.

Data used to calculate the formulas were a combination of concentrations measured chemically and the values for CO_2 calculated from x-ray diffraction data. Concentration of CO_2 measured in an apatite crystal lattice and change in relative angular location of the 004 and 410 reflections are linearly correlated. The formula used for the calculation was derived by Mathews and Nathan (1977). Delta 2-Theta for 004 reflection - 410 reflection was measured from x-ray diffraction data. Their formula calculated CO_2 values which compared very well with the values measured by ascarite absorption of the CO_2 from a mild HCl digestion of the concentrate product.

An average formula for the top split from all the cores was calculated. Slight differences between this formula and the formula calculated for the remaining splits were consistent with all the data generated from other sources.

% BPL	78.57

---

1. Mathews, A. and Nathan Y., 1977. The Decarbonation of Carbonate-Fluorapatite (Francolite); American Mineralogist Vol 62 pp. 565-573.

Average Formula for Francolite in the top split:

FORMULA WEIGHT 975.3

$Ca_{9.42}\ Mg_{.1}\ Na_{.2}\ K_{.03}\ (PO_4)_{5.0}\ (CO_3)_{.97}\ (SO_4)_{.16}\ F_{2.15}$

TOTAL CATIONS 9.76	TOTAL POSITIVE CHARGE 19.28
TOTAL ANIONS 8.28	TOTAL NEGATIVE CHARGE 19.40

CHARGE DEFICIT -0.12

% P_2O_5	37.66
% BPL	82.29

Average Formula for Francolite in the Other Splits:

FORMULA WEIGHT 921.2

$Ca_{8.97}\ Mg_{.12}\ Na_{.23}\ K_{.04}\ (PO_4)_{4.51}\ (CO_3)_{1.05}\ (SO_4)_{.16}\ F_{2.06}$

TOTAL CATIONS 9.37	TOTAL POSITIVE CHARGE 18.47
TOTAL ANIONS 7.88	TOTAL NEGATIVE CHARGE 18.17

CHARGE DEFICIT +0.30

% P_2O_5 35.96

5. Phosphatic clays (- 150 mesh)

Chemistry and Mineralogy.

The chemistry of samples representing the 150 mesh fraction from different splits are determined as given in the first annual report.

The average of all data can be summarized as:

P_2O_5	4.5		
CaO	21.1	±	5.5
MgO	9.5	±	5.0
Fe_2O_3	0.7	±	0.4
Al_2O_3	1.5	±	1.5
SiO_2	1.7	±	0.7
A.I.	28.8	±	15.3

Considering only top splits to represent the upper matrix, the following chemistry may describe the phosphatic clays from the upper matrix:

P_2O_5	12.3	±	3.9
CaO	18.8	±	4.0
MgO	1.9	±	1.5
Fe_2O_3	1.1	±	0.4
Al_2O_3	3.5	±	1.7
SiO_2	2.3	±	0.7
A.I.	43.1	±	7.8

Using the remainder of the splits to represent the lower zone, the chemistry of the phosphatic clays generated from the lower zone matrix may be described by:

P_2O_5	2.6	±	2.2
CaO	21.6	±	5.7
MgO	11.2	±	3.7
Fe_2O_3	0.6	±	0.4
Al_2O_3	1.1	±	1.0
SiO_2	1.5	±	0.6
A.I.	25.4	±	14.7

The above summaries clearly indicate that the phosphatic clays from the upper matrix are of higher P_2O_5 content and lower MgO content than the Phosphatic clays from the lower matrix.

Mineralogical and chemical analyses were made on selected available phosphatic clay samples from the upper and lower matrix zones. Five samples were selected from the upper matrix and seven from the lower matrix.

The samples were sized at 44 microns by screening at 325 mesh, and at 2 microns by differential settling in graduated cylinders. X-ray differential analyses were run on the as-received phosphatic clays. More detailed X-ray and chemical analyses were made on the minus 2 micron fractions of these splits to better identify what clay minerals were present and make an estimate of the mineralogy. The +44 and -44+2 micron fractions were also examined using optical microscopy.

The splits were divided into upper and lower matrix. The upper matrix samples end in 1, which means they are the topmost splits in each core hole. The lower matrix samples end in 2, 3, or 4.

In this report the group mineral name smectite is used, which includes the clay mineral montmorillonite. For practical purposes, montmorillonite and smectite are essentially the same for Central Florida phosphatic clays. Palygorskite is used rather than of attapulgite, mineralogically they are similar.

Chemical analyses unsized phosphatic clays are are shown in Table PC-2. The analyses for P_2O_5, CaO, MgO, Fe_2O_3, and Al_2O_3 were made on the aqua regia soluble fraction.

Table PC-I

CHEMICAL ANALYSES UNSIZED PHOSPHATIC CLAYS

	Aqua Regia Soluble %							
	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Insol	CO_2	LOI
Upper Matrix								
AA17	14.25	20.16	0.85	1.00	3.27	42.08	2.94	11.81
AB17	9.14	14.78	1.69	1.06	2.28	52.90	4.46	17.23
AD17	6.39	21.69	6.75	0.43	0.85	37.38	18.46	22.02
AH17	9.10	18.22	3.12	0.77	1.37	41.10	8.92	14.65
AR17	12.14	16.68	1.49	1.82	8.16*	38.10	3.76	15.77
Average %	10.27	18.31	2.78	1.02	3.29	42.31	7.74	16.30
Lower Matrix								
AA47	0.58	19.76	12.80	0.36	0.68	28.00	28.60	33.41
AB37	0.58	26.96	13.43	0.63	0.34	15.78	32.58	38.28
AD37	0.37	18.05	10.61	0.54	0.70	38.70	24.84	30.39
AD47	1.98	9.79	5.14	0.66	1.25	61.78	11.04	21.81
AH27	3.50	22.46	11.19	0.62	0.79	19.08	26.28	31.23
AR37	4.14	21.35	11.54	0.54	0.78	24.00	26.52	35.06
AS37	2.15	20.99	11.11	0.63	0.96	26.94	26.56	44.41
Average %	1.94	19.91	10.83	0.57	0.79	30.90	25.20	33.40

* = High clay content

The chemical data indicate the average upper matrix is higher in insol (42 % versus 31%), soluble P_2O_5 (10% versus 2%), Fe_2O_3 (1.0% versus 0.6%), and Al_2O_3 (3% versus 0.8%). The average upper matrix contains less MgO (3% versus 11%), CO2 (8% versus 25%), and LOI

(16% versus 33%) compared to the average lower matrix.

Table PC-II shows the chemical analyses minus two (2) micron phosphatic clays. The minus 44 micron material was suspended in a 0.05% solution of sodium hexametaphosphate, placed in a 1,000 ml graduate cyclinder and allowed to settle for 24 hours. The top 900 ml contained less than 2 micron material, and the bottom 100 ml was approximately 2 X 44 microns.

These more detailed chemical analyses were made to better aid in estimating the percent of specific minerals present in the minus two micron fraction. Aqua Regia insolubles and a few HCl insolubles were run. The smectites, palygorskite and sepiolite clays are more soluble in aqua regia than HCl. There was insufficient sample to run HCl insols on the fines in all the splits. The P_2O_5, CaO, Fe_2O_3, and Al_2O_3 analyses were run on the Aqua Regia (Aq. R.) soluble fraction. Fusion (total) analyses were run for CaO, MgO, Al_2O_3, and SiO_2.

Table PC-II

Clay Chemistry

Reported Values as Percents

Sample ID	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	CO_3 as CO_2	L.O.I.	A.R. Insol	CaO/ P_2O_5
AA1C	11.35	16.03	0.71	1.57	6.50	1.68	15.40	41.08	1.41
AB1C	6.68	10.30	1.14	1.49	5.18	1.24	17.80	53.54	1.54
AD1C	10.51	15.62	1.09	1.82	6.01	2.24	16.90	41.72	1.49
Avg.	9.51	13.98	0.98	1.63	5.90	1.72	16.70	45.45	1.48
AE3C	18.96	30.45	2.35	1.72	4.86	5.08	14.40	19.04	1.61
AA4M	1.87	0.00	4.26	0.00	0.00	0.00	22.81	58.14	0.00
AB3M	2.56	0.00	3.91	0.00	0.00	0.00	0.00	55.46	0.00
AR3M	4.94	0.00	4.48	0.00	0.00	5.80	0.00	49.80	0.00
AS3M	2.50	0.00	4.18	0.00	0.00	0.00	0.00	53.30	0.00
Avg.	2.97	0.00	4.21	0.00	0.00	1.45	5.70	54.18	0.00
AA4F	2.02	0.00	3.93	0.00	0.00	2.88	22.23	60.10	0.00
AB3F	2.38	0.00	3.43	0.00	0.00	3.94	22.64	57.86	0.00
AR3F	4.94	0.00	4.10	0.00	0.00	3.78	0.00	50.44	0.00
AS3F	2.60	0.00	3.50	0.00	0.00	0.00	0.00	54.86	0.00
Avg.	2.99	0.00	3.74	0.00	0.00	2.65	11.22	55.82	0.00

The chemical analyses of the minus 2 micron phosphatic clays indicate the upper matrix is higher in P_2O_5 but contains less aqua regia insol, CO_2, aqua regia soluble MgO, and LOI.

Fusion (Total) analyses indicate the average upper matrix contains more CaO, less MgO and SiO_2, compared to the lower matrix. The amount of Al_2O_3 and Fe_2O_3 are similar in the upper and lower units.

X-Ray diffraction analyses and microscopic examinations were made on the phosphatic clay samples, and also on oriented -2 micron fractions, both glycolated and unglycolated.

In this report the group mineral name smectite is used, which includes the clay mineral montmorillonite. For practical purposes, montmorillonite and smectite are essentially the same for Central Florida phosphatic clays. Palygorskite is used instead of attapulgite, mineralogically they are similar. The upper matrix contains considerably more francolite (34% versus 7%) and kaolinite (12 % versus trace) and much less dolomite (9% versus 51%) compared to the lower matrix. The lower matrix averages 6% palygorskite, about 1% is prest in the upper matrix. The amount of quartz and smectite are similar in each. Minor to trace amounts of sepiolite and illite are locally present in both the upper and lower matrix fractions.

The clay minerals smectite, palygorskite, kaolinite, sepiolite and illite are essentially present only in the minus 2 micron fraction. This minus 2 micron fraction contains much less quartz and dolomite than the plus 2 micron fractions.

The weight percent of the sixed samples indicates the amount of +44 micron material is similar in the upper and lower matrixes. The amount of -44=2 micron material is less, and the amount of minus 2 micron is higher in the upper matrix relative to the lower.

A summary of the results is given in Table PC-IV below. These results show that the upper matrix phosphatic clays are higher in kaolimite and francolite content, and that the lower matrix phosphic clays are higher in dolomite and palygorskite. content. Smectite content is about the same order of magnitute in both matrix zones. Since the clay minerials occur almost exclusively in the minus two micron fraction, the analyses of this fraction of the two zones are also given. Chemical and X-Ray data corroborate the above conclusions.

Size analyses of the phosphatic clays show that the upper matrix contains twice the amount of minus two micron material as the lower matrix (45% vs. 22%).

Table PC-IV
MINERALOGY OF PHOSPHATIC CLAY

	Unsized Sample		-2mm Fraction	
	Upper	Lower	Upper	Lower
Kaolinite	12%	Trace	27%	2%
Palygorskite	1	6	3	25
Smectite	12	9	27	40
Francolite	34	7	30	10
Dolomite	9	51	2	10
Quartz, Feldspar	30	26	7	8
Other	2	1	4	5
	100%	100%	100%	100%

Consolidation Behavior.

As mentioned in the experimental section, a laboratory centrifuge was used to study the consolidation behavior of the -150 mesh material representing upper and lower zone matrixes. For this purpose, only a few holes were tested.

The following models were developed to represent the consolidation behavior of different splits as indicated:

e_t = Void ratio at time t
e_{in} = Initial void ratio at time t=0
σ = Effective stress, lbs/sq.ft.
K = permeability, ft/day

Sample AC17:

$$e_t = e_{in} \exp^{-kt} + 2.79\, e_{in}\, \sigma^{-0.275} [1-\exp_{-kt}]$$
$$k = 1.46 \times 10^{-8}\, e_{in}^{2.54}$$
$$K = 2.98 \times 10^{-9}\, e^{4.32}$$

Sample AC47:

$$e_t = e_{in} \exp^{-kt} + 1.70\ e_{in}\ \sigma^{-0.25} [1 - \exp^{-kt}]$$

$$k = 4.41 \times 10^{-9}\ e_{in}^{3}.55$$

$$K = 5.61 \times 10^{-}8\ e^{4}.47$$

Sample AD17:

$$e_t = e_{in} \exp^{-kt} + 1.70\ e_{in} - 0.25 [1 - \exp^{-}kt]$$

$$k = 5.2 \times 10^{-6}\ e_{in}^{1.4}$$

$$K = 1.85 \times 10^{-7}\ e^{5.73}$$

Sample AD57:

$$e_t = e_{in} \exp^{-kt} + 2.64\ e_{in}\ \sigma^{-0.32} [1 - \exp^{-kt}]$$

$$k = 9.2 \times 10^{-6}\ e_{in}^{1.01}$$

$$K = 8.0 \times 10^{-7}\ e^{4.49}$$

Sample AE17:

$$e_t = e_{in} \exp^{-kt} + 3.32\ e_{in}\ \sigma^{-0.30} [1 - \exp^{-kt}]$$

$$k = 3.38 \times 10^{-5}\ e_{in}^{0.34}$$

$$K = 3.92 \times 10^{-6}\ e^{3.22}$$

Sample AE57:

$$e_t = e_{in} \exp^{-kt} = 9.10\ e_{in}\ \sigma^{-0.44} [1 - \exp^{-kt}]$$

$$k = 2.29 \times 10^{-7}\ e_{in}^{2.34}$$

$$K = 3.92 \times 10^{-6}\ e^{3.09}$$

Sample AR37:

$$e_t = e_{in} \exp^{-kt} = 4.4\ e_{in}\ \sigma^{-0.33} [1 - \exp^{-kt}]$$

$$k = 2.11 \times 10^{-6}\ e_{in}^{1.94}$$

$$K = 9.88 \times 10^{-6}\ e^{3.44}$$

Sample AR57:

$$e_t = e_{in} \exp^{-kt} = 4.85\, e_{in}\, \sigma^{-0.36} \left[1-\exp^{-kt}\right]$$

$$k = 4.35 \times 10^{-6}\, e_{in}^{1.31}$$

$$K = 1.75 \times 10^{-6}\, e^{3.21}$$

Using the void ratio model for an initial height of 1.0 ft. (30.5 cm), e_{in} = 20.0 and N= 30g, the following plots of height of clays vs. centrifuge time are generated and plotted in Figures 7-10. The shown data indicate that there is no consistent behavior of clays. In other words, some lower matrix phosphatic clays (clays) consolidate faster than the top cores. For instance, Core #D (Figure 8) shows faster consolidation of the clays from the upper matrix. In order to explain these results, the mineralogical content of each of the studied splits are given in Table PC-V. In the same table, the consolidation rate is described qualitatively. These preliminary data indicate slower rates always correlate with high& clay montmorillonite and/or palygorskite. Also, faster rates are correlated with higher dolomite content. In addition, clays containing palygorskite are slower than the ones containing other clay minerals. Nevertheless, this complex relationship is still under investigation.

Figure 7

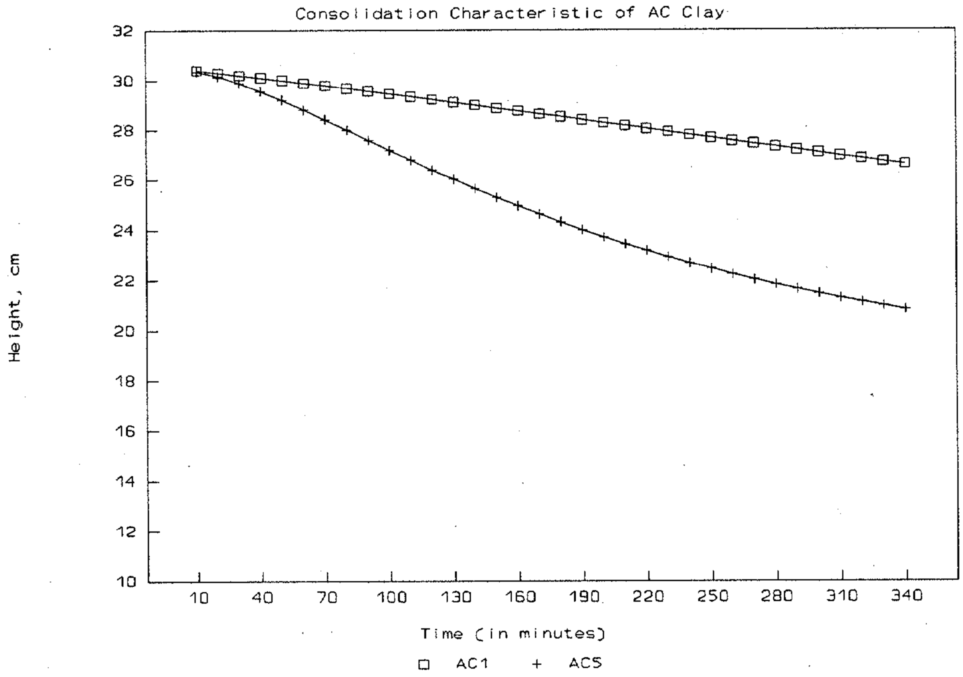


Figure 8

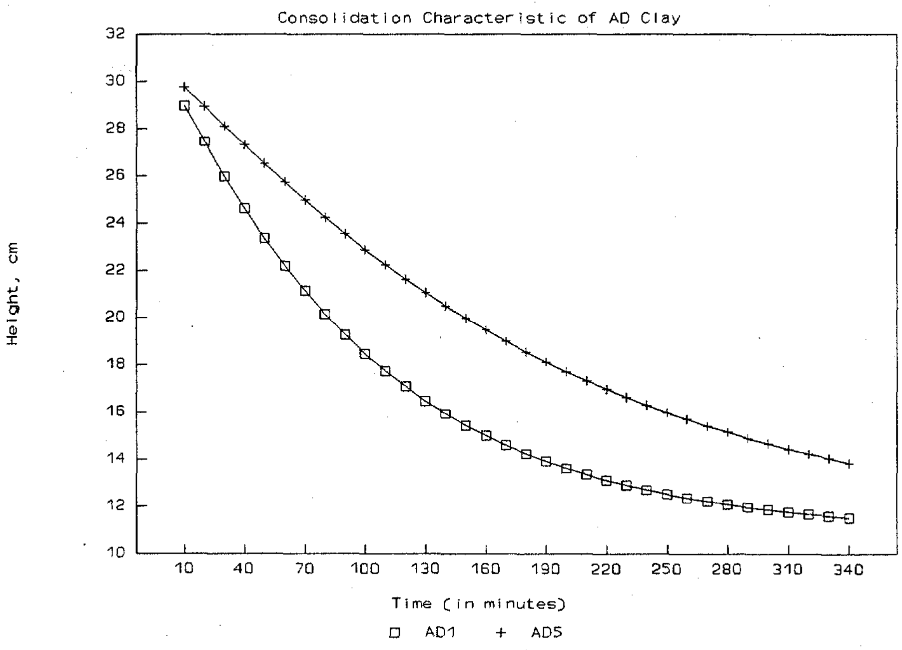


Figure 9

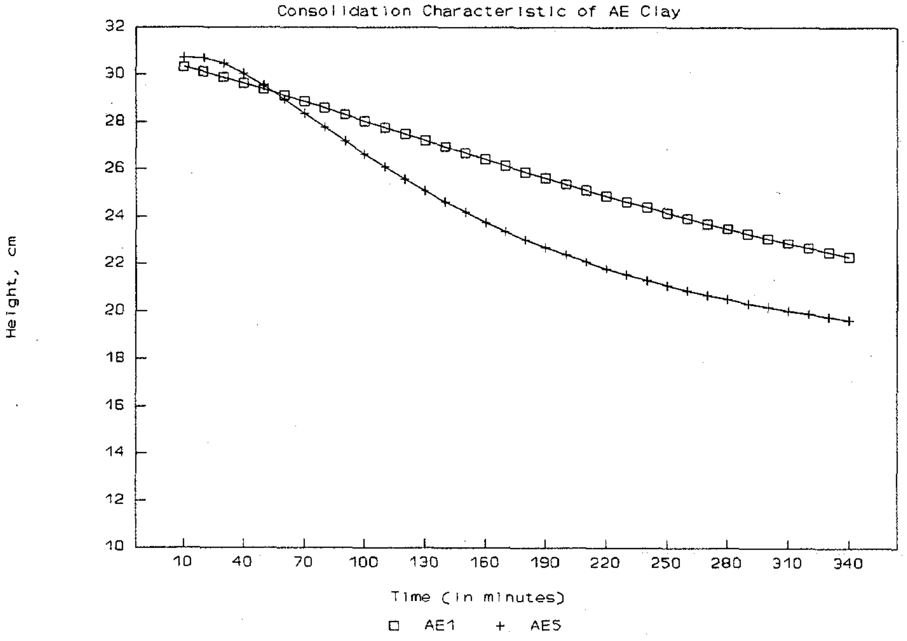


Figure 10

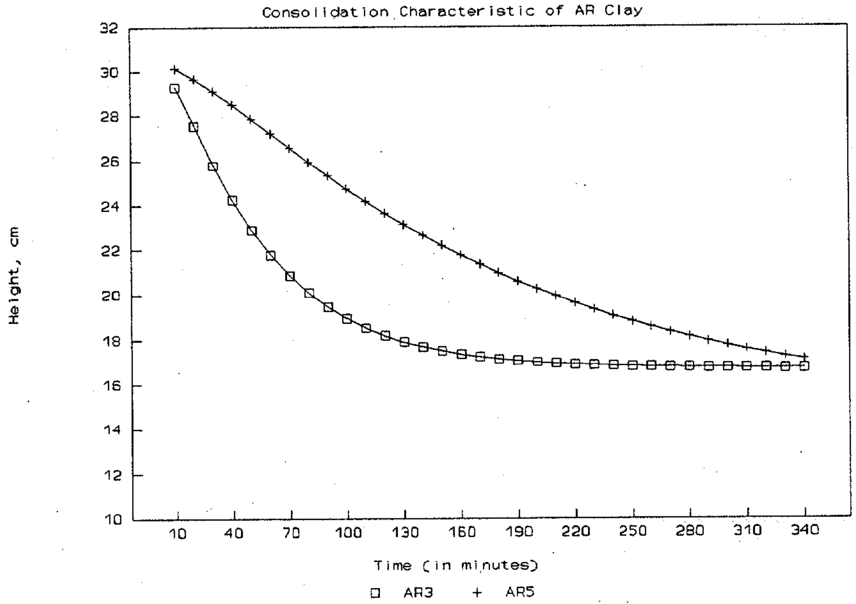


Table PC-V

Correlation of Clay Mineralogy (%Content) and the Consolidation Rate

Mineral	C17	C47	D17	D57	E17	E57	R37	R57
Francolite	23	1	4	4	39	-	2	1
Quartz	57	19	18	3	49	9	3	5
Dolomite	7	75	75	69	6	79	93	92
Calcite	7	-	-	12	-	-	-	-
Wavellite	-	-	-	-	-	-	-	-
Palygorskite	-	1	-	-	-	3	-	2
Montmorillonite	7	-	3	12	6	-	2	-
Consolidation	Slow	Fast	Fast	Slow	Slow	Fast	Fast	Slow

Using the permeability models for a pond of 30 feet high (corresponding to 1.0 ft. model height and N = 30g), the following tables PC-VI to PC-IX show the calculated consolidation behavior as a function of time in years.

It is important to note that these data correlate well with the plots from the centrifugal times.

Confirmation of the validity of the developed models is still under investigation.

Table PC-VI

Consolidation Characteristics of Phosphatic Clay from Hole #AC, Splits #1 and #4

Elapsed Time Split #1	Split #4	Height/ft.	Void Ratio	% Solids by Weight
0	0	30.0	27	10.0
21.0 days	3.0 days	24.6	22	12.0
104.0 days	20.0 days	19.3	17	15.0
1.45 yrs.	0.36 yrs.	13.9	12	20.0
3.12 yrs.	0.88 yrs.	11.8	10	23.1
7.65 yrs.	2.55 yrs.	9.6	8	27.3
23.27 yrs.	3.59 yrs.	7.5	6	33.3

Table PC-VII

Consolidation Characteristics of Phosphatic Clay from Hole #AD, Splits #1 and #5

Elapsed Time Split #1	Split #5	Height/ft.	e	% Solids
0	0	30.0	27	10.0
2.9 hrs.	13.2 hrs.	24.6	22	12.0
18.0 hrs.	2.9 days	19.3	17	15.0
5.3 days	16.0 days	13.9	12	20.0
13.6 days	35.8 days	11.8	10	23.1
41.3 days	92.4 days	9.6	8	27.3
0.45 yrs.	0.82 yrs.	7.5	6	33.3
3.1 yrs.	4.12 yrs.	5.4	4	42.9

Table PC-VIII

Consolidation Characteristics of Phosphatic Clay from Hole #AE, Splits #1 and #5

Elapsed Time				
Split #1	Split #5	Height/ft.	e	% Solids
0	0	30.0	27	10.0
2.50 days	3.0 days	24.6	22	12.0
11.8 days	14.0 days	19.3	17	15.0
58.0 days	60.0 days	13.9	12	20.0
122.0 days	117.0 days	11.8	10	23.1
0.80 yrs.	0.70 yrs.	9.6	8	27.3
2.34 yrs.	1.85 yrs.	7.5	6	33.3
10.15 yrs.	6.89 yrs.	5.4	4	42.9

Table PC-IX

Consolidation Characteristics of Phosphatic Clay from Hole #AR, Splits #3 and #5

Elapsed Time				
Split #3	Split #5	Height/ft.	e	% Solids
0	0	30.0	27	10.0
11.0 hrs.	5.2 days	24.6	22	12.0
2.2 days	22.8 days	19.3	17	15.0
10.5 days	100.1 days	13.9	12	20.0
21.8 days	199.0 days	11.8	10	23.1
51.5 days	1.2 yrs.	9.6	8	27.3
149.2 days	3.26 yrs.	7.5	6	33.3
1.74 yrs.	12.45 yrs.	5.4	4	42.9

Appendix III

Data for Area B

Matrix

Product Content

Data obtained for the product content, e.g. pebble (+16 mesh), feed (16/150 mesh), and phosphatic clays (-150 mesh) expressed as tons/acre foot are shown in second annual report. The data represent different splits from 17 drill holes. The % P_2O_5 content in these products is also given. The statistical averages of these data can be summarized as:

Product	Tons/acre foot	BPL%	Weight%
Pebble	92 (21-163)	47 (36-59)	6 (1-11)
Feed	1016 (778-1254)	15 (12-18)	67 (51-83)
Phosphatic clays	417 (253-581)	8 (1-16)	27 (16-38)

Detailed chemistry and mineralogy of each of these constituents are given in later sections of this report.

Rheology

Pumping of slurry depends on its rheological characteristics such as viscosity or fluidity which in turn depends on the solids content as well as on the mineralogical and physical properties of the solids.

For the purpose of investigating the difference in rheology of matrix slurries from the upper and lower zone material, the torque exerted on an impeller rotating in such slurries was measured. The obtained data for matrix slurries from different splits of several drill holes are given in Figures 1-3. The size distribution of the tested samples are also given for comparison.

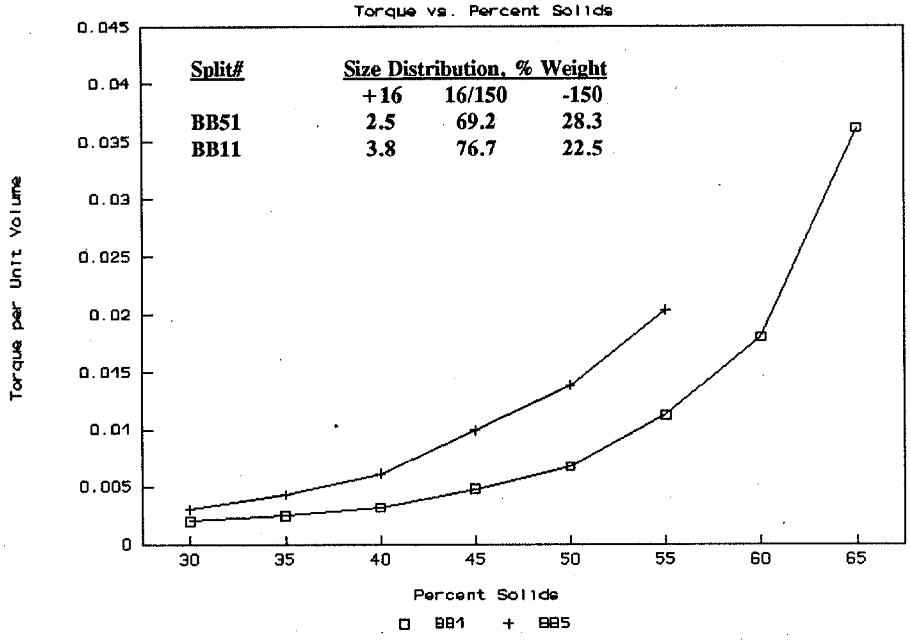
Generally, the data indicate that energy consumption increases exponentially with the increase in the % solids content.

Figure 1 suggests that more energy is needed to pump slurry from split BB-5 than pumping slurry from split BB-1. This could be attributed to the presence of more fines (-150 mesh) in split BB-5 as shown by the weight distribution of different size fractions. It is also worthy to mention that more smectite and palygorskite clays are found in the matrix as compared to the matrix from split BB-1.

The same argument can be applied to the matrix from hole BW as shown in Figure 2.

On the other hand, more energy is needed to pump matrix of split BB-1 in hole G as compared to split BG-4 even though the weight distribution indicates presence of more fines in the lower matrix (BG-4) (see Figure 3). Examining the mineralogical data of the -150 mesh fraction suggests the presence of a larger quantity of montmorillonite in the upper matrix. In addition, the feed size distribution (16/150 mesh) tends to be finer than in the lower matrix. These two differences may explain the observed increase in energy.

Figure 1



Split#	Size Distribution, % Weight		
	+16	16/150	-150
BB51	2.5	69.2	28.3
BB11	3.8	76.7	22.5

Figure 2

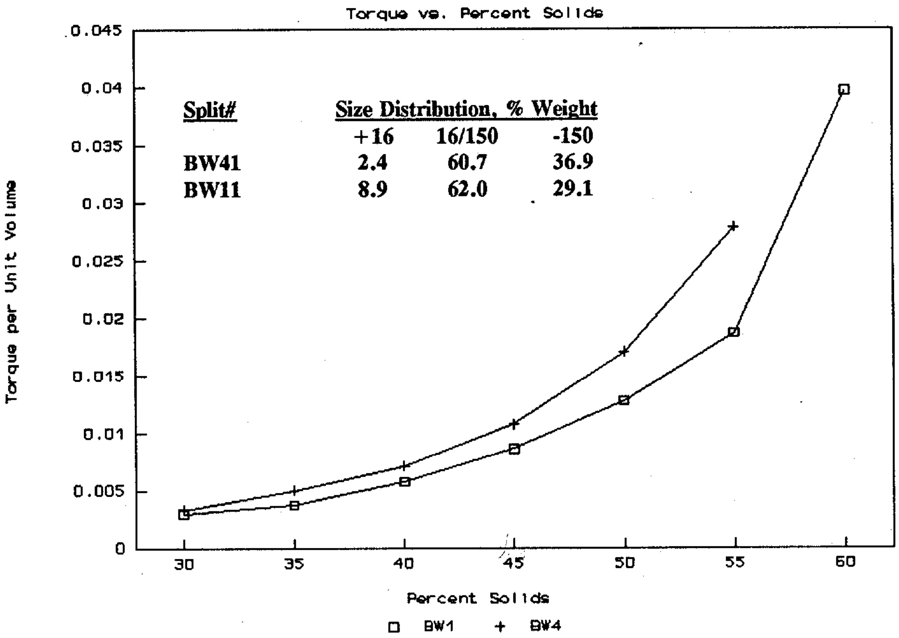
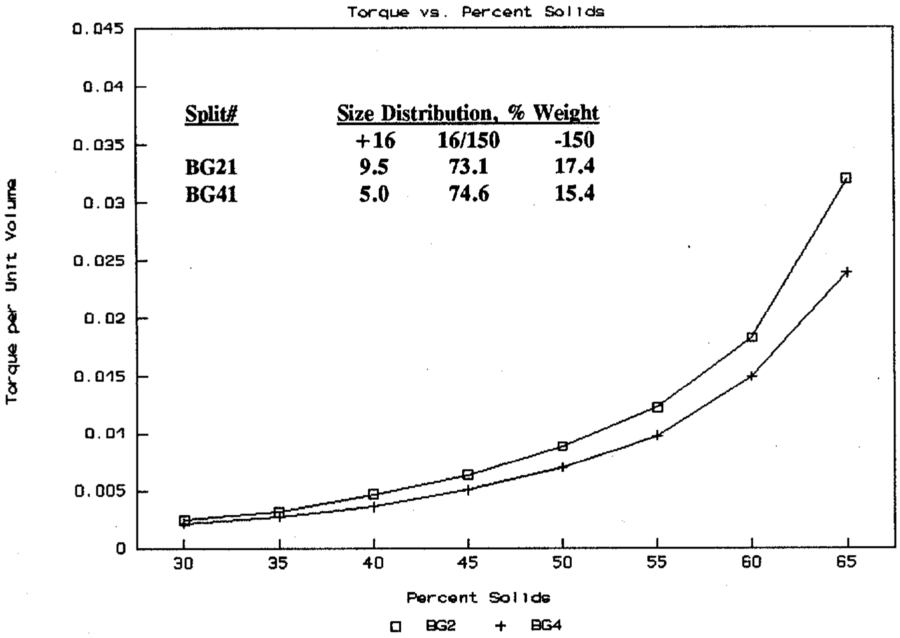


Figure 3



Pebble

The pebble fractions as received from Area B consisted of minus 1/2 inch by plus 14 mesh material. Some of the splits also contained separate samples of plus 1/2 inch pebble, but since this product is usually very high in dolomite and would be discarded in a production plant, no further work was planned.

Samples of the -½" + 14 mesh pebble were examined using optical microscopy, X-ray diffraction, and chemical analyses. Samples of pebble were treated with Titan Yellow dye which imparts a red stain to dolomite. These samples were then -hand-sorted into francolite/non-francolite fractions to evaluate free versus locked gangue minerals. Heavy liquid separation was also used in an attempt to separate francolite from gangue minerals. The stained products and sink-float fractions were further examined by the procedures mentioned above.

The analytical data (X-ray, microscopic and chemical) indicate that the pebble products fall for the most part into two categories.

The upper zone pebble product consists of francolite with locked sand grains (quartz and minor feldspar) and only traces of dolomite as surface smears. The pebble product from split BB-1, however, contains some free dolomite. The only other minerals noted were minor amounts of free quartz and feldspar grains and some free clay aggregates.

The lower zone pebble product is similar but also contains variable amounts of free dolomite pebbles, and dolomite inclusions. Minor amounts of free, pale yellow calcite crystals and crystal fragments are occasionally present. Most of the free and locked dolomite is white, porous, and relatively soft consisting of aggregates of 5 micron rhombs. However, some dolomite is tan to gray, hard, and dense, and can be mistaken for francolite. Much of the soft, porous dolomite, calcite, and clay might be removed by attrition scrubbing.

A description of the as-received pebble samples is given in Table P-I. X-ray diffraction analysis of the pebble products, given in Table P-II, shows the major difference between the upper matrix zone and the lower matrix zone to be the presence of dolomite.

Chemical analyses were made on the samples of as-received pebble fractions. Results are shown in Table P-III. The average P_2O_5 content in the upper matrix is 30% higher than the average P_2O_5 in the lower matrix: 27.01% vs. 20.58%. The higher insol, MgO, and CO_2 in the lower matrix reflect the presence of more diluents such as quartz and dolomite. Average chemical analysis of the upper and lower matrix zones for as-received pebble is as follows:

As-received Pebble Grade

	Upper	Lower
P_2O_5	27.01	20.58
CaO	41.12	35.46
MgO	0.86	2.28
Fe_2O_3	1.12	1.32
Al_2O_3	0.94	0.69
Na_2O	0.60	0.66
K_2O	0.13	0.13
F	3.52	2.88
CO_2	5.33	8.01
Organic C	1.00	1.34
LOI	7.66	10.37
Insol	11.53	18.63
CaO/P_2O_5	1.53	1.73

Table P-I

DESCRIPTION OF AS-RECEIVED PEBBLE

BB1	Francolite, brown, some dolomite surface smears, about 10% locked fine sand; approximately 10% free dolomite, white porous, some dense; minor free quartz; approximately 5% free clay pellets, yellowish buff, rounded; trace calcite.
BB5	Francolite, gray to black, approximately 10% locked fine sand, some dolomite surface smears; approximately 10% free dolomite, white porous; approximately 5% sandstone, francolite and quartz grains, white crystalline dolomite cement; approximately 3% free calcite, pale yellow inter-grown fine crystals; minor free quartz.
BD3	Francolite, dark gray, approximately 10% locked fine sand, dolomite surface smears; approximately 5% free dolomite, white porous and compact; 1% white clay; minor free sand.
BG2	Francolite, white to tan to brown to black, some is white earthy and looks like dolomite, approximately 10% locked fine quartz; trace free quartz; trace free clay; no dolomite noted.
BG4	Francolite, black, some light colored, scattered fine sand, some white dolomite surface smears; approximately 10% free dolomite, light colored which closely resemble francolite; 5-10% clay with fine sand.

BI1	Francolite, mainly white, some black, scattered fine sand; trace dolomite noted.
BI4	Francolite, mainly gray, approximately 10% locked fine sand, minor dolomite inclusions and surface smears; approximately 2% free dolomite, white porous; trace free sand; trace free calcite.
BM2	Francolite, brown and gray, approximately 10% locked fine sand; minor free dolomite, white; trace free sand; minor free clay pellets, light color.
BM7	Francolite, black, approximately 15% locked fine sand, some white dolomite surface smears; approximately 5% compact and porous free dolomite, white; trace free sand.
BT1	Francolite, gray and brown, some white, approximately 10% locked fine sand; minor free dolomite, white, porous trace free sand; trace clay pellets.
BT4	Francolite, dark gray, approximately 10% locked fine sand, dolomite surface smears; approximately 15% free dolomite, compact and white, porous; trace free sand; approximately 2% free clay pellets.
BW1	Francolite, brown, gray, white, approximately 10% locked fine sand; trace free quartz, trace free dolomite.
BW4	Francolite, dark gray, approximately 15% locked fine insol; dolomite inclusions and surface smears; approximately 15% free dolomite white, fine sugary to dull, porous; trace free sand.

Table P-II

X-RAY DIFFRACTION ANALYSES OF AS RECEIVED PEBBLE

SPLIT	FRANCOLITE	QUARTZ-FELDSPAR	DOLOMITE
Upper Zone			
BB1	Major	Moderate	Moderate
BG2	Major	Moderate	-
BI1	Major	Moderate	Trace
BM2	Major	Major	Trace
BT1	Major	Moderate	Trace
BW1	Major	Major	Trace
Lower Matrix			
BB5	Major	Major	Major
BD3	Major	Major	Minor
BG4	Major	Major	Minor
BI4	Major	Moderate	Minor
BM7	Major	Moderate	Major
BT4	Major	Moderate	Moderate
BW4	Major	Moderate	Moderate

Table P-III

Chemical Analysis of As Received Pebble

Upper Matrix

ID	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	F	CO_2	Org C	L.O.I.	A.R. Insol	CaO/ P_2O_5
BB1	22.53	38.88	2.47	1.00	1.08	0.58	0.13	3.25	9.20	0.84	11.29	12.64	1.73
BG2	27.58	40.70	0.41	0.77	0.94	0.63	0.14	3.70	4.10	1.03	7.00	12.16	1.48
BI1	28.87	42.97	0.32	0.66	1.30	0.46	0.10	3.69	4.02	0.67	6.44	10.72	1.49
BM2	27.49	40.81	0.45	1.59	0.62	0.62	0.14	3.62	4.90	1.23	6.42	12.40	1.48
BT1	27.56	42.00	0.88	1.26	0.89	0.67	0.16	3.56	4.82	1.41	7.44	9.54	1.52
BW1	28.00	41.36	0.65	1.46	0.81	0.61	0.13	3.30	4.96	0.84	7.35	11.72	1.48
Average	27.01	41.12	0.86	1.12	0.94	0.60	0.13	3.52	5.33	1.00	7.66	11.53	1.53

Lower Matrix

ID	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	F	CO_2	Org C	L.O.I.	A.R. Insol	CaO/ P_2O_5
BB5	18.22	34.45	3.50	1.00	0.70	0.55	0.12	2.82	12.38	1.01	14.34	15.50	1.89
BD3	22.91	37.18	1.34	1.20	0.66	0.69	0.14	3.31	7.00	1.09	9.79	16.78	1.62
BG4	18.61	29.43	1.03	1.56	0.66	0.54	0.12	2.55	4.62	1.39	7.01	33.40	1.58
BI4	23.03	38.59	1.46	1.33	0.66	0.75	0.14	3.39	6.48	1.52	8.51	14.42	1.68
BM7	21.72	36.58	1.79	1.30	0.59	0.77	0.12	3.15	6.52	1.66	8.78	18.32	1.68
BT4	20.22	36.94	3.60	1.13	0.85	0.66	0.14	2.49	10.44	1.30	12.26	14.56	1.83
BW4	19.35	35.05	3.22	1.74	0.70	0.66	0.14	2.45	8.60	1.40	11.92	17.42	1.81
Average	20.58	35.46	2.28	1.32	0.69	0.66	0.13	2.88	8.01	1.34	10.37	18.63	1.73

The selected pebble samples were treated with Titan Yellow staining solution which imparts a red color to the dolomite. The particles were then hand-sorted into francolite and non-francolite fractions so that the maximum grade of the phosphate pebble and the composition of the free gangue minerals could be determined. A description of the products and their weight distribution is given in Table P-IV.

The mineral composition, as determined by observation under a low-power microscope, is shown in Table P-V. This table shows significant differences between the upper matrix zone and lower zone products. The most important difference is that the phosphate pebble of the lower zone contains some locked dolomite and more included insoluble material (largely quartz and feldspar).

The hand-sorted phosphate pebble fractions were chemically analyzed. Results are given in Table P-VI. The average maximum grade, shown below, is what can be attained if all of the free gangue mineral (dolomite, quartz, feldspar, calcite, and clay) could be removed.

	Hand Picked Pebble Grade	
	Upper	Lower
P_2O_5	29.24	24.81
CaO	45.14	40.61
MgO	0.49	1.22
Fe_2O_3	1.27	1.58
Al_2O_3	0.86	0.72
F	3.49	3.12
CO_2	4.34	5.76
Insol	7.93	12.55
CaO/P_2O_5	1.54	1.64

Table P-IV

DESCRIPTION OF STAINED HAND-SORTED PEBBLE

Split	Wt %	Fraction
BB1	83	Francolite, brown, approximately 5% locked sand; less than 1/2% locked dolomite mainly as surface stains
	17	Nonfrancolite, estimate 40% light yellow clay; 50% dolomite; 10% sand
	100	

BB5	82	Francolite, mainly dark gray, 10% locked fine sand; approximately 3% locked dolomite as surface smears and locked
	14	75% dolomite, 25% clay
	3	Calcite, aggregates of fine crystals
	1	Quartz
	100	
BD3	95	Francolite, mainly gray to dark gray, 10% locked fine sand; trace dolomite mainly as surface smears; 2% free compact dolomite
	5	Nonfrancolite, estimate 50% dolomite; 40% quartz; 10% clay pellets
	100	
BG2	99	Francolite, mainly white to tan to brown to black, estimate 10% locked sand - no dolomite
	1	95% sand; 5% dolomite
	100	

BG4	80	Francolite, dark gray, 10% locked sand; 3% locked dolomite as smears and inclusions
	14	Dolomite
	5	Clay, gray
	1	Sand
	100	
BI1	100	Francolite, white, earthy, some black, 10% locked sand, trace dolomite
BI4	97	Francolite, dark gray, 10% locked sand, 3% locked dolomite
	3	Nonfrancolite, estimate 50% dolomite; 35% sand; 10% calcite; 5% sandstone
	100	
BM2	99	Francolite, brown and gray rounded, polished; 10% locked fine sand - no dolomite
	1	Nonfrancolite, 80% sand; 20% dolomite
	100	
BM7	92	Francolite, black, 10% locked sand, 1-1/2% locked dolomite
	5	Nonfrancolite, 85% porous dolomite, 15% sand
	3	Nonfrancolite, hard compact dolomite
	100	
BT1	98	Francolite, tan, rounded, polished, 10% fine sand, minute traces dolomite
	2	Non-francolite, 70% dolomite, 30% sand
	100	

BT4	86	Francolite, black, 10% sand, 5% dolomite as smears and inclusions
	10	Nonfrancolite; 60% porous dolomite; 20% sand; 20% clay pellets
	4	Nonfrancolite; hard compact dolomite
	100	
BW1	99	Francolite, brown and gray and white earthy, 6% locked sand, traces of dolomite
	1	Nonfrancolite, 70% sand, 30% dolomite
	100	
BW4	84	Francolite, black, 15% locked sand, 5% dolomite, smears and inclusions
	16	Nonfrancolite - 95% dolomite, 5% sand
	100	

Table P-V

MINERAL COMPOSITION - STAINED HAND-SORTED PEBBLE

Upper Zone

	Francolite Fraction			Non-Francolite Fraction			
Split	Francolite	Dolomite	Quartz-Feldspar	Dolomite	Quartz-Feldspar	Calcite	Clay
BB1	75	1	7	9	2	6	-
BG2	92	-	7	Trace	-	-	-
BI2	91	-	9	-	-	-	-
BM2	91	Trace	9	Trace	1	-	-
BT1	91	Trace	7	1	1	-	-
BW1	93	6	1	1	-	-	-
Average	89	Trace	8	2	1	1	

Lower Zone

	Francolite Fraction			Non-Francolite Fraction			
Split	Francolite	Dolomite	Quartz-Feldspar	Dolomite	Quartz-Feldspar	Calcite	Clay
BB5	68	3	11	11	13	3	-
BD3	82	1	10	5	2	1	-
BG4	68	2	10	13	2	5	-
BI4	84	3	10	2	1	1	Trace
BM7	78	2	12	7*	1	-	-
BT4	72	4	10	10*	2	2	-
BW4	65	4	15	15	1	-	-
Average	74	3	11	9	1	1	1

* Includes hard dense dolomite that will be difficult to separate from the francolite

Table P-VI

Chemical Analysis Stained Hand Sorted Pebble

Upper Zone

Split	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	F	CO_2	A.R. Insol	CaO/ P_2O_5
BB1	26.69	41.36	0.80	0.83	0.68	0.93	0.13	3.32	5.57	7.36	1.55
BG2	30.12	46.62	0.45	0.93	1.00	0.98	0.14	3.35	3.98	7.20	1.55
BI1	30.52	45.28	0.33	0.80	1.10	0.90	0.10	3.45	3.93	8.80	1.48
BM2	28.60	45.42	0.50	1.79	0.70	0.75	0.14	3.60	4.86	9.20	1.59
BT1	29.30	45.85	0.45	1.53	0.91	0.71	0.16	3.60	3.81	7.60	1.56
BW1	30.20	46.30	0.43	1.77	0.77	0.88	0.13	3.63	3.87	7.40	1.53
Average	29.24	45.14	0.49	1.27	0.86	0.86	0.13	3.49	4.34	7.93	1.54

Lower Zone

Split	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	F	CO_2	A.R. Insol	CaO/ P_2O_5
BB5	24.64	39.47	1.19	1.17	0.70	1.08	0.14	3.05	5.70	11.06	1.60
BD3	24.88	40.86	1.11	1.46	0.66	1.05	0.16	3.05	5.82	13.40	1.64
BG4	26.62	42.84	0.91	1.84	0.72	0.98	0.13	3.25	4.74	9.60	1.61
BI4	25.61	42.23	1.28	1.56	0.68	1.06	0.14	3.15	5.74	12.20	1.65
BM7	23.40	38.23	0.99	1.44	0.66	1.00	0.14	3.15	5.13	14.92	1.63
BT4	24.46	40.81	1.64	1.56	0.89	1.08	0.24	3.10	6.62	11.66	1.67
BW4	24.03	39.82	1.43	2.03	0.76	0.97	0.17	3.12	6.58	15.00	1.66
Average	24.81	40.61	1.22	1.58	0.72	1.03	0.16	3.12	5.76	12.55	1.64

Heavy Liquid Separations

A 2.75 Sp. Gr. heavy liquid was initially used to separate the francolite from the gangue, but too much francolite reported to the float fraction. Separation at Sp. Gr. 2.60 also gave poor results as the free calcite, free quartz, and some of the free dolomite reported to the sink fraction along with most of the francolite. The sink-float separation did not yield products comparable to the "pure fractions" attained by staining and hand sorting. Table VII lists the description and weight percents of the sink-float fractions at 2.60 Sp. Gr., splits BB1 and BD3 have three separations sink (at 2.75), mid, Float at 2.75 sink at 2.60), and float (at 2.60). Table VIII lists the chemical analyses of the sink-float fractions.

Table P-VII

MICROSCOPIC DESCRIPTION SINK FLOAT FRACTIONS

Split	Wt %	Fraction	Description
BB1	46	Sink	Francolite, mainly tan, rounded, polished, minor locked sand, minor dolomite stains; approximately 3% free white to tan compact dolomite
	25	Mid	Francolite, tan, rounded, polished,some dull white dolomite stains, some locked fine sand; 2% free sand; 2% free light colored dolomite
	29	Float	Francolite, dark, 2% white dolomite stains, 10% locked fine sand; 10% free white dolomite; 3% pale yellow calcite; 1% clay; 5% free sand
BB5	88	Sink	Francolite, dark, 2% white dolomite stains, 10% locked fine sand; 10% free white dolomite; 3% pale yellow calcite; 1% clay; 5% free sand
	12	Float	Locked fine sandstone; 40% francolite, white to tan to gray, 10% locked fine sand; 15% gray clay; 35% dolomite, white earthy; 5% calcite
BD3	49	Sink	Francolite, gray; 10% locked fine sand, 3% locked dolomite, 3% dolomite stains; 3% free dolomite
	44	Mid	Gray francolite, 15% locked fine sand, 3% dolomite stains; 2% free sand.
	7	Float	Locked fine sandstone 50% francolite, 10% locked sand, dolomite stains; 25% light dolomite; 25% gray clay
BG2	71	Sink	Francolite, white to tan to gray, 10% locked fine sand; minor free sand
	29	Float	Francolite, as in sink
BG4	67	Sink	Francolite, mainly dark, 1% dolomite stains, 5% locked fine sand
	33	Sink	40% francolite; 20% Free gray sandy clay; 25% white earthy dolomite

TABLE P-VII

MICROSCOPIC DESCRIPTION SINK FLOAT FRACTIONS

Split	Wt %	Fraction	Description
BI1	83	Sink	Francolite, black, rounded, polished, some white earthy, approximately 10% fine sand
	17	Float	Francolite, mainly white earthy, 10% locked sand
BI4	95	Sink	Francolite, dark, approximately 10% locked fine sand, 1% dolomite stains; 3% free white dolomite; minor free sand; trace calcite
	5	Float	Francolite, gray, some locked sand, minor dolomite strains; trace gray clay, some free dolomite
BM2	85	Sink	Francolite, tan to brown to gray,rounded, polished, 5% locked fine sand; 5% sandstone, quartz-francolite grains, francolite cement; trace-free sand
	15	Float	Francolite, white to tan to gray, 10% locked sand, 2% white dolomite; 5% light gray sand
BM7	83	Sink	Francolite, black, 10% locked fine sand, 2% locked dolomite; 2% free dolomite; trace free sand
	17	Float	Francolite; 30% white dolomite
BT1	90	Sink	Francolite, brown and gray, rounded, polished, trace dolomite strains, 15% locked fine sand; trace free sand; trace free dolomite with sand and francolite grains
	10	Float	85% Francolite; 10% free dolomite; 5% free clay
BT4	70	Sink	Francolite, dark, minor dolomite stains, 10% locked fine sand; approximately 3% free dolomite; trace free sand
	30	Float	70% francolite, approximately 10% locked sand; 15% free dolomite; 10% free clay
BW1	90	Sink	Francolite, brown to gray, trace dolomite stains, 10% locked fine sand, trace free sand
	10	Float	90% francolite, 10% locked fine sand, 10% free buff porous dolomite
BW4	84	Sink	Francolite, black, approximately 12% locked fine sand, 1% dolomite stains; 5% free white compact dolomite; 1% free sand
	16	Float	65% francolite, approximately 10% locked fine sand; 35% dolomite, white light gray, dull, porous

Table P-VIII

Chemical Analyses Sink Float Pebble Fractions

ID	Weight %	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	F	CO_2	Org C	A.R. Insol	CaO/ P_2O_5
BB1F	29.0	14.00	32.10	5.67	1.12	1.42	0.40	0.12	1.80	17.28	0.63	15.26	2.29
BB1M	25.0	24.37	41.98	1.26	0.63	0.64	0.70	0.12	3.12	6.12	0.90	14.34	1.72
BB1S	46.0	27.59	46.05	0.80	0.90	0.64	0.73	0.14	3.50	5.32	0.72	7.82	1.67
Wt. Avg.		22.84	40.98	2.33	0.89	0.87	0.63	0.13	2.91	8.42	0.74	11.61	1.86
BB5F	12.0	13.26	33.71	6.20	0.94	1.02	0.44	0.14	1.65	18.74	1.21	13.74	2.54
BB5S	88.0	20.01	39.15	3.48	1.02	0.62	0.67	0.12	2.52	9.48	0.74	15.00	1.96
Wt. Avg.		19.20	38.50	3.81	1.01	0.67	0.65	0.12	2.42	4.17	0.80	14.85	2.03
BD3F	7.0	19.27	36.63	4.58	1.23	0.98	0.61	0.16	2.25	14.14	1.36	11.18	1.90
BD3M	44.0	23.00	39.18	1.16	1.10	0.70	0.77	0.14	2.95	4.08	1.55	16.92	1.70
BD3S	49.0	24.84	42.14	1.19	1.60	0.60	0.80	0.14	3.15	5.92	1.54	11.62	1.70
Wt. Avg.		23.64	40.45	1.42	1.36	0.67	0.77	0.15	3.00	8.35	1.53	13.92	1.71
BG2F	29.0	28.08	40.14	0.45	0.67	1.04	0.61	0.13	3.12	4.98	0.94	10.40	1.43
BG2S	71.0	28.26	44.59	0.41	0.80	0.89	0.63	0.14	3.50	4.25	1.17	9.42	1.58
Wt. Avg.		28.21	43.30	0.42	0.76	0.93	0.63	0.14	3.39	6.62	1.10	9.70	1.53
BG4F	33.0	16.77	31.13	3.95	1.97	1.42	0.53	0.18	2.14	10.64	1.25	19.01	1.86
BG4S	67.0	25.58	41.54	0.88	2.19	0.64	0.77	0.13	3.12	5.02	1.18	11.60	1.62
Wt. Avg.		22.67	38.11	1.89	2.12	0.90	0.69	0.15	2.80	5.26	1.20	14.05	1.70
BI1F	17.0	28.58	40.12	0.33	0.73	2.53	0.36	0.08	2.95	3.78	1.24	14.22	1.40
BI1S	83.0	28.63	44.09	0.32	0.53	0.70	0.53	0.08	3.42	4.19	0.49	10.12	1.54
Wt. Avg.		28.62	43.41	0.32	0.56	1.01	0.50	0.08	3.34	6.37	0.61	10.82	1.52
BI4F	5.0	21.96	37.44	3.32	1.24	0.68	0.70	0.13	2.74	10.84	1.69	9.38	1.71
BI4S	95.0	24.09	40.69	1.79	1.52	0.62	0.78	0.14	2.90	6.83	0.29	13.30	1.69
Wt. Avg.		23.98	40.53	1.87	1.50	0.63	0.78	0.14	2.89	5.64	0.36	13.10	1.69

Table P-VIII (continued)

ID	Weight %	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	F	CO_2	Org C	A.R. Insol	CaO/ P_2O_5
BM2F	15.0	28.92	43.53	0.75	0.94	0.76	0.66	0.11	3.60	5.56	1.30	5.76	1.51
BM2S	85.0	27.34	43.82	0.46	2.06	0.60	0.69	0.13	3.35	4.64	0.82	10.32	1.60
Wt. Avg.		27.58	43.78	0.51	1.89	0.63	0.68	0.13	3.39	6.95	0.89	9.64	1.59
BM7F	17.0	21.21	36.32	2.89	1.23	0.66	0.69	0.14	2.69	9.46	1.54	11.28	1.71
BM7S	83.0	23.95	40.20	1.26	1.52	0.64	0.82	0.14	2.90	5.80	1.27	16.32	1.68
Wt. Avg.		23.48	39.54	1.54	1.47	0.65	0.80	0.14	2.86	5.59	1.31	15.46	1.68
BT1F	10.0	26.24	41.63	1.59	1.00	0.79	0.61	0.12	3.20	7.30	0.66	9.50	1.59
BT1S	90.0	28.07	44.70	0.46	1.64	0.85	0.71	0.14	3.40	4.62	0.73	9.46	1.59
Wt. Avg.		27.89	44.40	0.58	1.58	0.84	0.70	0.14	3.38	6.60	0.72	9.46	1.59
BT4F	30.0	18.18	36.65	5.47	1.33	1.36	0.61	0.23	N.A.	9.74	1.62	13.20	2.02
BT4S	70.0	22.15	39.28	2.98	1.44	0.60	0.71	0.13	2.80	9.20	1.24	12.20	1.77
Wt. Avg.		20.96	38.49	3.73	1.41	0.83	0.68	0.16	1.96	2.80	1.35	12.50	1.85
BW1F	10.0	28.71	42.38	0.56	1.12	0.85	0.62	0.14	3.35	7.02	0.09	8.76	1.48
BW1S	90.0	29.02	46.17	0.38	1.80	0.66	0.66	0.10	3.50	4.45	0.86	8.66	1.59
Wt. Avg.		28.99	45.79	0.40	1.73	0.68	0.66	0.10	3.49	6.85	0.79	8.67	1.58
BW4F	16.0	11.26	31.29	8.36	1.37	0.70	0.39	0.11	1.25	21.66	1.06	14.04	2.78
BW4S	84.0	23.02	38.62	1.03	2.09	0.60	0.73	0.14	2.85	5.50	1.85	16.08	1.68
Wt. Avg.		21.14	37.44	2.20	1.97	0.62	0.67	0.14	2.59	4.10	1.72	15.75	1.85

Summary of the Pebble Data

The selected samples were two different types. The splits from the upper zone consisted of francolite with locked fine-grained sand (quartz, minor feldspar) and disseminated organic matter, and only traces of dolomite as surface stains. The only other minerals were minor amounts of free clay and free quartz-feldspar. The other type is assigned to the lower zone. It consists of the above but also contains dolomite as stains, inclusions and as free pebbles. Minor amounts of free pale yellow calcite are locally present. Most of the free and locked dolomite is white porous and relatively soft, but some might be mistaken for francolite because it is tan, hard, dense and compact. Much of porous relatively soft dolomite and clay might be removed by attrition scrubbing.

(1) The +1/2" fraction is a poor quality product having a very low P_2O_5 and very high MgO content.

(2) Generally, MgO content increases with hole depth, while P_2O_5 decreases with depth.

(3) Chemical analyses data of all as received -1/2" pebble products are summarized as:

P_2O_5	21.32 ± 5.86
CaO	37.56 ± 3.83
MgO	3.15 ± 3.00
Fe_2O_3	1.08 ± 0.35
Al_2O_3	0.81 ± 0.19
Na_20	0.58 ± 0.12
K_20	0.13 ± 0.02
F	2.94 ± 0.75
A.I.	14.13 ± 3.77
L.O.I.	9.10 ± 5.20
CaO/P_2O_5	1.94 ± 0.75
F/P_2O_5	0.14 ± 0.01

(4) The average chemical analysis of the selected pebble splits of the upper zone compared to the lower zone are listed:

	P_2O_5	CaO	Insol	MgO	CO_2
Upper Zone	27.0	41.1	11.5	0.6	5.3
Lower Zone	20.6	35.5	18.6	2.3	8.0

The MgO and CO_2 analyses of the Lower Zone is equivalent to 10% dolomite.

Samples of the selected splits were stained with Titan Yellow and the francolite/nonfrancolite grains hand-sorted. The nonfrancolite grains in the upper zone consisted of free clay pellets and quartz/feldspar coarse sand. The nonfrancolite minerals in the lower zone were the same as upper zone but also much free dolomite and locally minor amounts of calcite.

Average chemical analyses of the stained hand-sorted francolite of selected pebble splits of the upper zone versus lower zone are listed. If all the free nonfrancolite can be removed.

The remaining pebble product would assay as follows:

	P_2O_5	CaO	Insol	MgO	CO_2
Upper Zone	29.2	45.1	7.9	0.49	4.3
Lower Zone	24.8	40.6	12.6	1.22	5.8

Feed (16/150 mesh)

Head samples from different splits of various drill cores were analyzed for P_2O_5, CaO, MgO, Al_2O_3 and Fe_2O_3. The obtained data are given in the second annual report. The data indicate, generally, that P_2O_5 content is dependent on the hole location. However, it is apparent that in most of the cases, such content does not depend on the hole depth. On the other hand, elemental concentration, e.g. Ca, Mg, Al, and Fe increase with depth. The ranges of the obtained data can be summarized as:

P_2O_5	6.80	±1.58
CaO	12.25	±2.22
MgO	0.39	±0.35
Fe_2O_3	0.38	±0.14
Al_2O_3	0.26	±0.06
A.I.	73.27	±4.84
CaO/P_2O_5	1.83	±0.18
MgO/P_2O_5	0.06	±0.05

Chemistry of size fractions from the selected holes was determined as shown in the second annual report. The data clearly show higher concentrations of P_2O_5 in the coarser sizes (16-35 mesh) as compared to finer material (-35 mesh). It should be mentioned here that the weight distribution of these size fractions indicate the fine fractions (-35 mesh) are higher than the coarse sizes. The size distribution of feed samples from all splits are given in the second annual report.

Description of Feed Samples is shown in Table F-1 The francolite samples in the upper matrix are mainly white to tan to brown in color with only minor amounts of gray to black. The francolite in BI1 is lighter in color than any of the other samples. Dolomite was not detected in any of the upper feeds except BB1. The upper matrix feeds consist of roughly 25% light colored francolite and 75% quartz sand.

The francolite in the lower matrix feeds is mainly gray to black in color with minor amounts of tan to brown. However, the color of the francolite in feed BT4 is different, the coarser francolite is gray to black, and the finer francolite is tan to brown. All splits contain minor amounts of light colored dolomite. Feeds in all the selected splits, except BT4, contain trace amounts of clear gypsum crystals, usually intergrown with each other. In BW4 the intergrown gypsum crystals are often associated with pyrite. The average lower matrix feeds consist of roughly 75% quartz sand, 25% dark colored francolite, a minor amount of light colored dolomite, and locally trace amounts of gypsum.

Table F-I

Description - Feed Samples

Upper Zone	
BB1	Francolite, mainly tan, minor black, earthy to polished,rounded; some white dolomite; much quartz-feldspar sand
BG2	Francolite, buff to tan and gray, rounded, polished; much quartz-feldspar sand
BI1	Francolite, white to buff, minor gray, rounded; much quartz-feldspar sand
BM2	Francolite, tan to brown and gray, rounded, polished; much quartz-feldspar sand
BW1	Francolite, tan, minor gray, rounded, polished; much quartz-feldspar sand
Lower Zone	
BB5	Francolite, gray to black, minor tan to brown, rounded, polished; minor amount white dolomite; trace amounts clear intergrown gypsum crystals; much quartz-feldspar sand.
BD3	Francolite, gray to black, rounded, polished; minor white dolomite; trace clear gypsum, usually as intergrown crystals; much quartz-feldspar sand
BG4	Francolite, gray to black, rounded, polished; minor white dolomite; few clusters of gypsum crystals; much quartz-feldspar sand.
BI4	Francolite, gray to black, rounded, polished; minor light colored dolomite; trace clear gypsum crystals; much quartz-feldspar sand
BM7	Francolite, dominantly gray to back, minor buff to brown, rounded, polished; minor amounts light colored dolomite; trace gypsum crystals; much quartz-feldspar sand
BT4	Francolite, finer size grains, buff to tan and coarser are gray to black, rounded, polished; minor white dolomite; much quartz-feldspar

BW4	Francolite, gray to black, minor buff to brown, rounded, polished; minor light colored dolomite; trace gypsum crystals sometimes associated with pyrite, much quartz-feldspar sand.

* The sand in all samples is dominantly quartz with minor amounts of feldspar.

Flotation concentrate

Chemical data for flotation concentrates are given in the second annual report. Considering all data (from all holes and all splits) the following summary may be applied to describe the quality of the flotation concentrate.

Summary of the Chemical Analysis of Flotation Concentrate

P_2O_5	29.96	±	1.73
CaO	45.89	±	1.47
MgO	0.77	±	0.38
Fe_2O_3	1.34	±	0.44
Al_2O_3	0.72	±	0.16
Na_2O	0.74	±	0.12
K2O	0.16	±	0.03
F	3.41	±	0.27
CO_2	4.85	±	0.81
A.I.	4.08	±	2.20
L.O.I.	7.45	±	0.94
P_2O_5 Recovery	82.63	±	10.19
CaO/P_2O_5 Ratio	1.53	±	0.05
MgO/P_2O_5 Ratio	0.026	±	0.014

The following summary describes the average analysis of concentrates from the upper zone.

Summary of the Chemical Analysis of Flotation Concentrate from the Upper Zone Matrix

P_2O_5	30.70	±	1.72
CaO	46.40	±	1.35
MgO	0.70	±	0.37
Fe_2O_3	1.33	±	0.47
Al_2O_3	0.74	±	0.21
Na_2O	0.68	±	0.12
K_2O	0.15	±	0.03
F	3.45	±	0.29
CO_2	4.66	±	0.67
A.I.	3.79	±	1.76
L.O.I.	7.24	±	0.98
P_2O_5 Recovery	80.43	±	10.54
CaO/P_2O_5 Ratio	1.52	±	0.05
MgO/P_2O_5 Ratio	0.023	±	0.011

Also, the characteristics of the flotation concentrates from the lower zone matrix can be summarized as follows:

Summary of the Chemical Analysis of Flotation Concentrate from the Lower Zone Matrix

P_2O_5	26.36	±	1.51
CaO	45.47	±	1.45
MgO	0.83	±	0.39
Fe_2O_3	1.34	±	0.42
Al_2O_3	0.71	±	0.10
Na_20	0.79	±	0.11
K_20	0.17	±	0.01
F	3.38	±	0.24
CO_2	5.01	±	0.88
A.I.	4.32	±	2.51
L.O.I.	7.63	±	0.88
P_2O_5 Recovery	84.42	±	9.69
CaO/P_2O_5 Ratio	1.55	±	0.04
MgO/P_2O_5 Ratio	0.031	±	0.021

The above data suggest that lower recoveries and concentrates of lesser quality (higher MgO content) are obtained from the lower zone matrix when compared to the upper zone matrix.

Concentrates from the selected splits from the upper and lower matrix samples were examined and described using binocular and petrographic microscopes. Chemical analyses were also run on these selected samples.

Microscopic Analyses

Description of Concentrate Samples is given in Table C-I. The francolite in the upper matrix samples is mainly light in color, with some dark, and with the exception of BB18 free quartz-feldspar sand is the only gangue noted. Concentrate BB18 contains about 1½ % free light colored dolomite. Sample B118 is the lightest in color of all the upper matrix concentrates and also has the highest P_2O_5 content.

The lower matrix francolite is mainly dark in color, and contains minor amounts of free quartz feldspar sand, trace to minor amounts of free light colored dolomite and traces of free clear aggregates of gypsum crystals. The gypsum in sample BW48 is often associated with pyrite.

Table C-1

Description - Concentrate Samples

Upper Zone	
BB1	Francolite, white to buff to tan, minor dark gray, some tan translucent; scattered soft white free dolomite consisting of aggregates of very fine crystals; scattered free sand.
BG2	Francolite, white to tan and dark gray; scattered free insol.
BI1	Francolite, mainly white, minor brown and dark gray to black, scattered red sand.
BM2	Francolite, mainly tan and gray, minor white; scattered free sand.
BW1	Francolite, tan, white and gray; considerable free sand.

Lower Zone

BB5	**Francolite, gray to black, minor buff to brown; minor free dolomite, white, soft, aggregates of fine crystals; trace amounts clear intergrown gypsum crystals; scattered free sand.**
BD3	**Francolite, gray to black, minor buff to brown; minor free dolomite; trace amounts clear intergrown gypsum crystals; scattered free sand.**
BG4	**Francolite, dark gray, minor amount white to buff; minor to trace amount of gypsum, aggregates of clear crystals; scattered free sand.**
BI4	**Francolite, mainly gray, some white to brown; trace amounts clear gypsum; trace free dolomite; trace amounts clear intergrown gypsum crystals; considerable free sand.**
BM7	**Francolite dominantly dark gray; trace free dolomite; minor gypsum as intergrowths of clear crystals; scattered free sand.**
BT4	**Francolite, tan and gray; minor free dolomite; scattered free sand.**
BW4	**Francolite, gray; trace free white dolomite; intergrown gypsum crystals often associated with pyrite; considerable free sand.**

Empirical Formula Calculation

Chemical analysis of the concentrate product was used to estimate the empirical formula of the phosphate mineral (francolite) in the feed. Weight percent of each element's oxide (corrected to be free of insoluble material) was converted to weight percent of the element. Elemental weight percents were divided by the atomic weight of the element to convert the values to moles. Multiplying the moles of each element by ten gives the number of atoms of each element. Ionic charge times the number of atoms or ions is the charge contribution from the ion of the element.

Oxide Wt. % x conversion factor = Elemental Wt. %

Elemental Wt. %/Atomic Wt. = Moles of Element

Moles of Element x 10 = Number of Atoms of the Element

Number of Atoms x Charge per Atom = Charge Contribution

After the number of atoms was calculated, cations were summed and anions were summed. Charges contributed by cations and anions were summed separately. Ionic charge difference was determined by subtracting the total anion charge from the total cation charge. The result is a positive number if the anion charge is larger.

Magnesium and calcium were adjusted to reflect the contribution from dolomite in the concentrate product. Splits with calcite were not used in the calculation of the overall francolite formula.

Data used to calculate the formulas were a combination of concentrations measured chemically and the values for CO_2 calculated from x-ray diffraction data. Concentration of CO_2 measured in an apatite crystal lattice and change in relative angular location of the 004 and 410 reflections are linearly correlated. The formula used for the calculation was derived by Mathews and Nathan (1977)[1]. Delta 2-Theta for 004 reflection - 410 reflection was measured from x-ray diffraction data. Their formula calculated CO_2 values which compared very well with the values measured by ascarite absorption of the CO_2 from a mild HCl digestion of the concentrate product.

An average formula for the top split from all the cores was calculated. A separate empirical formula for the francolite in the other splits was calculated. There is no significant differences between the formula for the upper splits and the formula calculated for the remaining splits. These formulas were consistent with formulas generated by other authors.

Average Formula for Francolite in the top split:

FORMULA WEIGHT 1019

$$Ca_{9.75}\ Mg_{0.08}\ Na_{0.35}\ (PO_4)_{5.10}\ (CO_3)_{1.15}\ (SO_4)_{0.25}\ F_{2.15}$$

TOTAL CATIONS 10.18 TOTAL POSITIVE CHARGE 20.0
TOTAL ANIONS 8.65 TOTAL NEGATIVE CHARGE 20.3

CHARGE DEFICIT -0.3

---

[1] Mathews, A. and Nathan Y., 1977. The Decarbonation of Carbonate-Fluorapatite (Francolite); American Mineralogist Vol 62 pp. 565-573.

Average Formula for Francolite in the Lower Splits:

FORMULA WEIGHT 1023

$Ca_{9.67}\ Mg_{0.08}\ Na_{0.28}\ (PO_4)_{5.12}\ (CO_3)_{1.17}\ (SO_4)_{0.26}\ F_{2.18}$

TOTAL CATIONS 10.03	TOTAL POSITIVE CHARGE 20.0
TOTAL ANIONS 8.73	TOTAL NEGATIVE CHARGE 20.4

CHARGE DEFICIT -0.4

Heavy Minerals

The heavy mineral fractions from the composite feeds and their flotation products of the upper and lower zones were examined using both binocular and petrographic microscopes. The low total weight percent of heavy minerals present, their relatively low concentration in the amine tailings, and the low content of heavies of potential value indicate recovery of heavies as a by-product would not be economically feasible.

Composites were made of the selected splits from the upper and lower matrix feeds to determine the amount, distribution, composition and economic potential of the heavy minerals. Standard laboratory flotation tests were made on these composite upper and lower feeds yielding a concentrate, rougher tailings, and amine tailings. These three flotation products plus a composite feed from both the upper and lower matrixes were dissolved in HCl to remove the carbonates and francolite. The fine suspended organic matter was decanted. The heavy minerals were separated from the quartz-feldspar sand residue using tetrabromoethane (Sp.Gr. 2.95). The heavy fractions were recovered and weighed. Optical microscopy studies were made on the separated heavies from the feed, concentrate, and amine tailings of the lower composite; and the heavies in the concentrate and amine tailings in the upper zone. The microscopic studies and percentages of heavies present in the fractions indicated the amount of valuable heavies were far too small to offer economic potential. Separations of the other fractions, therefore did not seem warranted.

Table HM-I lists Heavy Mineral Distribution. This table indicates the upper zone contains about 0.18 wt % heavy minerals, and the lower zone 0.13 wt% . The heavies in the upper matrix are concentrated in the amine tailings which make up 4.3 % of the upper feed and contain 53 % of the heavies.

Table HM-I

Heavy Mineral Distribution

Upper Zone Composite

Fraction	% in Feed	% HM	% of total HM
Concentrate	20	0.31	34
Amine Tail	4	2.19	53
Rougher Tail	76	0.03 *	13 *
	100	0.18 *	100 *
Feed	100	0.18 *	

Lower Zone Fraction

Fraction	% in Feed	% HM	% of total HM
Concentrate	27	0.31	64
Amine Tail	5	0.57	20
Rougher Tail	69	0.03 *	16 *
	100	0.13 *	100 *
Feed	100	0.13	

* Estimated from optical microscopy

The amine tailings in the lower matrix make up 4.6% of the feed, but contain only 0.57% heavy minerals which amounts to only 20% of the total heavy minerals content. A much higher percentage of the heavies in the lower composite are in the concentrate, which are probably not available for recovery using current fertilizer technology.

Table HM-II lists the Heavy Mineral Composition of the fractions that were separated using tetrabromoethane. The mineralogy was determined using both binocular and petrographic microscopes. The only valuable heavy minerals noted were rutile-ilmenite and zircon, with minor to trace amounts of monazite also probably present. About 16% of the heavy mineral in the upper matrix amine tailings can be considered valuable, while 25 % of the heavy minerals in the lower matrix amine tailings are considered valuable.

Table HM-II

Heavy Mineral Composition

	Upper Matrix		Lower Matrix		
	Conc	AmTail	Feed	Conc	AmTail
Rutile-Ilmenite	22%	10%	19%	29%	17%
Zircon	7	6	5	7	8
Pyrite	3	-	14	11	5
Garnet	9	10	10	12	9
Staurolite	23	23	17	14	20
Tourmaline	10	12	7	8	13
Sillimanite	13	30	16	14	20
Epidote	13	9	12	2	8
Other	-	-	-	3	-
	100%	100%	100%	100%	100%

To put these heavy mineral analyses into economic perspective, let us start with 1,000,000 tons of upper and 1,000,000 tons lower matrix.

		Tons	
		Upper	Lower
Start matrix			
70% feed		1,000,000	1,000,000
4.5% amine tailings		700,000	700,000
2.2% HM upper, 0.57% HM lower		31,500	31,500
Valuable heavies	16% upper	700	180
	25% lower	110	50

From the 1,000,000 tons of matrix, there are about 110 tons of heavies in the upper and 50 tons in the lower matrix, and this is calculated at 100% recovery. The costs of recovering these valuable heavy mineral concentrates would far exceed their value.

Phosphatic Clays

Chemistry and Mineralogy

The chemistry of samples representing all the -150 mesh fractions from all splits are given in the second annual report.

The average of all data can be summarized as:

P_2O_5	3.10		variance is very large
CaO	15.70	±	5.20
MgO	8.60	±	5.20
Fe_2O_3	1.29	±	0.48
Al_2O_3	2.29	±	1.37
A.I.	35.43	±	13.20

Considering some top splits to represent the upper matrix, the following chemistry describes the phosphatic clays from the upper matrix:

P_2O_5	5.10	±	4.60
CaO	17.10	±	4.70
MgO	7.30	±	6.10
Fe_2O_3	1.20	±	0.50
Al_2O_3	2.80	±	2.00
A.I.	35.00	±	14.20

Using the remainder of the splits to represent the lower zone, the chemistry of the phosphatic clays generated from the lower zone matrix may be described by:

P_2O_5	1.80	±	1.40
CaO	14.90	±	5.40
MgO	9.40	±	4.40
Fe_2O_3	1.20	±	0.48
Al_2O_3	1.80	±	0.60
A.I.	36.56	±	12.90

The above summaries clearly indicate that the phosphatic clays from the upper matrix are of higher P_2O_5 content and lower MgO content than the phosphatic clays from the lower matrix.

Mineralogical, chemical and X-ray analyses were conducted on the selected samples of phosphatic clays from the upper and lower matrix zones. Six samples are from the upper and seven from the lower zones. The upper zone samples end in 1 or 2 which means they are the topmost splits in each core hole. The other splits examined are in the lower zone.

The samples were sized at 44 microns by screening at 325 mesh, and at 2 microns by differential settling in graduated cylinders. X-ray diffraction and chemical analyses were run on the as received phosphatic clays and the sized fractions: +44, 2 X 44, and nominal minus 2 micron. More detailed X-ray and chemical analyses were made on the minus 2 micron material where the clay minerals are concentrated. Chemical analysis methods include determination of the aqua regia soluble components as well as total (fusion) analysis. The sized fractions were also examined using optical microscopy.

The unsized phosphatic clay fines were chemically analyzed by aqua regia digestion. The results, given in Table PC-I, show that the upper zone fines are higher in P_2O_5 (francolite) and lower in MgO and CO_2 (dolomite) than the lower zone fines.

Analyses by aqua regia digestion Were also made on the three sized fractions of the phosphatic clays: +44, -44+2, and -2 microns. These results are shown in Table PC-II. It should be noted that the 44 X 2 micron fraction is higher in MgO and CO_2 for both upper and lower matrix samples. This indicates more dolomite in this size than in others and that there is considerably more dolomite in the lower zone. The upper zone is higher in P_2O_5 in all size fractions.

The minus 2 micron clay rich fraction was separately analyzed for total (fusion) components. These results are presented in Table PC-III. They indicate the upper zone is higher in francolite, lower in dolomite, and that this zone contains clays higher in $A1_2O_3$ than the lower zone.

Table PC-I

Unsized Clays/Phosphatic clays Chemistry (Aqua Regia)
Reported Values as Percents

Sample ID	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	CO_2	L.O.I.	A.R. Insol
Upper Zone								
BB1	1.50	19.87	11.84	0.77	1.15	27.32	34.86	26.12
BG2	6.72	10.44	0.73	0.60	3.04	1.60	12.17	53.00
BI1	15.08	20.27	0.73	0.84	6.31	2.08	12.50	36.06
BM2	7.41	12.28	2.70	1.86	3.21	3.42	16.64	48.56
BT1	8.03	13.42	2.26	1.86	4.44	4.12	17.97	45.62
BW1	9.60	14.19	1.06	2.20	5.39	2.38	15.14	48.02
Average	8.06	15.08	3.22	1.36	3.92	6.82	18.21	42.90
Lower Zone								
BB5	0.67	13.77	10.99	0.94	1.74	21.30	30.39	38.20
BD3	1.40	14.34	10.36	1.13	1.74	19.60	29.84	36.48
BG4	4.85	9.99	3.23	2.63	2.65	5.40	16.88	52.48
BI4	1.79	10.30	7.23	1.54	2.25	14.12	24.64	46.34
BM7	1.19	13.70	10.30	1.06	1.95	19.64	29.62	37.48
BT4	1.52	12.17	8.56	1.02	1.97	21.44	28.16	42.48
BW4	0.21	14.51	10.55	1.40	1.53	21.40	30.68	35.92
Average	1.66	12.68	8.75	1.39	1.97	17.56	27.17	41.34

Table PC-II

Analysis of Aqua Regia Soluble Material

Phosphatic Clay Chemistry by Split and Particle Size (in microns)

Upper Zone

SPLIT B1	Wt. %	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	CO_2	LOI	Insol
+44	0.60	6.80	16.37	1.09	N.A.	N.A.	N.A.	N.A.	59.04
44X2	66.90	0.80	29.06	17.74	0.51	0.36	41.24	42.63	6.82
-2	32.50	2.54	7.61	4.08	1.67	3.33	6.80	22.61	55.40
Comp	100.00	1.40	22.01	13.20	0.89	1.32	29.80	35.87	22.92
SPLIT G2									
+44	2.10	6.68	11.35	0.28	N.A.	N.A.	N.A.	N.A.	73.94
44X2	26.30	9.72	17.88	2.60	0.54	0.93	7.80	8.38	55.76
-2	71.60	5.48	7.84	0.65	0.88	5.29	1.79	15.52	61.29
Comp	100.00	6.62	10.56	1.16	0.77	4.03	3.33	13.31	60.10
SPLIT I1									
+44	1.50	20.00	27.59	0.30	N.A.	N.A.	N.A.	N.A.	38.44
44X2	15.90	21.58	28.24	0.76	1.14	5.57	3.44	8.75	27.32
-2	82.60	14.52	17.53	0.91	1.43	4.52	1.87	14.17	35.89
Comp	100.00	15.72	19.38	0.88	1.36	4.62	2.09	13.10	34.57
SPLIT M2									
+44	3.60	9.91	15.67	0.32	N.A.	N.A.	N.A.	N.A.	61.42
44X2	22.40	13.76	23.88	3.52	1.49	2.04	8.56	12.37	37.48
-2	74.00	5.67	8.84	2.62	2.59	4.76	1.36	18.44	53.14
Comp	100.00	7.63	12.46	2.74	2.25	3.98	2.92	16.42	49.93
SPLIT T1									
+44	1.60	11.64	17.22	0.86	N.A.	N.A.	N.A.	N.A.	53.28
44X2	13.70	12.96	27.65	5.36	1.39	2.04	14.08	16.38	28.64
-2	84.70	8.11	12.14	1.58	2.64	6.28	3.30	17.05	46.68
Comp	100.00	8.84	14.35	2.08	2.43	5.60	4.72	16.68	44.32
SPLIT W1									
+44	1.50	6.72	10.68	0.73	N.A.	N.A.	N.A.	N.A.	70.78
44X2	20.50	11.88	20.43	2.01	2.53	4.50	4.88	11.64	41.68
-2	78.00	9.83	14.38	1.13	2.56	7.12	1.50	15.62	45.27
Comp	100.00	10.20	15.56	1.30	2.52	6.47	2.17	14.57	44.92

UPPER Avg.

+44	1.8	10.29	16.48	0.60	N.A.	N.A.	N.A.	N.A.	59.48
44X2	27.6	11.78	24.52	5.33	1.27	2.57	13.33	16.69	32.95
-2	70.6	7.69	11.39	1.83	1.96	5.22	2.77	17.23	49.61
Comp.		8.40	15.72	3.56	1.70	4.34	7.51	18.32	42.79

Analysis of Aqua Regia Soluble Material
Phosphatic Clay Chemistry by Split and Particle Size

LOWER ZONE

SPLIT B5

	Wt. %	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	CO_2	LOI	Insol
+44	1.50	5.10	14.08	4.11	N.A.	N.A.	N.A.	N.A.	61.20
44X2	51.20	0.68	24.37	17.46	0.93	0.59	37.80	38.74	15.46
-2	47.30	1.30	3.12	5.21	1.47	4.14	3.00	21.32	60.42
Comp	100.00	1.04	14.17	11.47	1.17	2.26	20.77	29.92	37.41

SPLIT D3

+44	1.90	5.30	9.91	1.14	N.A.	N.A.	N.A.	N.A.	75.21
44X2	52.90	0.84	24.93	17.41	1.20	0.60	38.44	37.97	14.12
-2	45.20	2.31	4.58	4.29	1.43	4.25	3.12	21.26	58.08
Comp	100.00	1.59	15.45	11.17	1.28	2.24	21.75	29.70	35.15

SPLIT G4

+44	6.00	6.20	12.09	0.81	N.A.	N.A.	N.A.	N.A.	49.39
44X2	32.00	7.80	16.40	4.86	4.92	2.74	10.12	15.20	40.26
-2	62.00	4.41	5.51	2.79	2.69	5.03	1.04	19.06	56.24
Comp	100.00	5.60	9.39	3.33	3.24	3.99	3.88	16.68	50.72

SPLIT I4

+44	1.70	7.17	11.26	0.41	N.A.	N.A.	N.A.	N.A.	72.75
44X2	45.50	2.00	18.44	12.34	2.34	1.76	24.28	30.00	29.58
-2	52.80	2.79	4.03	3.83	1.67	4.89	1.32	20.25	58.98
Comp	100.00	2.51	10.71	7.64	1.95	3.38	11.74	24.34	45.84

SPLIT M7

+44	2.40	7.72	15.15	2.14	N.A.	N.A.	N.A.	N.A.	61.42
44X2	39.30	1.28	25.90	17.45	0.99	0.43	37.88	38.15	13.42
-2	58.30	1.95	5.96	6.35	1.60	4.25	6.68	22.97	52.80
Comp	100.00	1.83	14.02	10.61	1.32	2.65	18.78	28.38	37.53

SPLIT T4

+44	2.20	6.74	14.02	2.34	N.A.	N.A.	N.A.	N.A.	61.43
44X2	51.40	1.61	22.43	14.36	0.94	1.08	33.44	35.30	21.48
-2	46.40	1.82	3.64	4.31	1.67	4.10	2.56	21.82	59.12
Comp	100.00	1.82	13.53	9.43	1.26	2.46	18.38	28.27	39.82

SPLIT W4

+44	1.20	8.15	16.62	3.48	N.A.	N.A.	N.A.	N.A.	49.05
44X2	31.90	0.80	27.76	17.74	0.80	0.34	39.96	40.80	9.90
-2	66.90	1.42	8.79	7.39	2.19	3.23	11.48	25.04	48.33
Comp	100.00	1.30	14.94	10.64	1.72	2.27	20.43	29.76	36.08

LOWER Avg.

+44	2.4	6.63	13.30	2.06	N.A.	N.A.	N.A.	N.A.	61.49
44X2	43.5	2.14	22.89	14.52	1.73	1.08	31.70	33.74	20.60
-2	54.1	2.29	5.09	4.88	1.82	4.27	4.17	21.67	56.28
Comp.		2.24	13.17	9.19	1.71	2.75	16.53	26.72	40.36

Analysis of Aqua Regia Soluble Material
Phosphatic Clay Chemistry Averages by Size

	Wt. %	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	CO_2	LOI	Insol
+44		8.46	14.89	1.33	N.A.	N.A.	N.A.	N.A.	60.49
44X2		6.96	23.71	9.92	1.50	1.82	22.52	25.21	26.78
-2		4.99	8.24	3.35	1.89	4.74	3.47	19.45	52.95

GRAND AVERAGES FOR PHOSPHATIC CLAY CHEMISTRY

P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	CO_2	LOI	Insol
5.32	14.45	6.37	1.70	3.54	12.02	22.52	41.58

Table PC-III

Analysis of Fusion Products

Phosphatic Clay Chemistry of Minus 2 Micron Fraction by Split

Upper Zone

	Wt. %	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	K_2O	CO_2	SiO_2	LOI
BB1	32.50	2.29	7.28	7.40	4.58	11.58	0.77	6.80	42.15	22.61
BG2	71.60	4.64	5.93	1.91	2.86	19.25	0.78	1.44	42.25	18.47
BI1	82.60	14.16	18.15	1.59	2.37	16.42	0.68	1.87	29.26	14.17
BM2	74.00	5.32	8.65	5.21	5.58	11.15	0.86	1.36	43.50	18.44
BT1	84.70	8.15	12.22	3.16	5.24	13.16	0.72	3.30	38.33	17.05
BW1	78.00	9.18	13.57	2.40	4.93	13.85	0.84	1.48	37.35	15.85
Upper Avg.	70.60	7.38	11.20	3.63	4.20	14.76	0.78	2.83	38.70	17.61

Lower Zone

	Wt. %	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	K_2O	CO_2	SiO_2	LOI
BB5	47.30	1.05	3.34	9.40	3.07	9.01	1.06	3.00	56.80	21.32
BD3	45.20	2.08	4.97	8.21	3.22	10.64	1.26	3.12	44.95	21.26
BG4	62.00	4.09	6.13	5.61	4.72	11.58	1.16	1.04	44.60	19.06
BI4	52.80	2.52	4.39	7.60	3.50	11.51	1.23	1.32	50.75	20.25
BM7	58.30	1.71	6.44	10.60	3.07	9.13	0.49	6.68	47.10	22.97
BT4	46.40	1.57	3.88	8.61	3.65	11.02	0.96	2.56	51.55	21.82
BW4	66.90	1.29	8.80	12.01	4.29	8.56	1.04	11.48	40.24	25.04
Lower Avg.	54.10	2.04	5.42	8.86	3.65	10.21	1.03	4.17	48.00	21.67
Upper & Lower Avg.	62.4	4.71	8.31	6.25	3.92	12.10	0.90	3.50	43.35	19.64

The mineralogical composition of these phosphatic clays were determined from chemical, X-ray, and petrographic analysis. The determination of francolite and dolomite by these methods is rather straight forward. The clay minerals, however, because of their extremely fine particle size, intimate admixture, and somewhat similar chemical composition, present a more difficult problem.

In this report, the group mineral name smectite is used, which includes the clay mineral montmorillonite. For practical purposes montmorillonite and smectite are essentially the same for Central Florida phosphatic clays. Palygorskite is used rather than attapulgite; mineralogically they are similar.

Mineralogy of the separate sized fractions for each split from the upper and lower zones is given in Table PC-IV. Sizing the phosphatic fines samples into three fractions produced two coarser products that were largely free of clay minerals. The minus two micron fraction contained most of the clay minerals. It can be seen, in general, that smectite is the major clay in the upper zone splits. Illite is present in minor amounts in four of the six upper zone splits examined. It is questionably present in trace amounts in two of the seven lower zone splits. Positive identification of illite could not be made in some samples because of the obscuring X-ray diffraction effects of other clays.

Kaolinite is present in minor amounts in the upper zone, and in trace amounts in the lower zone. Sepiolite was detected in only one sample in the upper zone.

Table PC-IV

Estimated Mineralogy of Phosphatic Clays Mineral Composition %

Upper Zone

Sample	Size Microns	Wt. %	Francolite	Dolomite	Quartz	Clays
BB1	+44	1	21	9	70	
	2X44	67	3	90	7	
	-2	32	7	14	4	75
		100	4	65	6	24
BG2	+44	2	22	2	76	
	2X44	26	30	14	56	
	-2	72	15	1	5	79
		100	19	4	20	57
BI1	+44	2	62	0	38	
	2X44	16	70	0	30	
	-2	82	44	0	2	54
		100	48	0	7	45
BM2	+44	4	32	1	67	
	2X44	22	45	15	40	
	-2	74	17	1	5	77
		100	24	4	15	57
BT1	+44	2	36	9	55	
	2X44	14	41	27	31	
	-2	84	25	3	4	68
		100	27	6	9	58
BW1	+44	2	21	3	65	11
	2X44	20	37	6	27	30
	-2	78	30	3	5	62
		100	31	4	10	55
Average	+44	2	32	4	62	2
	2X44	28	38	25	32	5
	-2	70	23	4	4	69

Table PC-IV (cont.)

Upper Zone

Sample	Size Microns	Wt. %	Smectite	Palygorskite	Sepiolite	Illite	Kaolinite
BB1	+44	1					
	2X44	67					
	-2	32	Major	Major			
		100	Minor	Minor			
BG2	+44	2					
	2X44	26					
	-2	72	Major	Minor		Minor	Moderate
		100	Moderate	Minor		Minor	Moderate
BI1	+44	1					
	2X44	16					
	-2	83	Moderate	Trace		Minor	Minor
		100	Moderate	Trace		Minor	Minor
BM2	+44	4					
	2X44	22					
	-2	74	Major	Minor	Minor		
		100	Major	Minor	Minor		
BT1	+44	2					
	2X44	14					
	-2	84	Major			Moderate	Minor
		100	Major			Minor	Trace
BW1	+44	2	Minor			Trace	
	2X44	20	Moderate			Minor	
	-2	78	Major			Minor	Minor
		100	Major			Minor	Minor
Average	+44	2	Trace				
	2X44	28	Trace				
	-2	70	Major	Minor	Trace	Minor	Minor
		100.0	Moderate	Minor	Trace	Minor	Minor

Table PC-IV

Estimates Mineralogy of Phosphatic Clays Mineral Composition %

Lower Zone

Sample	Size Microns	Wt. %	Francolite	Dolomite	Quartz	Clays
BB5	+44	2	18	19	63	
	2X44	51	2	79	13	6
	-2	47	4	6	10	80
		100	3	44	12	41
BD3	+44	2	18	5	77	
	2X44	53	3	80	11	6
	-2	45	8	7	5	75
		100	6	46	10	37
BG4	+44	6	21	3	36	40
	2X44	32	26	21	23	30
	-2	62	15	1	10	74
		100	19	8	16	58
BI4	+44	2	24	2	74	
	2X44	45	7	51	20	22
	-2	53	9	2	9	80
		100	8	24	15	52
BM7	+44	2	27	10	63	
	2X44	39	4	79	9	8
	-2	59	6	13	11	70
		100	6	39	11	44
BT4	+44	2	23	10	57	10
	2X44	51	5	70	15	10
	-2	47	5	5	10	80
		100	5	39	14	42
BW4	+44	1	27	16	40	17
	2X44	32	3	83	5	9
	-2	67	5	24	6	65
		100	5	43	6	47
Average	+44	2	23	9	59	10
	2X44	44	7	66	14	13
	-2	54	7	8	9	75
		100.0	8	33	12	46

Table PC-IV (cont.)

Lower Zone

Sample	Size Microns	Wt. %	Smectite	Palygorskite	Sepiolite	Illite	Kaolinite
BB5	+44	2					
	2X44	51	Trace	Trace			
	-2	47	Major	Major			
		100	Moderate	Moderate			
BD3	+44	2					
	2X44	53	Trace	Trace			
	-2	45	Major	Major			
		100	Moderate	Moderate			
BG4	+44	6		Major			
	2X44	32		Moderate			
	-2	62		Minor			Minor
		100		Major			Minor
BI4	+44	2					
	2X44	45	Moderate	Moderate			
	-2	53	Major	Major			
		100	Moderate	Moderate			
BM7	+44	2					
	2X44	39	Trace	Trace			
	-2	58	Major	Major			
		100	Moderate	Moderate			
BT4	+44	2	Minor	Trace			
	2X44	51	Minor	Trace			
	-2	47	Moderate	Major		Minor?	Trace
		100	Moderate	Moderate		Trace	Trace
BW4	+44	1	Minor	Trace			
	2X44	32	Minor	Trace			
	-2	67	Moderate	Moderate		Minor?	
		100	Moderate	Moderate		Minor?	
Average	+44	2	Trace	Trace			
	2X44	44	Trace	Minor			
	-2	54	Major	Major	Trace	Trace?	Trace
		100	Moderate	Major	Trace	Trace?	Trace

Summary of Clay Mineralogy.

The average mineral composition of the upper and lower matrix phosphatic clay fractions is given in Table PC-V. The average composition for the unsized phosphatic clay and the minus 2 micron fraction are shown, as the actual clay minerals are essentially all in the minus 2 micron fraction. In the upper matrix zone the minus 2 micron fraction makes up 71% of the phosphatic clays, in the lower matrix 54 % . Smectite is the dominant clay material in the upper matrix with minor amounts of palygorskite, illite, kaolinite, and a trace amount of sepiolite. In the lower matrix the dominant clay material is palygorskite with a moderate amount of smectite, plus trace amounts of kaolinite and possibly illite.

The non-clay minerals occur mainly in the plus 2 micron fraction. Francolite predominates in the upper matrix and dolomite is dominant in the lower. Quartz is about equal in both zones.

Table PC-V

Summary - Mineralogy of Phosphatic clays

	Unsized Sample		Minus 2 Micron Fraction	
	Upper	Lower	Upper	Lower
Francolite	27	8	23	7
Dolomite	10	33	4	8
Quartz*	13	12	4	9
Clays	50	47	69	76
Smectite	Moderate	Moderate	Major	Moderate
Palygorskite	Minor	Moderate	Minor	Major
Sepiolite	Trace	---	Trace	---
Illite	Minor	Trace	Minor	Trace
Kaolinite	Minor	Trace	Minor	Trace

* Includes minor feldspar and heavy minerals.

Consolidation Behavior

As mentioned in the experimental section, a laboratory centrifuge was used to study the consolidation behavior of the -150 mesh material representing upper and lower zone matrixes. For this purpose, only a few holes were tested.

The following models were developed to represent the consolidation behavior of different splits as indicated:

BB1

$$e_t=e_\epsilon \exp^{-kt}+2.63e_\epsilon \sigma^{-0.325}[1-\exp^{-kt}]$$
$$k=5.90*10^{-7}*N*e_\epsilon^{1.98}$$
$$K=3.92*10^{-7}e^{4.09}$$

BB5

$$e_t=e_\epsilon \exp^{-kt}+4.12e_\epsilon \sigma^{-0.334}[1-\exp^{-kt}]$$
$$k=2.96*10^{-6}*N*e_\epsilon^{1.27}$$
$$K=6.48*10^{-7}e^{4.39}$$

BG2

$$e_t=e_\epsilon \exp^{-kt}+3.99e_\epsilon \sigma^{-0.35}[1-\exp^{-kt}]$$
$$k=3.38*10^{-6}*N*e_\epsilon^{1.47}$$
$$K=1.27*10^{-6}e^{3.98}$$

BG4

$$e_t=e_\epsilon \exp^{-kt}+1.32e_\epsilon \sigma^{-0.21}[1-\exp^{-kt}]$$
$$k=3.38*10^{-6}*N*e_\epsilon^{1.10}$$
$$K=6.64*10^{-9}e^{4.88}$$

BI1

$$e_t=e_\epsilon \exp^{-kt}+4.11e_\epsilon \sigma^{-0.34}[1-\exp^{-kt}]$$
$$k=3.38*10^{-5}*N*e_\epsilon^{0.34}$$
$$K=2.77*10^{-6}e^{3.77}$$

Note: e_ϵ = initial void ratio

BI4

$$e_t=e_\epsilon \exp^{-kt}+4.26e_\epsilon \sigma^{-0.315}[1-\exp^{-kt}]$$
$$k=5.38*10^{-7}*N*e_\epsilon^{1.81}$$
$$K=5.07*10^{-7}e^{3.32}$$

BM2

$$e_t=e_\epsilon \exp^{-kt}+1.47e_\epsilon \sigma^{-0.37}[1-\exp^{-kt}]$$
$$k=3.00*10^{-5}*N*e_\epsilon^{0.29}$$
$$K=2.69*10^{-6}e^{2.73}$$

BM7

$$e_t=e_\epsilon \exp^{-kt}+7.69e_\epsilon \sigma^{-0.428}[1-\exp^{-kt}]$$
$$k=1.97*10^{-7}*N*e_\epsilon^{2.01}$$
$$K=6.99*10^{-7}e^{3.13}$$

BT1

$$e_t=e_\epsilon \exp^{-kt}+1.30e_\epsilon \sigma^{-0.23}[1-\exp^{-kt}]$$
$$k=3.50*10^{-7}*N*e_\epsilon^{1.63}$$
$$K=5.45*10^{-9}e^{4.60}$$

BT4

$$e_t=e_\epsilon \exp^{-kt}+1.84e_\epsilon \sigma^{-0.252}[1-\exp^{-kt}]$$
$$k=6.97*10^{-6}*N*e_\epsilon^{0.93}$$
$$K=3.93*10^{-7}e^{3.44}$$

BW1

$$e_t=e_\epsilon \exp^{-kt}+1.33e_\epsilon \sigma^{-0.235}[1-\exp^{-kt}]$$
$$k=1.48*10^{-6}*N*e_\epsilon^{1.41}$$
$$K=4.99*10^{-7}e^{3.88}$$

BW4

$$e_t=e_\epsilon \exp^{-kt}+5.00e_\epsilon \sigma^{-0.387}[1-\exp^{-kt}]$$
$$k=2.57*10^{-6}*N*e_\epsilon^{1.28}$$
$$K=9.32*10^{-7}e^{3.39}$$

Table PC-VI

Correlation of Phosphatic clays Mineralogy (% content) and the Consolidation Rate

	Sample Number							
Mineral	BB1	BB5	BG2	BG4	BM2	BM7	BW1	BW4
Francolite	4	3	19	19	24	6	31	5
Dolomite	65	44	4	8	4	13	4	43
Quartz	6	12	20	16	15	11	10	6
Clays	24	41	57	58	57	44	55	47
Smectite	Minor	Moderate	Moderate		Major	Moderate	Major	Moderate
Palygorskite	Minor	Moderate	Minor	Major	Minor	Moderate		Moderate
Sepiolite					Minor			
Illite			Minor				Minor	Minor
Kaolinite			Moderate	Minor			Minor	
% -2 micron	33	47	72	62	74	58	78	67
Rate of Consolidation	Fast	Slow	Fast	Slow	Slow	Fast	Fast	Slow

Using the void ratio model for an initial height of 1.0 ft. (30.5 cm), e_{in} = 20.0 and N = 30g, the following plots of height of clays vs. centrifuge time are generated and plotted in Figures 4-9. The shown data indicate that there is no consistent behavior of clays. In other words, some lower matrix phosphatic clays (clays) consolidate faster than the top cores. For instance, Core BT-4 (Figure 8) shows faster consolidation of the clays from the upper matrix. In order to explain these results, the mineralogical content of each of the studied splits are given in Table VI. In the same table, the consolidation rate is described qualitatively. These preliminary data indicate slower rates always correlate with higher clay montmorillonite and/or palygorskite.

Using the permeability models for a pond of 30 feet high (corresponding to 1.0 ft. model height and N = 30g), the following tables VII to IX show the calculated consolidation behavior as a function of time in years. It is important to note that these data correlate well with the plots from the centrifugal times.

Table PC-VII

Consolidation Characteristics of Phosphatic Clay from Hole #BB, Splits #1 and #5

Elapsed Time,years		Height,ft	Void Ratio	% Solids Weight
Split #1	Split #5			
0.00	0.00	30.00	20.00	13.00
0.02	0.10	19.30	17.00	15.00
0.14	0.50	13.90	12.00	20.00
0.34	1.04	11.80	10.00	23.10
0.96	2.49	9.60	8.00	27.30
3.44	7.29	7.50	6.00	33.30

Table PC-VIII

Consolidation Characteristics of Phosphatic Clay from Hole #BG, Splits #2 and #4

Elapsed Time,years		Height,ft	Void Ratio	% Solids Weight
Split #2	Split #4			
0.00	0.00	30.00	20.00	13.00
0.01	0.15	19.30	17.00	15.00
0.06	1.20	13.90	12.00	20.00
0.14	3.30	11.80	10.00	23.10
0.37	10.98	9.60	8.00	27.30

Table PC-IX

Consolidation Characteristics of Phosphatic Clay from Hole #BM Splits #2 and #7

Elapsed Time,years		Height,ft	Void Ratio	% Solids Weight
Split #2	Split #7			
0.00	0.00	30.00	20.00	13.00
0.16	0.12	19.30	17.00	15.00
0.62	0.56	13.90	12.00	20.00
1.15	1.16	11.80	10.00	23.10
2.37	2.73	9.60	8.00	27.30
5.73	7.80	7.50	6.00	33.30

Table PC-X

Consolidation Characteristics of Phosphatic Clay from Hole #BW Splits #1 and #4

Elapsed Time,years		Height,ft	Void Ratio	% Solids Weight
Split #1	Split #4			
0.00	0.00	30.00	20.00	13.00
0.03	0.09	19.30	17.00	15.00
0.18	0.43	13.90	12.00	20.00
0.42	0.89	11.80	10.00	23.10
1.12	2.09	9.60	8.00	27.30
3.79	5.96	7.50	6.00	33.30

Figure 4

Consolidation Characteristic of BB Clay

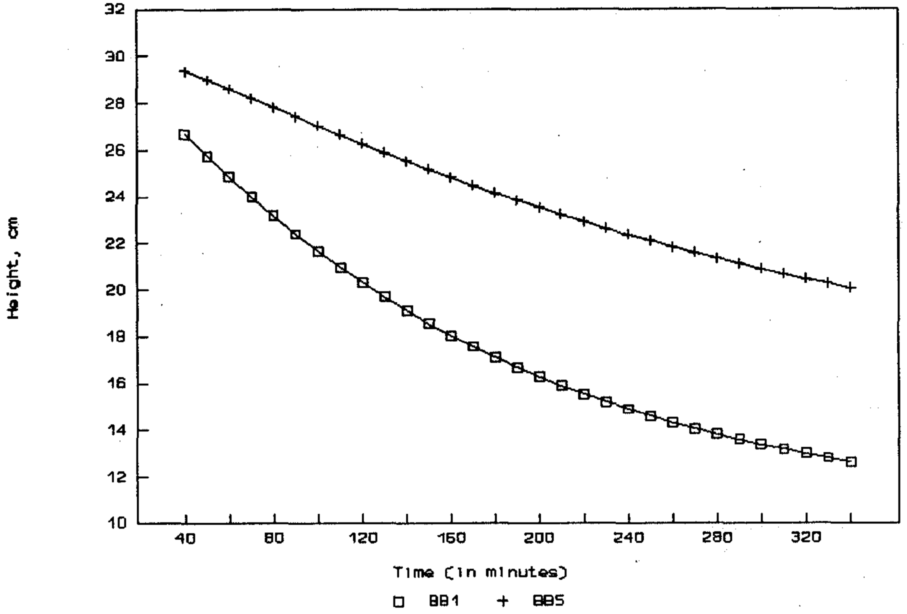


Figure 5

Consolidation Characteristic of BG Clay

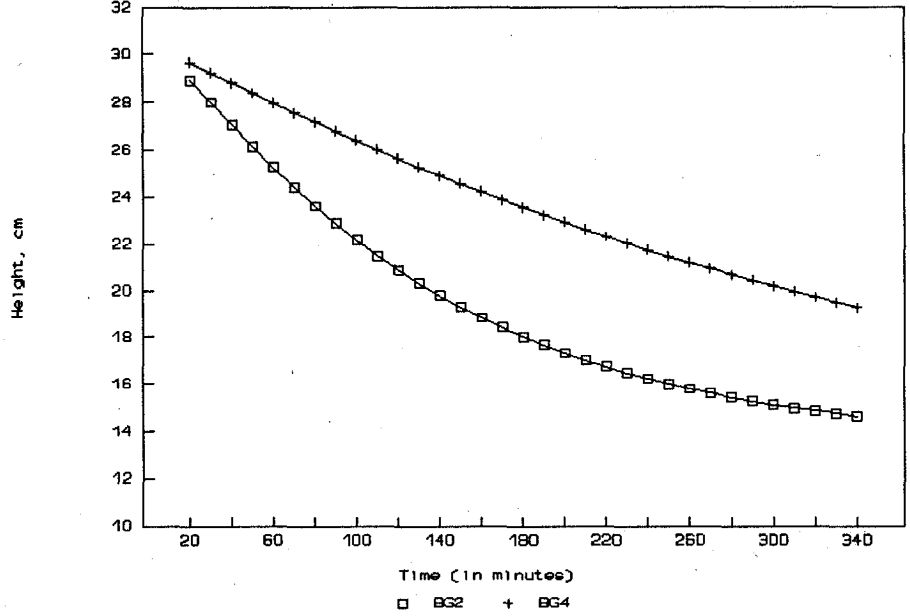


Figure 6

Consolidation Characteristic of BI Clay

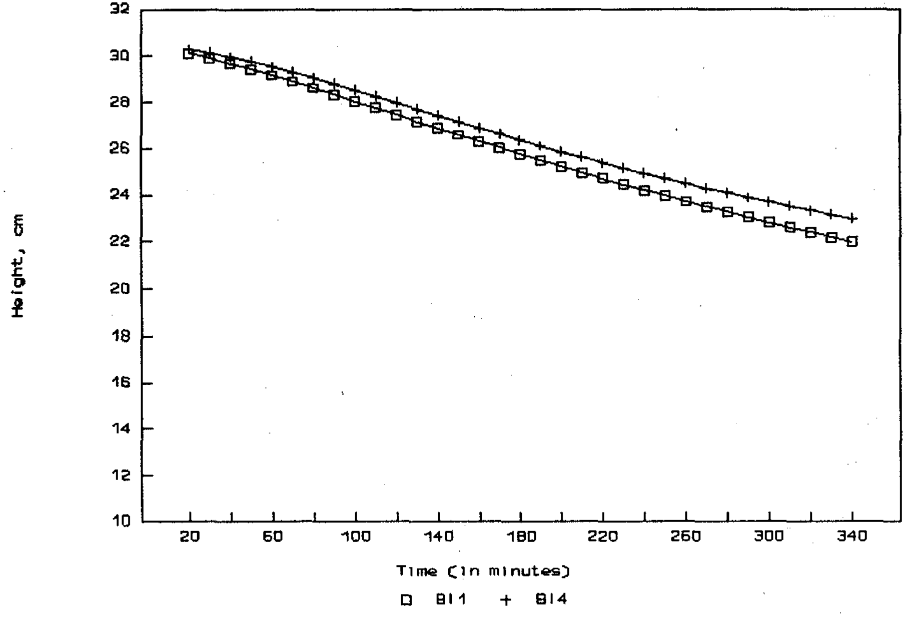


Figure 7

Consolidation Characteristic of BM Clay

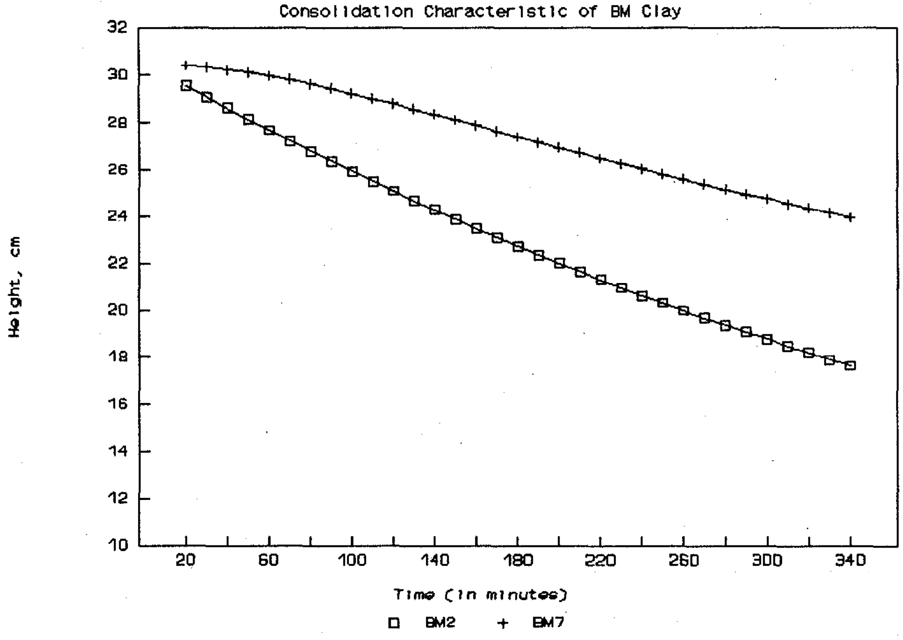


Figure 8

Consolidation Characteristic of BT Clay

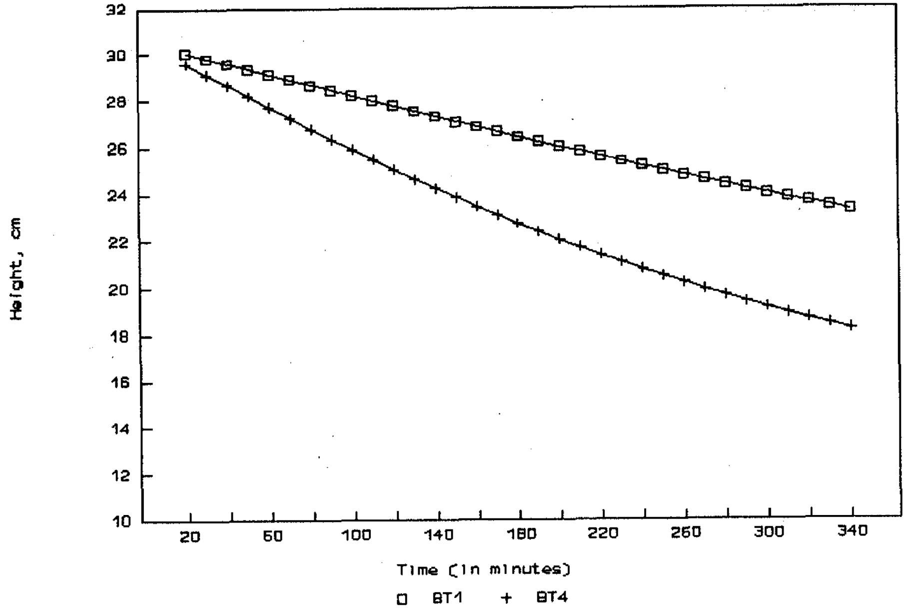
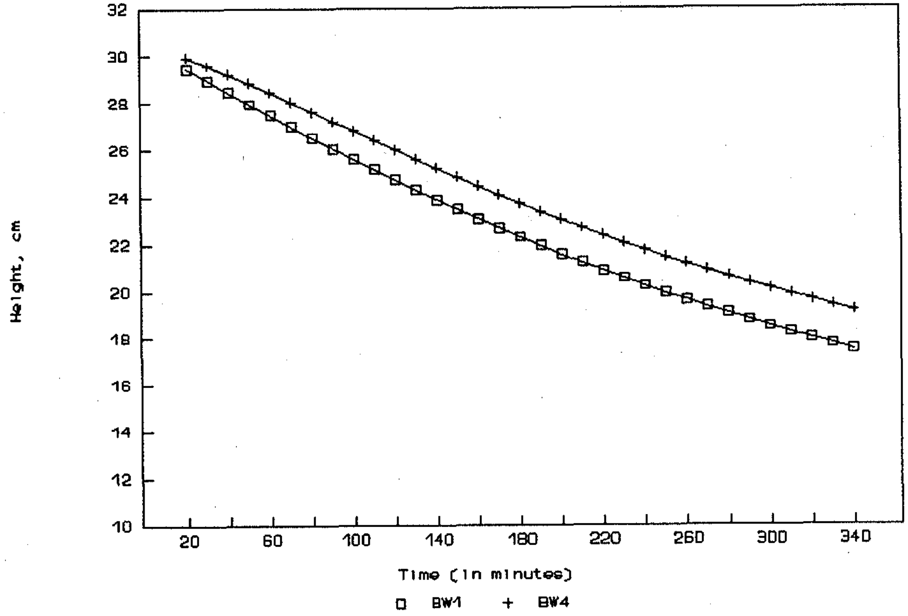


Figure 9

Consolidation Characteristic of BW Clay



Appendix IV

Data for Area C

MATRIX

Samples of material from all the as received matrix were sized at +14 mesh (pebble) 14 x 150 mesh (feed), and -150 mesh (phosphatic clay). Each size fraction was analyzed for P_2O_5. Short tons per acre foot of each sized fraction were calculated using a dry matrix density of 88 pounds per cubic foot. The results for all the size and chemical analyses are in third annual report with the analyses for the LP, Upper, and Lower Zones in third annual report. The average results with ranges for all the samples and averages and ranges differentiated into the three zones are listed below:

	Wt %		% P_2O_5		Tons/Acre Foot	
	Avg	Range	Avg	Range	Avg	Range
All Samples						
Pebble	10	Tr-36	18	1-30	196	2-692
Feed	59	26-96	7	2-12	1135	534-1845
Phos Clay	31	2-66	3	Tr-12	596	32-1271
	100		7		1927	
LP Zone						
Pebble	11	3-17	11	7-25	203	61-327
Feed	46	31-57	7	6-10	884	589-1092
Phos Clay	43	26-66	2	1-2	883	501-1271
	100		6		1920	
Upper Zone						
Pebble	15	Tr-28	28	26-30	291	2-692
Feed	70	51-96	7	5-10	1139	986-1845
Phos Clay	15	2-36	6	2-12	290	32-686
	100		10		1920	
Lower Zone						
Pebble	9	Tr-28	16	3-24	170	4-529
Feed	57	28-82	7	2-12	1094	534-1578
Phos Clay	34	14-63	2	Tr-7	664	271-1216
	100		6		1928	

The Upper Zone contains a higher percentage of pebble and feed size material with less phosphatic clay than the LP and Lower Zones. The P_2O_5 content of the pebble in the Upper Zone is much higher than the other zones.

Feeds in all three zones average 7% P_2O_5. With the exception of the P_2O_5 content in the pebble from the Upper Zone, the ranges of the listed analyses of all three zones for the pebble feed and phosphatic clay size fractions are very large.

Rheology.

Pumping of slurry depends on its rheological characteristics such as viscosity or fluidity which in turn depends on the solids content as well as on the mineralogical and physical properties of the solids.

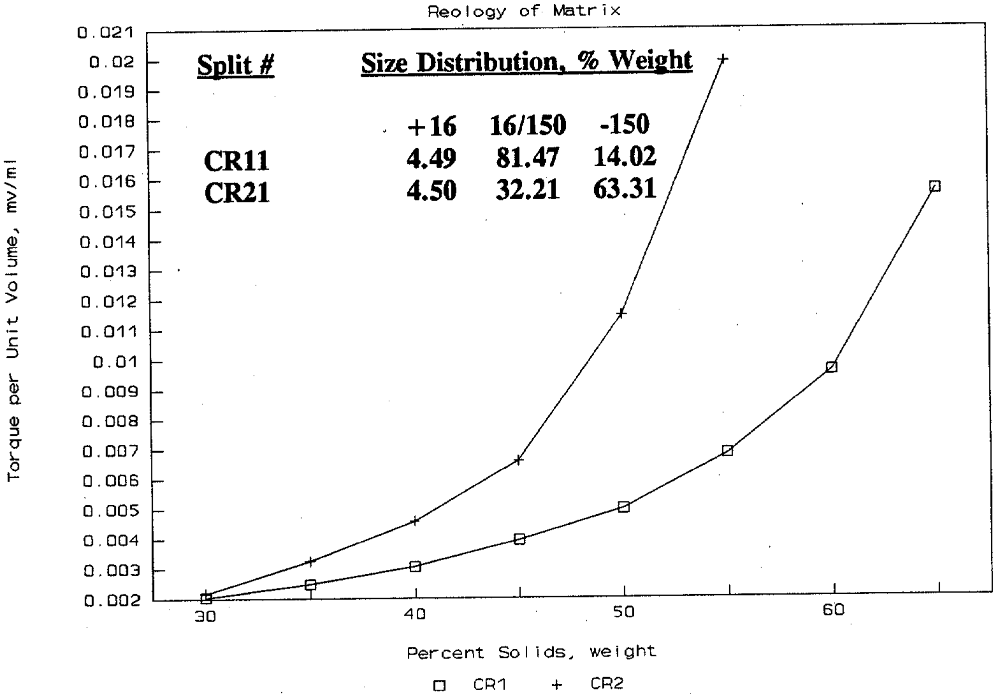
For the purpose of investigating the difference in rheology of matrix slurries from the upper and lower zone material, the torque exerted on an impeller rotating in such slurries was measured. The obtained data for matrix slurries from different splits of several drill holes are given in Figures 1-5. The size distribution of the tested samples are also given for comparison.

Generally, the data indicate that energy consumption increases exponentially with the increase in the % solids content.

Results shown in Figure 1 suggest that more energy is needed to pump slurry from split CR-2 than pumping slurry from split CR-1. This could be attributed to the presence of more fines (-150 mesh) in split CR-2 as shown by the weight distribution of different size fractions. It is also worthy to mention that more smectite and palygorskite clays are found in the matrix as compared to the matrix from split CR-1.

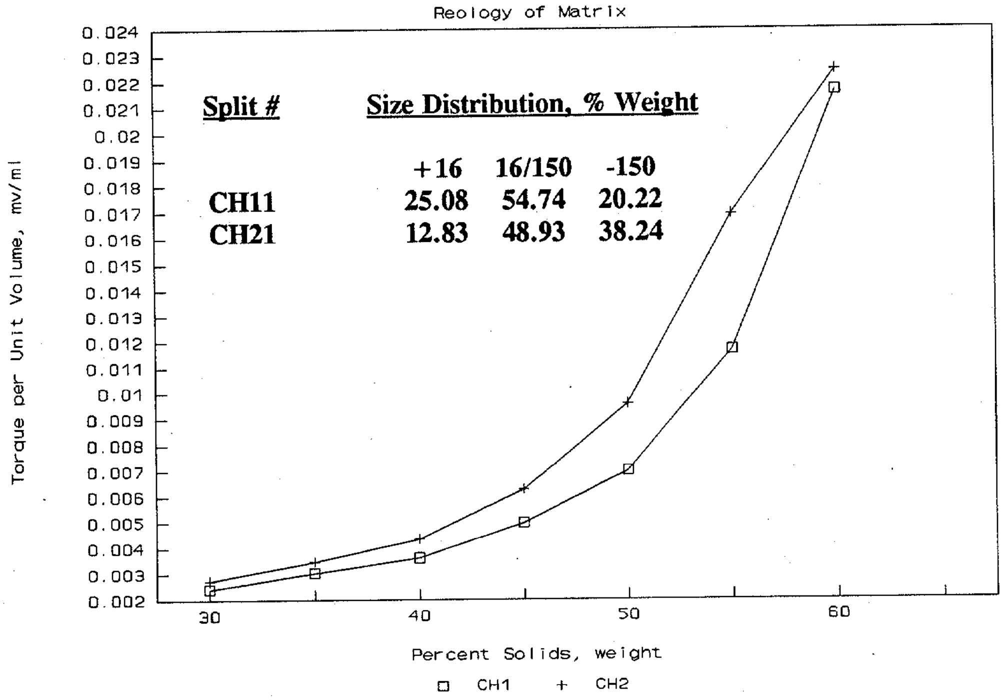
The same argument can be applied to the matrix from hole CH as shown in Figure 2. On the other hand, more energy is needed to pump matrix of split CA-1 in hole A as compared to split CA-3 even though the weight distribution indicates presence of more fines in the lower matrix (CA-3) (see Figure 3). Examining the mineralogical data of the -150 mesh fraction suggests the presence of a larger quantity of fines (-2 μ) and palygorskite in the upper matrix. In addition, the feed size distribution (16/150 mesh) tends to be finer than in the lower matrix. These two reasons could be responsible for the observed increase in energy.

Figure 1



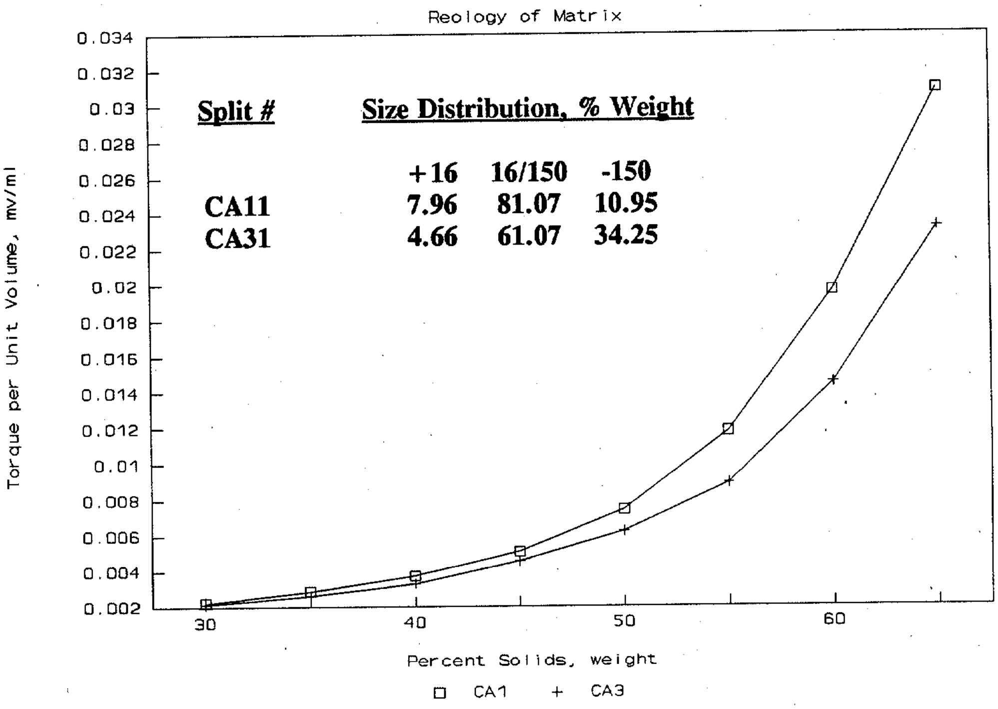
Split #	Size Distribution, % Weight		
	+16	16/150	-150
CR11	4.49	81.47	14.02
CR21	4.50	32.21	63.31

Figure 2



Split #	Size Distribution, % Weight		
	+16	16/150	-150
CH11	25.08	54.74	20.22
CH21	12.83	48.93	38.24

Figure 3



PEBBLE

The pebble products as received from Area C had been sized at 1/4 " and 14 mesh. Chemical analysis by Area C indicated that the + 1/4 " pebble contains considerable dolomite. This product is not currently a saleable product, nor shows much potential for improvement due to the high dolomite content. This oversize pebble makes up about 1% of the matrix. No work was done on this fraction.

The selected samples of the 1/4" X 14 mesh ground pebble splits from the LP Zone, and the Upper and Lower Zones, were examined by X-ray diffraction, chemical analysis, and optical microscopy. Samples of unground 1/4 " X 14 mesh pebble from these zones were examined under the microscope.

Portions of the unground selected samples were treated with Titan Yellow dye which imparts a red stain to dolomite. The stained samples were then hand sorted into francolite and nonfrancolite fractions. The francolite fractions were further examined to determine (1) the maximum grade of the “all francolite” product and (2) the relative amounts of locked dolomite and insol.

Heavy liquid separation was not attempted as this procedure did not produce usable separations on pebble samples from other companies.

Chemical analysis of all the 1/4 " X 14 mesh pebble received from Area C from all 19 core holes are in third annual report.

RESULTS - As Received Selected Pebble

The selected splits of l/4" x 14 mesh as-received pebble from each of the three zones were examined using optical microscopy, chemical analysis, and x-ray diffraction. Description of the unground pebble is shown in Table P-I, and chemical analysis of the ground products is given in Table P-II. X-ray data were used to confirm the mineralogy.

The pebble in the selected split from the LP Zone consisted of dark colored grains of rounded and polished francolite containing appreciable amounts of locked fine insol. Surface smears of ‘dolomite are common. There is some free soft, white, earthy to sugary aggregates of dolomite containing scattered locked fine sand. Minor amounts of free hard dense dolomite were also present. Chemical data indicates this pebble would average 25% P_2O_5 with 2-1/2% MgO. This zone consists of about 79% francolite, 11% dolomite and 10% insol.

The Upper Zone pebble consists largely of brown to black francolite containing some fine locked quartz sand. Traces of dolomite are present as surface smears. Trace amounts of free quartz are present. The average chemical analysis shows 28% P_2O_5 and 0.6% MgO. This zone consists of about 91% francolite, 9% insol with traces of dolomite.

The Lower Zone pebble is similar to the LP Zone pebble, but contains more free dolomite aggregates, and more surface smears and some locked dolomite on and in the francolite pebbles. The francolite also contains more locked fine insol. The average chemical analyses for this pebble indicates 16% P_2O_5 and 7% MgO. The Lower Zone consists of about 54% francolite, 31% dolomite, and 15 % insol (mainly locked with francolite and free dolomite).

Table P-I

DESCRIPTION OF AS-RECEIVED SELECTED PEBBLE

LP ZONE

CM-1	Francolite is tan to black, rounded, polished and contains fine locked sand; Dolomite consists largely of white, fine-grained, earthy to sugary aggregates, containing scattered fine locked sand. A minor amount of free brown, hard compact dolomite is present.

Upper Zone

CA-1	Francolite is brown to dark gray, rounded, polished and contains scattered fine locked sand. Some pebbles are lighter shades and some have a dull finish. About 1% free quartz grains were noted. No dolomite was observed.
CA-2	Francolite is buff to brown to black in color and subrounded to rounded and polished. The darker colored grains show considerable locked fine sand. About 10% free sand and a minor amount of dolomite aggregates are present. this pebble fraction makes up only 0.1% of the matrix.
CE-1	The francolite is largely black, rounded and polished; a minor amount is lighter in color and dull textured. A minor amount of locked sand is present. No dolomite was noted.
CE-2	No sample received. This product makes up 1.3% of the matrix.
CE-3	The francolite is black, rounded, and polished, and contains some scattered locked fine sand. A minor amount of free sand and free, white dolomite aggregates are present.
CM-2	The francolite is brown to black, rounded and polished and contains minor amounts of locked fine sand. A trace of free sand is present. No dolomite was observed.
CR-1	Francolite is predominantly brown, and is rounded and polished, and contains minor amounts of fine locked sand. Traces of free sand and dolomite were noted.
CH-1	Francolite is brown to dark gray, rounded and polished, and contains minor locked sand. No dolomite was observed.

Lower Zone

CA-3	The francolite is brown to black, rounded, polished, and contains some locked fine sand. Some surface smears and locked dolomite are present. About 40% of the sample consists of dull, white, porous free aggregates of dolomite containing scattered locked quartz and francolite grains. A minor amount of rounded, frosted, free quartz grains are present.
CA-4	Francolite is largely gray to black, with some pebbles white to brown. They are rounded and polished and contain some white, sugary to dense free dolomite. Some clay pellets were also noted. This pebble product makes up only 1.8% of this matrix split.

CE-4	The francolite is black, rounded to sub-angular, and contains scattered, fine, locked sand. Some surface smears and locked dolomite were noted. The free dolomite consists largely of dull white, sugary aggregates containing scatteredlocked sand and francolite grains. Some free dolomite occurs as brown to gray, hard, dense pebbles that closely resemble francolite. There is a trace of free sand.
CH-2	The francolite is brown to black, rounded and polished, and contains a small amount of locked fine sand. Minor dolomite surface smears are present with some locked dolomite. The free dolomite is light colored, earthy to sugary to dense, and contains minor locked fine sand and francolite. A trace of free sand is present.
CR-2	Francolite is largely brown to black, rounded and polished. A minor amount is light colored and dull textured. Minor amounts of locked fine sand and dolomite are present. The free dolomite is present as soft, white aggregates having a fine sugary texture. A trace of free quartz is present.

Table P-II

CHEMICAL ANALYSIS OF AS RECEIVED SELECTED PEBBLE

	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	F	Insol	CaO/ P_2O_5	P_2O_5 /F
LP ZONE											
CM1	25.32	39.75	2.44	0.99	0.87	0.61	0.12	2.70	10.00	1.57	9.38
Upper Zone											
CA1	29.02	42.68	0.47	1.14	0.89	0.70	0.13	3.60	8.92	1.47	8.06
CA2	23.29	35.74	0.81	0.95	2.06	0.63	0.15	3.00	20.14	1.53	7.76
CE1	29.52	45.29	0.46	0.87	2.40	0.56	0.18	3.90	6.10	1.53	7.57
CE3	30.14	44.47	0.48	0.88	0.88	0.79	0.13	3.60	6.40	1.48	8.37
CH1	28.92	45.04	0.55	1.19	0.79	0.84	0.17	3.80	5.86	1.56	7.61
CM2	27.32	42.33	0.63	0.99	0.73	0.72	0.16	3.50	9.32	1.55	7.81
CR1	29.58	44.77	0.58	1.72	0.70	0.77	0.14	3.80	5.86	1.51	7.78
Average	28.48	43.45	0.58	1.17	1.11	0.72	0.14	3.58	8.48	1.53	7.85
St. Dev.	1.74	0.25	0.25	0.50	0.09	0.02	0.24	3.68	0.03	0.28	

Table P-II (Cont.)

CHEMICAL ANALYSIS OF AS RECEIVED SELECTED PEBBLE

	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	F	Insol	CaO/ P_2O_5	P_2O_5 /F
Lower Zone											
CA3	15.24	33.24	7.78	1.41	1.03	0.46	0.12	2.00	14.76	2.18	7.62
CA4	13.71	26.50	5.02	3.87	3.23	0.47	0.29	1.80	24.40	1.93	7.62
CE4	16.21	34.34	7.23	1.97	0.60	0.77	0.13	2.20	14.38	2.12	7.37
CH2	20.09	39.02	6.02	1.34	0.87	0.69	0.14	2.70	9.60	1.94	7.44
CR2	16.70	35.93	8.24	0.76	0.92	0.49	0.13	2.20	10.60	2.15	7.59
Average	16.06	34.49	7.04	1.78	1.01	0.53	0.14	1.96	15.11	2.15	7.53
St. Dev.	4.19	3.16	3.04	0.66	0.51	0.13	0.04	0.54	3.83	3.58	0.12

Results - Staining & Hand Sorting of Selected Pebble

Samples of the selected 1/4" x 14 mesh pebble splits were stained with Titan Yellow dye which selectively imparts a red color to dolomite. The stained pebble samples were then hand-sorted into francolite and non-francolite fractions. Chemical analysis of the francolite product would show the maximum pebble grade attainable if all. the free non-francolite components could be removed.

The mineral composition of each of these hand-sorted selected splits is shown in Table P-III. These analyses show that the LP Zone pebble contains about 14% free dolomite with locked insol, and that the francolite contains some locked dolomite. The Upper Zone pebble contains only traces of dolomite and very little free gangue. The Lower Zone contains much freedolomite with locked insol (36%) and some locked dolomite with the francolite. Much of the free dolomite (with locked insol) in the LP and Lower Zones would need to be removed to produce an acceptable product. A description of the francolite and non-francolite fractions for each split in the three zones is shown in Table P-IV.

Chemical analysis of the hand-sorted fraction of the pebble is shown in Table P-V. A comparison of the average percent P_2O_5, MgO and insol content, and the estimated mineral composition for the three zones is shown below (the dolomite and insol are locked with the francolite).

Comparison of Chemical Analysis

	LP	UPPER	LOWER
P_2O_5	29.56	29.86	26.71
MgO	0.81	0.47	1.36
INSOL	6.84	6.96	8.64

These results indicate that if all of the free gangue minerals could be selectively removed, the resulting phosphate pebble product would contain about 30% P_2O_5 in the LP Zone and Upper Zone, and about 27% P_2O_5 in the Lower Zone. The MgO content of the LP Zone would be about 0.8%, and only 0.5% in the Upper, while the Lower Zone would still contain about 1.4%.

Table P-III

STAINED HAND-SORTED SELECTED PEBBLE MINERAL COMPOSITION (WT %)

	FRANCOLITE FRACTION LOCKED			NON-FRANCOLITE FRACTION FREE	
LP ZONE	FRANCOLITE	DOLOMITE	INSOL**	DOLOMITE*	INSOL**
CM-1	77	2	7	14	Tr
Upper Zone					
CA-1	92	Tr	7	Tr	1
CE-1	96		4	Tr	Tr
CE-3	89	Tr	9	1	1
CH-1	95	Tr	5	Tr	
CM-2	91		9		
CR-1	93	_	7	Tr	Tr
AVG:	93	Tr	7	Tr	Tr
Lower Zone					
CA-3	48	4	4	42	2
CE-4	52	3	7	37	1
CH-2	62	5	4	29	
CR-2	54	2	6	38	_
AVG:	54	4	5	36	1

* Includes locked insol

** Mostly quartz, minor feldspar

NOTE: {CA-2, CE-2, CA-4} = NO SAMPLE

Table P-IV

DESCRIPTION OF STAINED HAND-SORTED SELECTED PEBBLE FRACTIONS

LP ZONE

CM-1	85.4%	Francolite, tan to brown to dark gray, rounded, with scattered locked fine sand: minor dolomite surface smears
	14.3	Dolomite, soft, white, finely crystalline aggregates and as brown, somewhat harder, dense pebbles; both types containing scattered, fine locked sand.
	0.3	Sand, mainly rounded, frosted quartz, plus minor feldspar.
	100.0%	

Upper Zone

CA-1	99.1%	Francolite, largely brown and dark gray, minor light colored, containing some fine locked sand. No dolomite noted.
	Tr	Dolomite, sandy aggregates.
	0.9	Quartz grains with minor feldspar.
	100.0%	
CA-2		No Sample
CE-1	100.0%	Francolite, largely dark gray and brown, hard, dense pebble; some dull white and relatively soft, minor amounts of locked fine sand.
	Tr	Dolomite
	Tr.	Quartz Sand
	100.0%	
CE-3	98.7%	Francolite, mostly rounded and polished, minor white to brown; scattered fine locked sand; smears of dolomite.
	0.6	Dolomite, white sugary aggregates
	0.7	Quartz grains
	100.0%	

Table P-IV cont.

CH-1	99.6%	Francolite, brown to gray, rounded and polished, with minor locked sand and traces of dolomite smears.
	0.1	Dolomite aggregates
	0.3	Quartz grains
	100.0%	
CM-2	99.6%	Francolite, buff to brown and black, mostly rounded and polished, some fine sand.
	0.4	Primarily quartz with a trace of dolomite.
	100.0%	
CR-1	99.1%	Francolite, mostly brown, rounded and polished, minor locked fine sand.
	0.2	Dolomite
	0.7	Quartz grains
	100.0%	
Lower Zone		
CA-3	55.7%	Francolite, brown and dark gray, some fine locked sand, some locked dolomite/francolite grains, some dolomite surface smears on the francolite.
	42.0	Dolomite, white, earthy to fine sugary aggregates containing scattered locked fine sand and minor francolite grains.
	2.3	Primarily quartz grains with some feldspar grains; a few sandstone aggregates composed of quartz and francolite grains cemented with dolomite.
	100.0%	
CA-4		No Sample

Table P-IV cont.

CE-4	61.7%	Francolite, dark gray to black, minor brown, pebble containing scattered fine locked sand, minor dolomite surface staining, and locked dolomite.
	15.0	Dolomite, brown to gray, hard and dense, containing scattered locked fine sand and black francolite. The dolomite superficially resembles francolite.
	22.3	Dolomite, brown to gray, hard, dense, scattered locked fine sand and black francolite. This dolomite is hard and resembles francolite
	1.0	Quartz grains
	100.0%	
CH-2	70.4%	Francolite, brown and black, rounded polished, minor locked fine sand, and minor dolomitic surface smears, minor locked dolomite.
	8.6	Dolomite, soft, white earthy to sugary aggregates, scattered fine locked sand.
	20.8	Dolomite, tan to brown and gray, hard and dense, resembling francolite pebble, scattered locked fine quartz and francolite.
	0.2	Quartz grains
	100.0%	
CR-2	61.6%	Francolite, white to brown to black, lighter colors dull, darker colors polished, some fine locked sand and trace of dolomitic surface smears and locked material.
	37.6	Dolomite, soft, white, sugary aggregates containing minor locked fine sand.
	0.8	Quartz grains
	100.0%	

Table P-V

Handpicked Pebble Chemistry
Reported Values as Percents

	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	F	CO_2	Org C	L.O.I.	A.R. Insol	CaO/ P_2O_5	P_2O_5 /F
LP ZONE														
CM1	29.56	45.81	0.81	1.19	0.94	0.66	0.16	3.60	5.12	0.62	9.28	6.84	1.55	8.21
Upper Zone														
CA1	31.28	47.40	0.42	1.64	0.95	0.77	0.13	3.40	4.56	0.59	7.52	7.64	1.52	9.20
CE1	31.78	46.76	0.41	0.87	0.93	0.82	0.14	3.80	5.00	0.95	7.24	3.72	1.47	8.36
CE3	27.42	42.38	0.50	1.43	0.70	0.78	0.16	3.40	4.08	1.18	7.54	10.34	1.55	8.06
CH1	30.12	46.82	0.48	1.17	0.76	0.77	0.17	3.70	4.44	1.06	8.69	4.78	1.55	8.14
CM2	28.58	44.55	0.50	0.89	0.72	0.74	0.14	3.90	6.48	0.53	6.95	9.04	1.56	7.33
CR1	30.00	46.43	0.53	1.66	0.78	1.01	0.13	3.39	4.89	0.73	7.72	6.22	1.55	8.85
Average	29.86	45.72	0.47	1.28	0.80	0.82	0.15	3.60	4.91	0.84	7.61	6.96	1.53	8.32
Std.Dev.	1.64	1.90	0.05	0.36	0.11	0.10	0.01	0.23	0.84	0.26	0.59	2.53	0.03	0.65
Lower Zone														
CA3	26.82	42.51	1.36	0.87	0.94	1.04	0.14	3.10	6.56	1.07	9.54	7.78	1.58	8.65
CE4	25.13	39.71	1.21	2.80	0.89	0.82	0.17	3.10	6.12	0.96	8.92	11.96	1.58	8.11
CH2	27.26	44.30	1.91	1.46	0.68	0.75	0.14	3.40	7.60	1.02	10.50	5.32	1.63	8.02
CR2	27.64	43.38	0.98	0.96	0.93	0.78	0.17	3.30	6.24	0.89	8.65	9.50	1.57	8.38
Average	26.71	42.47	1.36	1.52	0.86	0.85	0.16	3.23	6.63	0.98	9.40	8.64	1.59	8.29
Std. Dev.	1.11	1.98	0.39	0.89	0.12	0.13	0.01	0.15	0.67	0.08	0.82	2.80	0.02	0.29

SUMMARY - Selected Splits

The average chemical and mineral composition of the as-received selected pebble splits for the three zones are shown in Table P-VI. These results indicate that the LP Zone and Lower Zone contain considerable dolomite and less francolite than the Upper Zone. The Upper Zone as mined should yield a 28 % P_2O_5 (61% BPL) pebble product with only traces of dolomite as surface smears. In the LP and Lower Zones, essentially all of the free dolomite would need to be removed to yield an acceptable product.

The chemical analysis and mineral composition of the francolite fraction, obtained by staining and hand sorting francolite from the non-francolite fractions, are shown in Table P-VII. These products would analyze 27-30% (59-66% BPL) P_2O_5 with MgO contents of 1.4% or less.

Listed below is a comparison of the mineralogy and chemical analyses for the as received and stained hand sorted francolite portion of the pebble fractions. The stained data indicate the analyses if all the free dolomite and free insol could be removed. This is the “Maximum Grade Attainable".

	LP ZONE		Upper Zone		Lower Zone	
	As Rcvd	Stained	As Rcvd	Stained	As Rcvd	Stained
P_2O_5	25%	30%	28%	30%	16%	27%
Insol	10	7	9	7	15	9
MgO	2.4	0.8	0.6	0.5	6.9	1.4
Francolite	77	91	91	93	54	86
Insol	10	7	9	7	15	9
Dolomite	13	2	Tr	Tr	31	5

The LP Zone would be upgraded from 25% to 30% P_2O_5 pebble with about 2% dolomite and 7% insol; the Upper Zone from 28% to 30% P_2O_5; and the Lower Zone would be upgraded from 16% to 27% P_2O_5 and MgO decreased from 6.9% to about 1.4%.

Table P-VI

As Received Selected Pebble Average Chemical Analyses

	LP Zone	Upper Zone	Lower Zone
P_2O_5	16.72 %	28.48 %	16.06 %
CaO	36.44	43.45	34.49
MgO	8.46	0.58	7.04
Fe_2O_3	1.35	1.72	1.78
Al_2O_3	0.71	1.11	1.01
Na_2O	0.46	0.72	0.53
K_2O	0.10	0.14	0.14
F	2.13	3.58	1.96
CO_2	12.85	4.93	14.78
Org. Carbon	1.76	0.71	0.95
LOI 900°C	18.99	7.38	18.05
AR Insol	9.01	8.48	15.11
CaO/P_2O_5	2.79	1.53	2.77

Average Mineral Composition

	LOCKED			FREE	
Zone	**Francolite**	**Dolomite**	**Insol**	**Dolomite***	**Insol**
LP	77 %	2 %	7 %	14 %	Trace
Upper	91	Trace	8½	Trace	½
Lower	54	4	5	36	1

* Includes locked insol

Table P-VII

Stained Hand Sorted Selected Pebble
Average Chemical Analyses

	LP Zone	Upper Zone	Lower Zone
P_2O_5	29.56 %	29.86 %	26.71 %
CaO	45.81	45.72	42.47
MgO	0.81	0.47	1.36
Fe_2O_3	1.19	1.28	1.52
Al_2O_3	0.94	0.80	0.86
Na_2O	0.66	0.82	0.85
K_2O	0.16	0.15	0.16
F	3.60	3.60	3.23
CO_2	5.12	4.91	6.63
Org. Carbon	0.62	0.84	0.98
LOI 900°C	9.28	7.61	9.40
AR Insol	6.84	6.96	8.64
CaO/P_2O_5	1.55	1.53	1.59

Average Mineral Composition

		LOCKED	
Zone	**Francolite**	**Dolomite**	**Insol**
LP	91 %	2 %	7 %
Upper	93	Trace	7
Lower	86	5	9

RESULTS - Pebble From All Splits

Chemical and x-ray diffraction analyses were made on all the -1/4 " x 14 mesh pebble samples from the 19 core holes. Chemical analyses along with differentiated LP, Upper and Lower Zones, are in third annual report. The average chemical analyses for all samples and the three zones are listed below:

	ALL SPLITS	LP ZONE	UPPER ZONE	LOWER ZONE
P_2O_5	18.4%	16.7%	28.5%	16.1%
CaO	36.4	36.4	43.5	34.5
MgO	5.7	8.5	0.6	7.0
Fe_2O_3	1.6	1.4	1.2	1.8
Al_2O_3	1.0	0.7	1.1	1.0
Na_2O	0.6	0.5	0.7	0.5
K_2O	0.1	0.1	0.1	0.1
F	2.3	2.1	3.6	2.0
CO_2	12.9	12.9	4.9	14.8
Org. C	0.9	1.8	0.7	0.1
LOI	16.1	19.0	7.4	18.1
INSOL	13.6	9.0	8.5	15.1
CaO/P_2O_5	2.7	2.8	1.5	2.1
# of splits	65	3	15	47

These averages analyses show the Upper Zone contains more P_2O_5, CaO, and F, and less MgO, LOI and CO_2. This composition is consistent with an Upper zone composition of mainly francolite with little dolomite.

The LP and Lower Zones contain less francolite and considerable dolomite. The X-ray data confirm the same results.

These analyses which include all the received splits from the 19 holes, agree with the data from the selected splits.

FEED

FEED (28/150 mesh) - Selected Splits

Fine feeds from the selected splits were analyzed for P_2O_5, CaO, MgO, Fe_2O_3, Al_2O_3, and insol. The results are shown in Table F-I. A description of these feed samples is given in Table

F-II. The mineralogy of the feed is relatively simple. Francolite grains in the LP Zone are brown to black, rounded to subrounded and polished. The dolomite aggregates are light-colored, and sugary textured. Quartz sand is the major component. Minor amounts of feldspar are present.

The Upper Zone samples contain francolite which is brown to black with some lighter grains. The darker colored pellets are rounded and polished, while the lighter ones are usually earthy and dull. Minor to trace amounts of gypsum and dolomite are locally present. The insol is predominantly quartz with minor amounts of feldspar.

The francolite in the Lower Zone is similar to the francolite in the LP zone. Feed from splits CE-4 and CH-2 contain some francolite that. has a distinctive reddish-brown color. Iron was suspected as the cause, but a spot test was negative. The dolomite in the Lower Zone is light colored and has a sugary texture. Minor amounts of gypsum occur as aggregates of small, clear intergrown crystals. The insol is essentially all quartz with minor amounts of feldspar.

Chemical, X-ray and petrographic analyses were made on fine feed samples from selected core splits. These show that the principal mineral in all three zones is quartz. The francolite in the LP and Lower Zones is almost entirely dark in color, while the francolite in the Upper Zone is also dark but also includes varying amounts of lighter colored pellets. Dolomite is present in minor amounts in the LP and Lower Zones. A minor amount of gypsum is present in the Lower Zone. Traces of gypsum were noted in the Upper Zone in sample CE-3.

The average chemical and estimated mineralogical analyses for feeds from the selected splits in the three zones are shown in Table F-III. The fine feeds (28 x 150 mesh) were used for testing as they made up over 95% of the total feed fraction. In many splits there was insufficient coarse feed (14 x 28 mesh) for analyses.

Table F-I

FEED CHEMISTRY - SELECTED SPLITS
REPORTED VALUES AS PERCENTAGES

	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	A.R. Insol	CaO/ P_2O_5
LP ZONE							
CM-1	5.85	10.33	1.09	0.33	0.25	75.76	1.77
Upper Zone							
CA-1	7.90	12.30	0.16	0.37	0.26	73.62	1.56
CA-2	4.13	6.20	0.19	0.19	0.15	85.92	1.50
CE-1	7.70	11.94	0.13	0.30	0.28	74.06	1.55
CE-2	5.03	7.67	0.09	0.20	0.25	80.80	1.52
CE-3	5.16	8.00	0.12	0.47	0.19	81.14	1.55
CH-1	4.93	7.39	0.12	0.30	0.30	82.98	1.50
CM-2	9.68	14.90	0.23	0.43	0.30	67.86	1.54
CR-1	7.04	10.58	0.16	0.51	0.19	74.46	1.50
Average	6.44	9.87	0.14	0.35	0.24	76.50	1.53
Lower Zone							
CA-3	6.31	10.47	0.66	0.56	0.23	74.94	1.66
CA-4	5.44	8.52	0.33	0.79	0.30	79.96	1.57
CE-4	6.35	10.02	0.48	0.71	0.23	75.74	1.58
CH-2	4.33	7.28	0.49	0.53	0.23	81.78	1.68
CR-2	6.75	10.97	0.65	0.33	0.23	74.94	1.63
Average	5.84	9.45	0.52	0.58	0.24	77.47	1.62

Table F-II

Description and Estimated Mineral Composition Feeds - Selected Splits

LP ZONE		
CM-1	20%	Francolite pellets, brown to black, rounded to subrounded and polished
	5%	Dolomite, porous, sugary textured aggregates
	75%	Quartz grains, trace feldspar
	100%	

Table F-II (continued)

Upper Zone		
CA-1	25%	Francolite, tan to brown and gray to black, rounded to subrounded and polished
	75%	Quartz with minor feldspar
	100%	
CA-2	15%	Francolite, tan to brown and black, rounded to subrounded and polished
	85%	Quartz with minor feldspar
	100%	
CE-1	25%	Francolite, 10% buff, dull and porous, 15% brown to black, rounded to subrounded and polished
	75%	Quartz grains
CE-2	15%	Francolite, brown and black, rounded to subrounded and polished
	85%	Quartz grains
	100%	
CE-3	15%	Francolite, brown to black, rounded polished
	84%	Quartz grains
	1%	Gypsum aggregates
	100%	
CH-1	15%	Francolite, 5% dull, buff grains, 10% brown to black, rounded to subrounded and polished
	85%	Quartz grains
	100%	
CM-2	30%	Francolite, brown to black, rounded and polished
	70%	Quartz grains
	TR	Clay, as dull, pale gray aggregates
	100%	
CR-1	25%	Francolite, brown with some black, rounded, polished
	75%	Quartz grains
	100%	
Lower Zone		
CA-3	20%	Francolite, brown to black, rounded to subrounded and polished
	8%	Dolomite, white sugary aggregates
	2%	Gypsum, light colored aggregates
	70%	Quartz grains
	100%	

Table F-II (continued)

CA-4	15%	Francolite, mostly black, rounded to subrounded and polished
	84%	Quartz grains
	1%	Clay, as dull, pale greenish aggregates
	Tr	Gypsum
	100%	
CE-4	20%	Francolite, brown and gray to black, rounded to subrounded and polished; some pellets have a distinctive reddish-brown color
	4%	Dolomite, white, sugary aggregates
	1%	Gypsum
	75%	Quartz grains
	100%	
CH-2	12%	Francolite, brown and black, rounded to subrounded and polished; minor amount of distinctive reddish-brown colored pellets
	4%	Dolomite, white, sugary aggregates
	1%	Gypsum
	83%	Quartz grains
	100%	
CR-2	25%	Francolite, mostly brown some black, rounded and polished
	5%	Dolomite, light colored, sugary aggregates
	70%	Quartz grains
	100%	

Table F-III

Average Chemical Analyses Feeds - Selected Splits - Weight Percent

	LP	Upper	Lower
P_2O_5	7.31	6.79	6.65
CaO	11.85	10.22	11.41
MgO	0.71	0.14	0.79
Fe_2O_3	0.79	0.69	1.08
Al_2O_3	0.25	0.29	0.24
AR Insol	72.70	76.50	72.78
CaO/P_2O_5	1.64	1.51	1.73

Estimated Mineral Composition

Francolite	19	22	19
Dolomite	5	Trace	3
Gypsum	--	Trace	1
Quartz*	76	78	77
	100	100	100

* Includes minor amounts of feldspar.

Chemical and size analyses of the fine feeds for the selected splits are in third annual report. The average analyses for the three zones are listed below.

LP ZONE

Size Fraction	WT %	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	INSOL
Sample No. CM1							
+ 28	4.5	16.67	29.26	3.22	0.86	0.68	38.08
28/35	7.9	11.23	20.25	2.39	0.60	0.51	53.12
35/48	18.3	7.21	12.27	1.14	0.36	0.28	71.20
48/65	30.4	5.23	8.58	0.61	0.26	0.19	78.48
65/100	30.4	4.13	7.01	0.61	0.20	0.17	83.14
-100	8.5	3.25	7.21	1.86	0.19	0.21	81.08

AVERAGE UPPER ZONE

Size Fraction	WT %	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	INSOL
+ 28	5.9	18.1	27.6	0.39	0.85	0.48	39.34
28/35	13.4	12.9	19.9	0.27	0.60	0.37	56.95
35/48	26.8	7.3	11.2	0.14	0.36	0.21	75.37
48/65	25.8	5.9	9.0	0.12	0.29	0.19	79.83
65/100	19.7	4.2	6.4	0.09	0.21	0.14	85.55
-100	8.6	3.0	4.6	0.08	0.16	0.16	89.18

AVERAGE LOWER ZONE

Size Fraction	WT %	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	INSOL
+ 28	3.0	16.42	27.54	2.07	0.91	0.43	36.07
28/35	7.1	10.54	17.41	1.09	1.10	0.39	58.65
35/48	16.5	7.44	11.99	0.58	0.79	0.29	71.64
48/65	25.8	5.64	9.00	0.37	0.52	0.19	79.17
65/100	31.1	4.26	6.80	0.26	0.36	0.18	84.02
- 100	16.5	2.70	4.37	0.44	0.33	0.21	88.09

These analyses show higher P_2O_5 content in the coarser mesh fractions (+35 mesh). The Upper Zone contains less MgO. There is little difference in the average size analysis for the three zones.

Feed - All Splits

Chemical, and x-ray diffraction analyses were run on all the received ground fine feeds (28 x 35 mesh) from all the splits from the 19 core holes.

Chemical analyses were run on all the fine feed samples and the results are in the third annual report. The average analyses for all samples, LP, Upper and Lower Zones are listed below.

	All Samples	Lp Zone	Upper Zone	Lower Zone
P_2O_5	6.7%	7.3	6.8	6.7
CaO	11.2	11.9	10.2	11.4
MgO	0.7	0.7	0.1	0.8
Fe_2O_3	1.0	0.8	0.7	1.1
Al_2O_3	0.3	0.3	0.3	0.2
CO_2	2.2	2.2	1.3	2.5
Insol	73.5	72.7	76.5	72.8
CaO/P_2O_5	1.7	1.6	1.5	1.7

The average P_2O_5 content of all the fine feed samples is similar. The major difference is that the feed in the Upper Zone contains little or no dolomite/calcite, while the LP and Lower Zones contain considerable amounts. X-ray diffraction data agrees with the above chemical analyses. The chemical analyses for all feeds and the selected feeds more or less agree.

The size analyses of all the fine feed samples and for each zone are in the third annual report. The averages for all samples, and the LP, Upper and Lower Zones are listed below:

Mesh	All	LP	Upper	Lower
+ 28	5.1	5.3	5.1	5.0
28 x 35	10.6	10.9	10.0	10.7
35 x 48	20.2	16.1	20.4	20.4
48 x 65	27.4	23.3	27.7	27.6
63 x 100	25.0	31.9	26.4	24.3
- 100	11.7	12.5	10.5	12.0
	100.0			
No. of Splits	78	3	15	60

There is essentially no difference in the size analyses for the three zones.

CONCENTRATE

The flotation concentrates received from Area C were already pulverized and were apparently portions of their analyzed samples. The results of analyses on all the flotation concentrates received from Area C are in the third annual report. Those samples representing the selected splits from the LP, Upper, and Lower Zones were analyzed chemically and were also examined by x-ray diffraction and optical microscopy.

Laboratory flotation tests were run at FIPR on the corresponding feed samples to obtain unground concentrates for microscopic examination. Chemical analysis of these concentrates did not yield the grade indicated by the as received pulverized concentrates. Microscopic examination confirmed high insol. It is believed that the poorer flotation results were due to the detrimental effects of aging on stored feed samples. The FIPR concentrates, however, were useful for x-ray diffraction and microscopic studies.

RESULTS - Selected Splits

A description of the phosphate concentrate from the three zones is given in Table C-I.

The francolite in the LP zone is generally dark in color, and the grains are rounded and polished. Minor amounts of free and locked insol are present. There are also minor amounts of light-colored, sugary dolomite aggregates and traces of locked dolomite.

The francolite in the Upper Zone is largely dark colored, but some light colored grains are also present. The grains are rounded and polished, and contain minor amounts of locked insol. Locally gypsum is, present.

The francolite from the Lower Zone is similar to LP Zone francolite. In concentrates from splits CE-4 and CH-2 distinctive reddish-brown phosphate grains were noted. Trace amounts of aggregated fine gypsum crystals were noted in samples CA-4 and CH-2. Traces of very fine pyrite are present in some Lower Zone samples.

The analyses of the concentrates from the three zones is shown in Table C-II. The Upper Zone is higher in P_2O_5 and lower in MgO and CO_2. The Lower Zone is higher in Fe_2O_3, sulfate and sulfide sulfur.

Table C-I

DESCRIPTION OF SELECTED FLOTATION CONCENTRATES

LP Zone

CM-1	Francolite is largely black and brown with some gray and is rounded and polished; minor white sugary dolomite aggregates and minor free quartz grains are also present.

Upper Zone

CA-1	Francolite pellets are white to brown to gray and are rounded and polished. Minor free sand grains.
CA-2	Francolite is gray and tan to amber, with some white. Pellets are rounded and polished. Minor free sand grains.
CE-1	Francolite is gray to black, with some tan and brown, and minor white, grains are rounded and polished. Minor free sand noted.
CE-2	Francolite is gray to black with some tan and brown; grains are rounded and polished. A trace of dolomite smears was noted. Minor free sand is present.
CE-3	Francolite is gray to black, and is rounded and polished. Minor free sand noted.
CH-1	Francolite is gray to black and brown; grains are rounded and polished. Minor free sand noted.
CM-2	Francolite is brown and gray to black, and is rounded and polished. Minor free sand noted.
CR-1	Francolite is largely brown with some gray, and is rounded and polished. Minor free sand is present.

Lower Zone

CA-3	Francolite is gray to brown with some minor white, Pellets are rounded and polished; Minor white, sugary dolomite aggregates.
CA-4	Francolite is gray to black with some tan to brown; minor free sand, dolomite and gypsum aggregates present. Trace finely disseminated pyrite?

Table C-1 (continued)

CE-4	Francolite is gray to black, some reddish-brown grains, minor free sand and sugary dolomite aggregates.
CH-2	Francolite is gray to black and brown, some reddish-brown grains; minor free sand, dolomite, and gypsum with a trace of finely disseminated pyrite.
CR-2	Francolite is white to brown and gray to black; the darker colors appear more rounded and polished; some dolomite is present as smears; minor free quartz sand and dolomite aggregates.

Table C-II

Chemical Analyses Selected Concentrates

	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	F	CO_2	Org C	SO_3	S^*	L.O.I.	A.R. Insol	CaO/ P_2O_5
L.P.Zone															
CM1	29.26	46.22	1.92	1.37	0.98	0.61	0.14	3.50	6.92	0.78	0.42	0.21	7.99	2.66	1.58
Upper Zone															
CA1	31.46	46.26	0.46	1.26	0.91	0.70	0.16	3.80	4.36	0.70	0.62	0.47	5.87	3.14	1.47
CA2	31.83	45.22	0.60	1.23	0.72	0.74	0.13	3.80	3.98	0.87	1.00	0.20	8.01	3.28	1.42
CE1	32.03	47.18	0.43	1.10	1.21	0.73	0.16	4.00	4.36	1.55	0.65	0.50	5.79	1.72	1.47
CE3	29.70	42.55	0.68	2.72	0.85	0.66	0.17	3.70	4.16	0.84	1.07	0.51	7.98	3.94	1.43
CH1	31.50	46.37	0.46	1.69	0.83	0.70	0.16	3.70	4.04	0.76	0.65	0.68	5.52	2.56	1.47
CM2	30.54	45.52	0.51	1.22	0.72	0.65	0.14	3.80	5.24	0.61	0.55	0.35	5.63	4.48	1.49
CR1	31.54	47.39	0.48	2.04	0.68	0.78	0.16	4.00	4.96	0.65	0.60	0.42	6.74	1.68	1.50
Avg.	31.23	45.78	0.52	1.61	0.84	0.71	0.15	3.83	4.44	0.85	0.73	0.45	6.51	2.97	1.47
Lower Zone															
CA3	27.73	41.36	1.63	2.14	0.83	0.71	0.17	3.40	5.58	1.00	1.00	0.61	9.48	6.02	1.49
CA4	27.61	41.72	0.96	3.27	0.74	0.80	0.17	3.50	3.74	1.11	1.20	1.18	8.31	3.68	1.51
CE4	28.41	42.67	1.08	1.63	0.81	0.67	0.16	3.30	5.45	0.59	1.07	0.78	8.52	2.44	1.50
CH2	28.17	45.18	1.34	2.49	0.89	0.70	0.17	3.40	5.76	1.06	0.82	0.83	6.33	4.20	1.60
CR2	29.98	45.57	1.06	1.24	0.68	0.70	0.16	3.80	5.60	0.77	0.52	0.24	7.41	4.26	1.52
Avg.	28.36	43.30	1.21	2.16	0.79	0.72	0.16	3.48	5.23	0.91	0.92	0.73	8.01	4.12	1.53

* Sulfide Sulfur

SUMMARY - Selected Splits

Concentrates from the selected splits of the LP, Upper, and Lower Zones were examined using optical microscopy, X-ray diffraction, and chemical analyses. The average chemical and mineral composition for each of these zones is shown in Table C-III.

The francolite pellets in all three zones are generally dark in color and well rounded and polished. There are more lighter colored grains in the Upper Zone concentrates. Dolomite is present mainly in the LP and Lower Zones, and is light colored and has a sugary texture. In the Lower Zone concentrate pyrite is probably present in trace amounts as very finely disseminated particles in the black francolite pellets. Gypsum is present in some of the Lower Zone concentrates as aggregates of small, clear intergrown crystals.

Chemically, the Upper Zone concentrates have a higher P_2O_5 content and a lower MgO and CO_2 content than concentrates from the other two zones. The greater Fe_2O_3, sulfate, and sulfide content in the Lower Zone concentrates are due to the presence of gypsum and pyrite.

Table C-III

Average Chemical Composition - Selected Concentrates
Weight Percent

	LP	Upper	Lower
P_2O_5	29.54	31.50	27.80
CaO	44.97	45.16	42.43
MgO	1.53	0.54	1.64
Fe_2O_3	1.57	1.60	2.11
Al_2O_3	0.80	0.94	0.76
Na_2O	0.61	0.64	0.64
F	3.47	3.79	3.24
CO_2	6.58	4.29	5.15
Org. C	0.77	0.82	0.74
SO_3	0.85	1.50	1.84
S *	0.04	0.25	0.36
LOI	9.18	6.81	8.73
AR Insol	2.51	3.16	5.15
CaO/P_2O_5	1.52	1.43	1.54

Table C-III (continued)

Mineral Composition - Weight Percent

Francolite	91	97	93
Dolomite	6	Trace	3
Insol	3	3	4
Gypsum	-	-	Trace
Pyrite	-	-	Trace
	100	100	100

* Sulfide Sulfur

RESULTS - All Splits

Chemical and x-ray diffraction analyses were run on all the ground concentrates from all the splits received from the 19 core holes received.

Chemical analyses for all the concentrates are in the third annual report. The average analyses are listed below:

	All Splits	LP	Upper	Lower
P_2O_5	28.6	29.5	31.5	27.8
CaO	43.1	45.0	45.2	42.4
MgO	1.4	1.5	0.5	1.6
Fe_2O_3	2.0	1.6	1.6	2.1
Al_2O_3	0.8	0.8	0.9	0.8
Na_20	0.6	0.6	0.6	0.6
F	3.3	3.5	3.8	3.2
CO_2	6.0	6.6	4.3	6.5
Acid Insol	4.6	2.5	3.2	5.2
L.O.I.	8.9	9.2	6.8	8.7
CaO/P_2O_5	1.5	1.5	1.4	1.5
# of Samples	75	3	15	57

The above data indicate that concentrates of lower quality (higher MgO content) are obtained from the LP and Lower Zone matrix as compared to the Upper Zone matrix.

X-ray data confirmed the above results. The average chemical analyses of all the samples and the selected samples are similar.

Empirical Formula Calculation

Chemical analysis of the concentrate product was used to estimate the empirical formula of the phosphate mineral (francolite) in the feed. Weight percent of each element's oxide (corrected to be free of insoluble material) was converted to weight percent of the element. Elemental weight percents were divided by the atomic weight of the element to convert the values to moles. Multiplying the moles of each element by ten gives the number of atoms of each element. Ionic charge times the number of atoms or ions is the charge contribution from the ion of the element.

Oxide Wt. % x conversion factor = Elemental Wt. %

Elemental Wt. %/Atomic Wt. = Moles of Element

Moles of Element x 10 = Number of Atoms of the Element

Number of Atoms x Charge per Atom = Charge Contribution

After the number of atoms was calculated, cations were summed and anions were summed. Charges contributed by cations and anions were summed separately. Ionic charge difference was determined by subtracting the total anion charge from the total cation charge. The result is a positive number if the anion charge is larger.

Magnesium and calcium were adjusted to reflect the contribution from dolomite in the concentrate product. Splits with calcite were not used in the calculation of the overall francolite formula.

Data used to calculate the formulas were a combination of concentrations measured chemically and the values for CO_2 calculated from x-ray diffraction data. Concentration of CO_2 measured in an apatite crystal lattice and change in relative angular location of the 004 and 410 reflections are linearly correlated. The formula used for the calculation was derived by Mathews and Nathan (1977)[1]. Delta 2-Theta for 004 reflection - 410 reflection was measured from x-ray diffraction data. Their formula calculated CO_2 values which compared very well with the values measured by ascarite absorption of the CO_2 from a mild HCl digestion of the concentrate product.

An average formula for the top split from all the cores was calculated. A separate empirical formula for the francolite in the other splits was calculated. There is no significant differences between the formula for the upper splits and the formula calculated for the remaining splits. These formulas were consistent with those generated by other authors.

---

[1] Mathews, A. and Nathan Y., 1977. The Decarbonation of Carbonate-Fluorapatite (Francolite); American Mineralogist Vol 62 pp. 565573.

Average Formula for Francolite in the Upper Zone:

FORMULA WEIGHT 1011

$Ca_{9.73}\ Mg_{0.11}\ Na_{0.26}\ (PO_4)_{5.25}\ (CO_2)_{1.05}\ (SO_4)_{0.09}\ F_{2.16}$

TOTAL CATIONS 10.18
TOTAL ANIONS 8.65

TOTAL POSITIVE CHARGE 20.0
TOTAL NEGATIVE CHARGE 20.3

CHARGE DEFICIT -0.3

Average Formula for Francolite in the Lower Zone:

FORMULA WEIGHT 1023

$Ca_{9.72}\ Mg_{0.11}\ Na_{0.28}\ (PO_4)_{5.32}\ (CO_2)_{1.07}\ (SO_4)_{0.14}\ F_{2.27}$

TOTAL CATIONS 10.03
TOTAL ANIONS 8.73

TOTAL POSITIVE CHARGE 20.0
TOTAL NEGATIVE CHARGE 20.4

CHARGE DEFICIT -0.4

HEAVY MINERALS

CONCLUSIONS

Heavy mineral separations from the Upper and Lower Zones were examined using both petrographic and binocular microscopes. The low total weight percent of heavies present and low concentration of the heavies of potential value, indicate recovery of heavy minerals as a by-product would not be economically feasible.

PROCEDURES AND RESULTS

Composite feeds were made of both the Upper and Lower matrix splits to determine the amount, composition, and economic potential of the heavy minerals in the feeds. Microscopic examination of these feeds indicated the valuable heavy mineral content to be low, so separations were made only on feeds, not feeds and flotation products. A heavy mineral separation was not made on the LP zone as it made up such a small proportion of the three zones. The composite feeds from both the Upper and Lower Zones were dissolved in HCl to remove the dolomite, francolite, etc. The fine suspended organic matter was decanted. The heavy minerals were separated from the quartz-feldspar sand using tetrabromoethane (Sp. Gr. 2.95). Microscopic studies were made on the heavy minerals separated from the Upper and Lower Zones.

Table HM-1 lists the heavy mineral composition and distribution.

Table HM-1

Estimated Heavy Mineral Composition and Distribution

	Upper Zone	Lower Zone
Rutile & Ilmenite	20%	20%
Zircon	10	5
Epidote	15	10
Garnet	10	10
Pyrite	3	15
Sillimanite	15	10
Staurolite	10	10
Tourmaline	10	5
Other	7	15
	100	100
Wt % HCl insol	85	74
Wt % Heavy Minerals	0.125	0.107
Wt% Rutile & Ilmenite	0.025	0.021
Wt % Zircon	0.0125	0.005

To put these heavy mineral analyses into perspective, let us start with 1,000,000 tons of feed from each zone.

	Tons	
	Upper	Lower
Start	1,000,000	1,000,000
Heavy Minerals	1,250	1,070
Rutile & Ilmenite	250	210
Zircon	125	50
Total Valuable	375	260

From the 1,000,000 tons of feed from each zone, there would be about 375 tons of valuable heavy minerals in the Upper Zone, and about 260 tons in the Lower, and this is calculated at

100% recovery. The cost of recovering and separating these heavy mineral concentrates would far exceed their value.

PHOSPHATIC CLAYS

The mineralogical composition of the -150 mesh phosphatic clay fraction of the selected splits was determined from chemical, X-ray diffraction, and petrographic analysis. The -150 mesh material was sized at 44 microns by screening at 325 mesh, and at 2 microns by differential settling in cylinders. X-ray diffraction, chemical, and petrographic analyses were also made on the sized fractions, +44, 2 x 44 and nominal minus 2 micron. The +44 and 2 x 44 micron fractions consist mainly of francolite, dolomite and insol (mainly quartz with minor amounts of feldspar, etc.). The clay minerals are concentrated in the minus 2 micron fraction.

In this report, the group mineral name smectite is used, which includes the clay mineral montmorillonite. For practical purposes montmorillonite and smectite are essentially the same for Central Florida phosphatic clays. Palygorskite is used instead of attapulgite; mineralogically they are similar.

CHEMISTRY - Selected Splits

The sized phosphatic clay samples of the special splits were examined using optical microscopy, x-ray diffraction and chemical analyses. The +44 and 2 x 44 micron fractions consisted mainly of francolite, dolomite, and quartz; therefore, chemical analyses were performed on the aqua regia soluble fraction. The chemical analyses made on the -2 micron fractions were made using a metaborate fusion method so that the clays minerals in this fraction would go into solution.

+ 44 MICRON

The chemical analyses for the +44 micron fraction of phosphatic clays are on Table PC-I. This fraction of the LP Zone fines is relatively high in MgO and insol, and has a moderate P_2O_5 content. The +44 micron fraction from the Upper Zone is very high in insol with a relatively high P_2O_5 content and low MgO. The +44 micron fraction from the Lower Zone phosphatic fines is high in MgO and insol and low in P_2O_5. The coarsest fraction of the phosphatic clays from all zones averages about 6-7 weight percent.

Table PC-I

Chemical Analysis - Phosphatic Clay +44 micron fraction

	Wt% of Phos Clay	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	L.O.I.	Insol
LP ZONE								
CM1	7.00	4.27	23.46	13.20	0.22	0.48	27.34	30.60
Upper Zone								
CA1	3.70	8.98	13.78	0.62	0.37	0.60	4.41	67.90
CE1	8.10	13.29	21.60	0.56	0.48	1.00	5.45	52.60
CH1	10.80	6.89	10.80	0.34	0.55	0.97	3.60	72.10
CM2	3.00	6.80	15.46	1.66	2.03	1.73	N.A.	53.60
CR1	2.10	4.92	6.06	0.45	0.28	0.30	2.66	83.90
Average	5.54	8.18	13.54	0.73	0.74	0.92	3.22	66.02
Lower Zone								
CA3	6.90	1.91	26.07	17.78	0.42	0.20	35.00	18.00
CA4	13.70	2.52	4.11	1.45	1.53	0.88	7.15	80.20
CE4	2.80	2.05	11.95	6.91	0.37	0.26	16.73	62.70
CH2	7.70	2.93	15.25	8.68	0.46	0.36	17.45	51.80
CR2	4.80	1.44	18.78	14.34	0.35	0.51	28.51	35.80
Average	7.18	2.17	15.23	9.83	0.63	0.44	20.97	49.70

2 X 44 MICRON FRACTION

The chemical analyses (aqua regia) for the 2 x 44 micron size fraction for the phosphatic clays are on Table PC-II. The average chemical composition of this fraction of the LP and Lower Zones are similar, MgO very high, moderate insol and minor P_2O_5. The average Upper Zone is relatively high **P_2O_5** and low in MgO. Although minor in percentage, the amount of **Al_2O_3** in the Upper Zone is considerably higher than in the other two zones due to higher clay mineral content. The average weight percentages of this size in the total phosphatic clay fractions varies from about 70% in the LP and Lower Zones to 30 % for the Upper Zone.

Table PC-II

Phosphatic Clay Chemical Analysis
2 X 44 micron Fraction

	Wt% of Phos Clay	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	L.O.I.	Insol
LP ZONE								
CM1	71.90	1.68	29.03	19.92	0.26	0.83	41.47	7.60
Upper Zone								
CA1	33.20	9.50	16.75	2.77	1.41	5.50	13.84	45.20
CA2	27.30	8.40	13.15	0.83	1.57	3.05	7.93	61.80
CE1	23.30	23.79	36.33	0.85	0.91	1.91	8.80	16.90
CH1	23.00	15.36	22.89	1.04	1.52	2.77	7.65	42.30
CM2	46.50	6.02	14.33	3.89	2.45	2.69	19.27	48.00
CR1	35.70	4.86	11.80	5.27	1.09	1.63	14.52	57.80
Average	31.50	11.32	19.21	2.44	1.49	2.93	12.00	45.33
Lower Zone								
CA3	79.30	0.31	30.72	22.06	0.80	0.12	44.39	2.50
CA4	44.30	1.23	17.39	12.97	3.16	1.20	28.22	33.60
CE4	74.70	0.19	29.55	22.02	0.48	0.13	42.61	5.10
CH2	71.90	0.94	24.88	18.24	1.22	0.83	40.21	16.80
CR2	66.00	0.25	30.52	23.16	0.10	0.07	43.28	3.80
Average	67.24	0.58	26.61	19.69	1.15	0.47	39.74	12.36

- 2 MICRON

The metaborate fusion chemical analyses for the minus 2 micron fraction of the selected splits of phosphatic clays are on Table PC-III. The analyses for all three zones are relatively similar being high in SiO_2, and Al_2O_3, and LOI, reflecting higher clay mineral content. The weight percentages of this size relative to the phosphatic clay fraction (-150 mesh) is 21% for the LP, 26 % in the Lower Zone and 64 % for the Upper.

Table PC-III

Phosphatic Clay Chemical Analysis
- 2 Micron Fraction

	Wt% of Phos Clay	P_2O_5	CaO	MgO	Fe_2O_3	Al_2O_3	Na_2O	K_2O	SiO_2	L.O.I.
LP ZONE										
CM1	21.10	4.20	6.38	2.30	2.78	19.73	0.33	1.04	41.84	19.82
Upper Zone										
CA1	63.10	3.66	5.31	1.29	2.97	24.32	0.61	1.85	43.00	17.68
CA2	72.70	1.85	2.26	1.37	2.18	28.19	0.81	0.50	44.12	19.31
CE1	68.60	6.02	8.48	1.72	2.76	17.11	0.85	1.46	37.61	20.35
CH1	66.20	5.20	7.73	1.78	2.82	16.02	0.53	2.04	39.33	17.13
CM2	50.50	1.86	2.52	2.62	3.65	16.93	0.76	2.09	43.13	19.02
CR1	62.20	1.13	1.80	2.44	3.40	15.40	0.33	1.34	44.18	20.60
Average	63.88	3.29	4.68	1.87	2.96	19.66	0.65	1.55	41.90	19.02
Lower Zone										
CA3	13.80	2.16	3.80	3.07	3.10	19.30	0.58	1.11	42.61	21.38
CA4	42.00	1.14	1.30	3.22	4.08	18.10	0.35	2.03	47.02	19.42
CE4	22.60	1.17	1.79	4.63	3.13	14.82	0.53	1.58	47.01	21.36
CH2	20.40	1.69	2.79	3.95	3.11	16.00	0.39	1.58	47.95	21.28
CR2	29.20	0.83	1.93	3.42	3.78	16.15	0.37	1.87	48.32	20.15
Average	25.60	1.40	2.32	3.66	3.44	16.87	0.44	1.64	46.58	20.72

MINERALOGY - Selected Splits

The mineral composition of the three size fractions +44, 2 x 44, and - 2 micron fractions for the LP, Upper and Lower Zones is in Table PC-IV.

The mineral composition of LP Zone for the +44 and 2 x 44 mesh fractions indicate the dominant constituent is dolomite with a major to minor amount of quartz and a minor amount of francolite. These two size fractions make up almost 80% of the phosphatic clay fraction. The - 2 micron fraction makes up 21% of the total phosphatic clay fraction and is composed mainly of illite/mica and kaolinite with moderate francolite and minor dolomite and quartz. The average mineral composition of the - 150 fraction of the LP Zone indicates dolomite as the major constituent with minor amounts of quartz, illite/mica, kaolinite, and francolite.

The average composition of the +44 micron fraction of the Upper Zone indicates the dominant mineral is quartz with francolite being a major constituent with a minor amount of dolomite. The 2 x 44 micron fraction is mainly francolite and quartz with minor amounts of dolomite and clay minerals. The average - 2 micron fraction consists mainly of francolite, quartz, smectite, illite/mica and kaolinite with a minor to moderate amounts of dolomite, francolite and quartz. The average composition of the phosphatic clay fraction to the Upper Zone contains moderate amounts of francolite, quartz, smectite, and kaolinite, with minor amounts dolomite and illite/mica.

The average composition of the +44 micron fraction of the Lower Zone indicates dolomite and quartz are dominant, with a minor amount of francolite. The average 2 x 44 mesh fraction has a dominant amount of dolomite, and a minor amount of quartz and francolite. The average - 2 micron fraction contains major amount of smectite, with moderate dolimite, sepiolite and illite/mica, and minor amounts of francolite, quartz, palygorskite, and kaolinite. The average mineral content for the -150 mesh phosphatic clay fraction of the Lower Zone consists of dolomite as the dominant mineral, with a moderate amount of quartz and smectite, and minor francolite, palygorskite, sepiolite, illite/mica and kaolinite.

Table PC-IV

MINERAL COMPOSITION - PHOSPHATIC CLAY FRACTIONS - SELECTED SAMPLES

LP ZONE

SAMPLE	SIZE MICRONS	WT% PHOS CLAY FRACTION	FRANCOLITE	DOLOMITE	QUARTZ	CLAY	ILLITE/ MICA	KAOLINITE
CM-1	+ 44	7.0	10	60	30			
	2 x 44	71.9	6	86	8			
	- 2	21.1	14	10	6	70	Major	Major
		100.0	8	70	7	15	Minor	Minor

Tr	< 1 %
Minor	1 - 10
Moderate	10 - 20
Major	20 - 50
Dominant	> 50

TABLE PC-IV CONT.

UPPER ZONE

	SIZE MICRON	WT% OF CLAY FRACTION	FRANCOLITE	DOLOMITE	QUARTZ	CLAY	SMECTITE	SEPIOLITE	ILLITE/ MICA	KAOLINITE
CA1	+ 44	3.7	30	2	68					
	2 x 44	33.2	32	11	45	12	Minor		Minor	Minor
	- 2	63.1	12	6	7	75	Major		Major	Major
		100.0	20	7	22	51	Moderate		Moderate	Moderate
CA2	+ 44	Tr	40	10	50					
	2 x 44	27.3	28	3	62	7				Minor
	- 2	72.7	6	6	8	80			Minor	Dominant
		100.0	12	5	23	60			Minor	Major
CE1	+ 44	8.1	44	3	53					
	2 x 44	23.3	79	3	18				Minor	
	- 2	68.6	20	8	7	65		Minor	Moderate	Minor
		100.0	36	6	13	45		Minor	Moderate	Minor
CH1	+ 44	10.8	23	5	72				Minor	Minor
	2 x 44	23.0	52	5	43				Minor	Minor
	- 2	66.2	17	8	5	70	Major		Moderate	Moderate
		100.0	26	7	21	46	Major		Minor	Minor

TABLE PC-IV CONT.

UPPER ZONE

	SIZE MICRONS	WT% OF CLAY FRACTION	FRANCOLITE	DOLOMITE	QUARTZ	CLAY	SMECTITE	SEPIOLITE	ILLITE /MICA	KAOLINITE
CM2	+44	3.0	25	20*	55					
	2 X 44	46.5	20	22*	48	10	Minor		Minor	
	- 2	50.5	6	12	7	75	Major		Major	
		100.0	13	17	28	42	Moderate		Moderate	
CR1	+44	2.1	15	1	84					
	2 x 44	35.7	16	19	59		Minor			
	- 2	62.2	4	11	10	75	Major		Minor	Minor
		100.0	9	14	29	48	Major		Minor	Minor
AVG	+44	4.6	30	7*	63					
	2 x 44	31.5	38	10*	46	5	Minor		Minor	Minor
	-2	63.9	11	8	7	74	Moderate	Tr	Moderate	Moderate
		100.0	20	9*	22	49	Moderate	Tr	Minor	Moderate

*** includes some calcite**

Note: No Palygorskite was found in any of the samples

TABLE PC-IV CONT.

LOWER ZONE

	SIZE MICRON	WT% OF CLAY FRACTION	FRANCOLITE	DOLOMITE	QUARTZ	CLAY	SMECTITE	PALYGO-RSKITE	SEPIOLITE	ILLITE/ MICA	KAOLINITE
CA3	+ 44	6.9	5	77	18						
	2 x 44	79.3	1	96	3						
	- 2	13.8	7	10	8	75	Major		Moderate	Moderate	Major
		100.0	2	83	5	10	Minor		Minor	Minor	Minor
CA4	+ 44	13.7	10	10	80						
	2 x 44	44.3	4	52	34	10	Minor			Tr	
	- 2	42.0	4	6	5	85	Major		Moderate	Moderate	Minor
		100.0	5	27	28	40	Moderate		Minor	Minor	Minor
CE4	+ 44	2.8	7	30	63						
	2 x 44	74.7	1	94	5		Tr			Tr	
	- 2	22.5	4	11	5	80	Moderate	Moderate	Moderate	Moderate	Minor
		100.0	2	75	5	18	Minor	Minor	Minor	Minor	Minor
CH2	+ 44	7.7	10	38	52						
	2 x 44	71.9	3	80	17						
	- 2	20.4	6	10	4	80	Major	Moderate	Moderate	Moderate	Minor
		100.0	4	62	17	17	Moderate	Minor	Minor	Minor	Minor

TABLE PC-IV CONT.

LOWER ZONE

	SIZE MICRONS	WT% OF CLAY FRACTION	FRANCOLITE	DOLOMITE	QUARTZ	CLAY	SMECTITE	PALYGORSKITE	SEPIOLITE	ILLITE/ MICA	KAOLINITE
CR2	+ 44	4.8	5	60	35						
	2 x 44	66.0	1	95	4						
	- 2	29.2	3	7	5	85	Moderate	Moderate			
		100.0	2	67	6	25	Moderate	Minor			
AVG	+ 44	7.2	7	43	50						
	2 x 44	67.2	2	86	12						
	- 2	25.6	5	10	5	80	Major	Minor	Moderate	Moderate	Minor
		100.0	3	64	13	20	Moderate	Minor	Minor	Minor	Minor

SUMMARY CLAY MINERALOGY - Selected Samples

The average mineral composition for the composite of all three size fractions and the - 2 micron fraction which contains essentially all the clay minerals for all three zones is on TABLE PC-V.

The - 150 mesh phosphatic clay fraction for the LP Zone consists mainly of dolomite with minor amounts of quartz, illite/mica and kaolinite and francolite. This - 150 mesh fractions makes up 26% of the matrix. The - 2 micron fraction consists mainly of illite/mica and kaolinite with a moderate to minor amount of francolite, dolomite and quartz. This - 2 micron fraction makes up about 21% of the -150 mesh and 5 % of the total matrix.

The - 150 mesh fraction of the Upper Zone consists mainly of francolite with moderate amounts of quartz, smectite and illite/mica, and makes up about 12% of the matrix. The - 2 micron consists mainly of francolite, quartz, smectite, illite/mica and kaolinite with minor quartz and dolomite. This - 2 micron fraction makes up 64% of the -150 mesh material and about 8% of the matrix.

The - 150 mesh phosphatic clay fraction of the Lower Zone consists dominantly of dolomite with moderate quartz and smectite, and minor amounts of francolite, palygorskite, sepiolite, illite/mica and kaolinite. The average Lower Zone makes up 42 % of the matrix. The - 2 micron consists mainly of smectite, moderate sepiolite, dolomite and illite/mica, and minor francolite, quartz, palygorskite and kaolinite. The - 2 micron fraction makes up 26% of the -150 mesh material, and about 11% of the matrix for the Lower Zone.

TABLE PC-V

SUMMARY - MINERALOGY OF PHOSPHATIC CLAYS

	Minus 150 Mesh Fraction			Minus 2 Micron Fraction		
MINERAL	LP	UPPER	LOWER	LP	UPPER	LOWER
Francolite	8	20	3	14	11	5
Dolomite	70	9	64	10	8	10
Quartz*	7	22	13	6	7	5
Calcite	--	Tr	--	--	Tr	--
Clays	15	49	20	70	74	80
Smectite	--	Moderate	Moderate	--	Moderate	Major
Palygorskite	--	--	Minor	--	--	Minor
Sepiolite	--	Tr	Minor	--	Tr	Moderate
Illite/Mica	Moderate	Minor	Minor	Major	Moderate	Moderate
Kaolinite	Moderate	Moderate	Minor	Major	Moderate	Minor
AVG WT% in Matrix	26	12	42	5	8	11
AVG WT% of -2μ in Phos. Clay Fraction			21	64	26	

* includes minor amounts of feldspar

RESULTS - All Splits

Chemical and x-ray diffraction analyses were run on all the as received - 150 mesh splits. The chemical results for all splits are in the third annual report. The averages for the three zones are listed below.

	All Splits	LP	Upper	Lower
P_2O_5	2.6	1.5	6.1	1.7
CaO	18.8	17.7	13.2	20.3
MgO	10.6	11.3	1.7	13.0
Fe_2O_3	1.4	0.8	1.9	1.3
Al_2O_3	2.3	2.0	5.6	1.5
LOI	27.2	29.1	16.8	29.9
A.I.	35.0	37.2	51.4	30.6

The Upper Zone is much higher in P_2O_5 (francolite) and Al_2O_3 (kaolinite) and much lower in MgO (dolomite). X-ray diffraction confirms these chemical mineralogical results.

Consolidation Behavior.

As mentioned in the experimental section, a laboratory centrifuge was used to study the consolidation behavior of the -150 mesh material representing upper and lower zone matrixes. For this purpose, only a few holes were tested.

The following models were developed to represent the consolidation behavior of different splits as indicated:

CM1

$$e_t = e_\epsilon \exp^{-kt} + 1.04 e_\epsilon \sigma^{-0.26}[1 - \exp^{-kt}]$$
$$k = 5.13 \times 10^{-6} \times N \times e_\epsilon^{1.53}$$
$$K = 6.15 \times 10^{-7} e^{4.95}$$

CM2

$$e_t = e_\epsilon \exp^{-kt} + 1.61 e_\epsilon \sigma^{-0.302}[1 - \exp^{-kt}]$$
$$k = 3.77 \times 10^{-7} \times N + e_\epsilon^{2.08}$$
$$k = 9.78 \times 10^{-8} e^{4.19}$$

Note: e_ϵ = initial void ratio

CR1

$$e_t=e_\epsilon \exp^{-kt}+1.24e_\epsilon \sigma^{-0.213}[1-\exp^{-kt}]$$

$$k=2.87\times10^{-7}\times N+e_\epsilon^{1.78}$$

$$k=7.91\times10^{-9}e^{4.86}$$

CR2

$$e_t=e_\epsilon \exp^{-kt}+0.77e_\epsilon \sigma^{-0.194}[1-\exp^{-kt}]$$

$$k=1.07\times10^{-6}\times N\times e_\epsilon^{1.82}$$

$$K=2.53\times10^{-6}e^{3.52}$$

Note: e_ϵ = initial void ratio

Using the void ratio model for an initial height of 1.0 ft. (30.5 cm), e_{in} = 20.0 and N= 30g, the following plots of height of clays vs. centrifuge time are generated and plotted in Figures 4-5. The shown data indicate that there is no consistent behavior of clays. In other words, some lower matrix clays consolidate faster than the top cores. For instance, Core CR2 (Figure 5) shows faster consolidation of the clays from the lower matrix. In order to explain these results, the mineralogical content of each of the studied splits are given in Table VI. In the same table, the consolidation rate is described qualitatively. These preliminary data indicate slower rates always correlate with higher clay content especially if montmorillonite and/or palygorskite are present.

Using the permeability models for a pond of 30 feet high (corresponding to 1.0 ft. model height and N = 30g), the following tables VII and VIII show the calculated consolidation behavior as a function of time in years.

It is important to note that these data correlate well with the plots from the centrifugal times.

Confirmation of the validity of the developed models need to be determined.

Figure 4

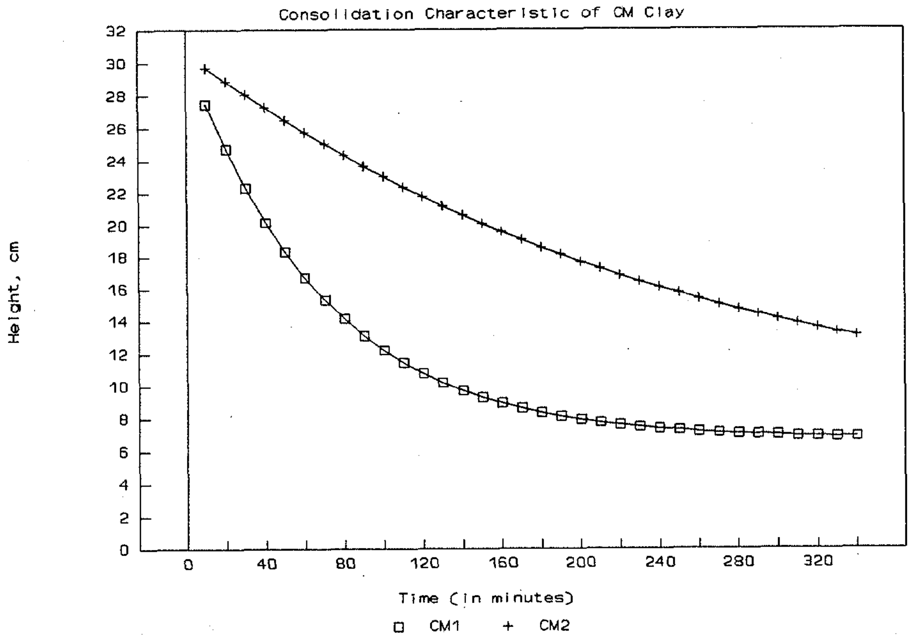


Figure 5

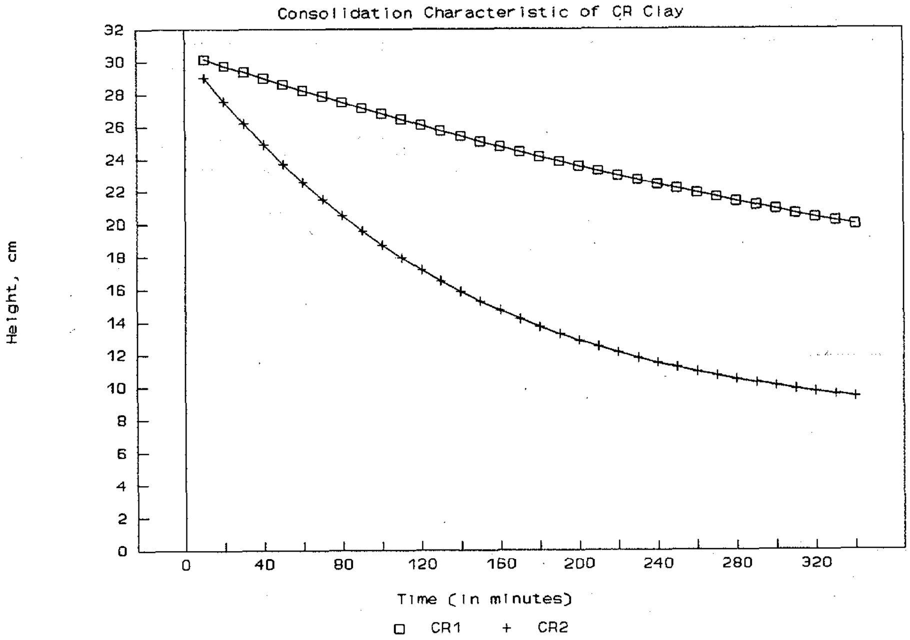


Table PC-VI

Correlation of Phosphatic Clays Mineralogy (% content) and the Consolidation Rate

	SAMPLE NUMBER			
MINERAL	CM1	CM2	CR1	CR2
Francolite	8	13	9	2
Dolomite	70	17	14	67
Quartz	7	28	29	6
Clays	15	42	48	25
Smectite		Moderate	Major	Moderate
Palygorskite				Moderate
Sepiolite				
Illite	Minor	Moderate	Minor	
Kaolinite	Minor		Minor	
-2 micron	21	50	62	29
Rate of Consolidation	Fast	Slow	Slow	Fast

Table PC-VII

Consolidation Characteristics of Phosphatic Clay from Hole # CM, Splits # 1 & 5

Elapsed Time Years		Height, ft	Void Ratio	% Solids, Wt
Split #1	Split #2			
0.00	0.00	30.00	20.00	13.00
0.00	0.07	19.30	17.00	15.00
0.01	0.45	13.90	12.00	20.00
0.03	1.10	11.80	10.00	23.10
0.10	3.13	9.60	8.00	27.30
0.47	11.53	7.50	6.00	33.30
3.82	68.91	5.40	4.00	42.90

Table PC-VIII

Consolidation Characteristics of Phosphatic Clay from Hole # CR, Splits # 2 & 4

Elapsed Time Years		Height, ft	Void Ratio	% Solids, Wt
Split #1	Split #2			
0.00	0.00	30.00	20.00	13.00
0.13	0.02	19.30	17.00	15.00
1.06	0.09	13.90	12.00	20.00
2.90	0.20	11.80	10.00	23.10
9.61	0.49	9.60	8.00	27.30
42.90	1.48	7.50	6.00	33.30
336.55	6.74	5.40	4.00	42.90