

**CONCENTRATION PROCESSES FOR LOW GRADE
CHILEAN PHOSPHATES AND THE PREPARATION OF
UTILIZABLE PRODUCTS THEREFROM**

by

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Purpose

Upon undertaking this work, it was the contention that preparation of a salable phosphatic fertilizer could be accomplished from the ores available in Chile, South America, as guano deposits. The purpose of the study was to prove or disprove this contention.

Contents

	<u>Page</u>
Abstract	i
Acknowledgments	ii
Purpose	iii
List of Tables	v
List of Illustrations	vii
Introduction	1
Description of Deposits	3
Some Properties of the Ore	8
Hydraulic Classification	34
Wet Screening	52
Tabling	56
Spiraling	59
Flotation	69
Pneumatic Concentration	92
Filtration	96
Conclusions and Recommendations	105
Items for Further Research	109
Bibliography and Author Index	110

List of Tables

<u>Table No.</u>	<u>Title</u>	<u>Page</u>
1	Sizing Analyses of Unground Phosphatic Sands (Individual Percent Retained)	13
1 (contd.)	Sizing Analyses of Unground Phosphatic Sands (Cumulative Percent Finer)	14
2	Loss of Weight by Heating	25
3	Distribution of Phosphoric Acid According to Sizing Analysis (Sacks No. 6 and No. 28)	28
3 (contd.)	Phosphoric Acid Content According to Ore Sizes (Sample used in Experiments)	29
4	Proximate Analysis of Raw Material	31
5	Sieve Analyses of Feed and Products (Run No. 2)	40
6	Grades in Overflow (Run No. 2)	41
7	Grades in Spigot (Run No. 2)	41
8	Proximate Composition of Overflow (Run No. 2)	42
9	Proximate Composition of Spigot (Run No. 2)	42
10	Sieve Analyses of Feed and Products (Run No. 3)	46
11	Proximate Composition of Overflow and Spigot (Run No. 3)	47
12	Sieve Analyses of Feed and Products (Run No. 4)	48

List of Tables (contd.)

<u>Table No.</u>	<u>Title</u>	<u>Page</u>
13	Proximate Composition of Overflow and Spigot (Run No. 4)	49
14	Results for Hydraulic Classification	51
15	Separation and Grades for Spiral Tests	65
16	Sizing Analyses of Spiral Products	65
17	Efficiencies in the Spiral	67
18	Results of Pneumatic Concentration	93
19	Composition of Pneumatic Concentration Products	93
20	Sizing Analyses of Pneumatic Concentration Products	94

List of Illustrations

<u>No.</u>		<u>Page</u>
	Map of Chile, between parallels 20 and 24	4
1	Microphotography of Unground Phosphatic Sand	11
2	Sizing Analysis of Unground Phosphatic Sand. Average of Seven Samples	15
3	Sizing Analysis of Unground Phosphatic Sand. Sample used in Experiments	16
4	Microphotograph of the 10/14-mesh Fraction	17
5	Microphotograph of the 20/28-mesh Fraction	17
6	Microphotograph of the 35/48-mesh Fraction	18
7	Microphotograph of the 48/65-mesh Fraction	18
8	Microphotograph of the 100/150-mesh Fraction	19
9	Microphotograph of the -150 mesh	19
10	Microphotograph of Shell-like Particles	22
11	Microphotograph of Quartz Grains	22
12	Plot of P_2O_5 Content According to Sizes	30
13	Equipment Arranged for Experimental Hydraulic Classification	37
14	Flow Diagram for Experimental Hydraulic Separation	38
15	Pilot Table "Overstrom Universal"	57
16	Crosswise Cycle in Spiraling	60

List of Illustrations (contd.)

<u>No.</u>		<u>Page</u>
17	Concentrating Action of Spiral	61
18	Spiral Concentrator. 5 Turn. Laboratory Test Unit	62
19	Spiral Concentrator. Close-up View. Laboratory Test Unit	63
20	Microphotography of Flotation Tailings	84
21	Microphotography of Product from Pneumatic Concentration	95
22	Plot of Results for Filtration at 5.2 psi	98
23	Plot of Results for Filtration at 10.2 psi	99
24	Plot of Results for Filtration at 15.2 psi	100
25	Graph for the Variation of Cake Resistance with Pressure	101
26	Curves for Washing Rates at Different Pressures	102
27	Filter Cloth Resistivity at 5.2 psi	103
28	Filter Cloth Resistivity at 15.2 psi	104
29	Proposed Flow Diagram for Hydraulic Concentration	107
30	Proposed Flow Sheet for Pneumatic Upgrading	108

CONCENTRATION PROCESSES FOR LOW GRADE CHILEAN PHOSPHATES AND THE PREPARATION OF UTILIZABLE PRODUCTS THEREFROM

INTRODUCTION

The Chilean market for phosphatic fertilizers has been particularly fond of a natural phosphate-bearing material. This commodity, which was obtainable in northern Chile, had a phosphoric acid content of slightly above 20% P_2O_5 . As indicated by the solubility in acid citric, the phosphorus was readily available. Furthermore, the coexistence of amounts, though small, of Potassium salts and of Nitrogen promoted the favorable consideration by consumers. The deposits now are about depleted. In approximately the same location, low grade phosphates of around 6% P_2O_5 have been found. This phosphorus is also readily available, but the P_2O_5 content is too low to permit economic shipment, handling, and use. Centers of consumption are located at least 1500 kilometers away from the deposits. Information regarding total possible tonnage is not available to the writer; however, he has been officially informed that should an efficient process be found for the economical upgrading of this ore, a medium-size plant will be constructed as an initial operation. It is also known that attempts have been made in Chile to attain significant concentration, but tests, as yet, have been unsuccessful. The author does not have

information regarding the nature of those attempts. It was with the thought of providing a solution to the concentration problem that this work was undertaken.

Measurement of the success or failure of procedures could be made by reference to established specifications.

Description of Deposits

The already known deposits are located on the Chilean coast, not farther than 3.5 kilometers inland. Lengthwise, they are distributed between parallels 21 and 23, South of the Equator. Within these boundaries, the deposits are scattered irregularly. The best known places appear on the map, page 4, underlined in red. Their altitude varies between a few meters and 500 meters above sea level. The beds are not uniform in dimensions, and are continuously interrupted with valueless material. They are commonly in ancient, dry creeks; cavities, in crevices, and in cracks. Their thickness is variable, sometimes reaching up to three meters. The samples upon which this investigation was conducted are from Punta de Lobos. In this location, the deposits are about three kilometers long and 0.5 km. wide.

There is no overburden, as a rule. In few places, the deposits have been slightly covered by a sandy clay accumulated by the wind. The material in situ is not consolidated. Because of the lack of cementing agents and vehicles, the material is easy to disintegrate, even that extracted from the bottom layers.

The ore is found on the surface as a loose agglomerate of grains of different sizes. The main components are silica, calcium carbonate and a brown phosphate-bearing complex. Also found, although in much less quantity, are: masses of



long crystals surrounded by sand and other cemented materials; concretions of crystals, probably gypsum structures; incrustations; fish vertebrae, and even bones of small mammals, likely turtles; bundles of mineralized fibers, etc.

It does not seem likely that the brown phosphate-bearing complex has its origin in bones because practically no calcium carbonate is present therein. The calcium carbonate present in the composite of unconsolidated mixture can be easily identified, macro and microscopically, as parts of marine shells and as small shells, which are completely soluble in acid hydrochloric. Moreover, the higher content of P_2O_5 is found in the finer fractions; in these, not even fragments of bones could be identified.

The phosphate-bearing complex could be the result of animal depositions over a ferruginous alkaline ground, which is typical of that part of the country; the action of animal organic acids upon the iron being responsible for the brownish shade of the complex. Observations made during the experiments lead to think that such organic-inorganic complex is of an acidoid nature. The fibers found are definitely not of feather structure, nor hair-like either. Not enough evidence has been accumulated to establish whether those animals were birds or mammals.

The almost complete solubility of the phosphatic portion in ammonium citrate discards the possibility of a

mineral origin. Partial investigation of the water-soluble fraction of the phosphate-bearing complex showed the presence of Sodium, Potassium, Chlorides, and traces of Sulphates. From these observed facts, the tentative conclusion that these deposits originated as a result of animal life in the neighborhood of the ocean can be derived. Investigation of Bromine, Iodine, other minor elements of marine nature, as well as further research upon the fossils remains found should throw more light on the question of the origin of this organic phosphatic rock. While the marine remains present,--mainly shells--, are characteristic of the Upper Cretaceous time, the organic excrementitious rock has been originated later, in the Quaternary period. The underlying rock in the Coast Range, which is not older than the Mesozoic time, is granitoid.

The land belongs to the Chilean State. The region is entirely barren, with no atmospheric precipitation whatsoever. No fresh water is available on the coast at those latitudes, except in ports supplied from the Andes by pipeline. Sea water is the only liquid that could be used in any benefitiation plant of commercial importance. Favored fuels in Northern Chile are: coal shipped from the South and oils imported from foreign countries. Larger nearby ports such as Iquique, Tocopilla, and Antofagasta can be easily reached by domestic planes, liners, or trains. In order to go from there to smaller ports or coves in

between, such as Mejillones and Punta de Lobos, use should be made of automobiles, motor boats, or coasting trade ships. All these places are connected to the main telegraph and telephone lines.

In 1943, funds were allocated to conduct a survey and drilling. It is not known whether or not this work has been done already. No information has been available as to tonnage of ore estimated. Further information concerning the appearance and chemical as well as size composition will be found in subsequent parts of this writing.

Some Properties of the Ore

Classification of Sacks of the Material Received from Punta de Lobos, Chile

Two metric tons of material for experimentation were received from Chile. The sacks were shipped from Iquique to New York by boat; from New York to Corvallis, Oregon, by rail, and trucked to the Chemical Engineering Department. As stated before, this sample was taken from the deposits of Punta de Lobos (see Map). The ore arrived in thirty sacks, and each one was given a tag, serially numbered from No. 1 to No. 30. This means of identification is used in this writing. Upon preliminary inspection, it was found that the phosphatic sand was not entirely uniform in size throughout the thirty sacks. Some sacks contained rock with few coarse grains, and in others, the material was non-uniform. No other differences could be determined by visual inspection. To provide representative samples for experimentation and the measurement of differences, the thirty sacks were divided in two groups: Group A, and Group B.

Group A: Sacks containing rock with few coarse grains, and of uniform appearance.

Group B: Sacks containing rock with abundant larger grains, of uneven appearance.

<u>Group A</u>					<u>Group B</u>				
Sacks No:	1	2	3	6	Sacks No:	4	5	8	9
	7	10	12	16		11	13	14	15
	17	19	23	25		18	20	21	22
	26	27	29	30		24	28		
Total: Sixteen Sacks					Total: Fourteen Sacks				

Color

The rock has a light brown shade (cinnamon). Upon close examination, white as well as dark-grey particles can be seen. The material is not phosphorescent under violet light.

Fluidity

The ore flows easily. It is dusty, but particles of dust settle fairly quickly in the air; the dust has a high absolute density.

Odor

When the material is handled and particles of the dust are floating, a slight odor is apparent. The odor is too sui generis to be described. After the particles settle, there is no odor. The material has been stored in a closed room for months where no particular odor can be detected. Apparently, there is no matter which is volatile at room temperature.

Hygroscopicity

The rock is not hygroscopic. Even more, when moistened or wet samples were left in a room under usual conditions, they always dried to the same degree as the original samples. When wet, the material is darker in color.

Bulk Specific Gravity

Determinations were made following the technique prescribed by the American Society for Testing Materials(1) A 250-c.c. Le Chatelier flask was used. Five determinations were made in all; two on sacks of Group A, and three on sacks of Group B.

<u>Group A</u>		<u>Group B</u>	
<u>Sack No.</u>	<u>Bulk Sp. Gr.</u>	<u>Sack No.</u>	<u>Bulk Sp. Gr.</u>
6	2.86	9	2.70
19	2.78	15	2.72
		28	2.70

Size Composition

A substantial idea of the appearance of the ore may be obtained from Fig. No. 1, page 11, which is a microphotograph of the whole, just as received. The presence of particles of different sizes in this unground, natural material, suggested a size analysis; this should eventually lead towards the identification of the major constituents.



Fig. 1 **Unground Phosphatic Sand**

The Tyler Standard Screen-scale and sieves were adopted and used throughout this work. The equipment consisted of the following:

One set of Tyler Standard Screen-scale Sieves
The W. S. Tyler Co., Cleveland, Ohio.

One RO-TAP Mechanical Sieve Shaker; No. 4218; actuated by a 1/3 h.p. General Electric motor.

One Tyler Automatic Timer; Model 8033B.

One Technical Balance. Sensitivity: 0.1 gram.

The weight of samples used in size analyses throughout this investigation varied, according to the predominant degree of fineness, from ten grams up to fifty grams. Samples were always shaken for ten minutes, which time proved to be sufficient to cause all corresponding sizes to pass through the respective sieves.

Seven sacks (1-6-12-19-25-28-30) were selected for sieve analyses. Results have been arranged in Table 1, appearing on page 13; they are also plotted in Figs. 2-3, pages 15 and 16. Examination of the figures indicate that for each and every particular size, the percentages retained are quite uniform for the seven sacks. Sacks No. 19 and 28 are richer in coarser particles and correspondingly low in the finer grains. Upon consideration of the average, it is seen that only four sizes; namely: -14/+20, -20/+28, -28/+35, and -35/+48, are responsible for more than half of the weight, i.e., 54 per cent; also, that almost one fifth of the material goes

TABLE I. SIZING ANALYSES OF UNGROUND PHOSPHATIC SANDS

Individual Percent Retained

No.	Sack No.	+ 10 Mesh	-10 +14	-14 +20	-20 +28	-28 +35	-35 +48	-48 +65	- 65 +100	-100 +150	- 150 Mesh	Total
1	1	8.1	3.4	12.6	18.8	11.7	9.1	7.2	6.5	3.5	19.1	100.0
2	6	3.8	3.7	14.0	19.6	11.9	9.5	7.4	7.1	3.8	19.2	100.0
3	12	4.9	3.9	13.0	17.9	11.4	9.0	7.5	7.4	4.1	20.9	100.0
4	19	4.1	3.8	14.2	20.1	12.3	10.0	8.2	7.4	3.5	16.4	100.0
5	25	3.9	4.0	12.2	17.1	11.2	9.5	7.8	8.0	4.8	21.5	100.0
6	28	3.0	5.5	16.6	20.2	12.3	11.0	7.9	6.7	3.1	13.7	100.0
7	30	2.4	3.4	12.7	18.8	12.4	10.3	8.4	7.9	4.5	19.2	100.0
	Ave	4.32	3.96	13.61	18.93	11.90	9.78	7.77	7.29	3.90	18.57	100.0

Table 1 (Contd)
Sizing Analyses of Unground Phosphatic Sands

Cumulative Percent Finer										
No.	Sack No.	Plus	14 Mesh	20 Mesh	28 Mesh	35 Mesh	48 Mesh	65 Mesh	100 Mesh	Minus
		10 Mesh								150 Mesh
1	1	91.9	88.5	75.9	57.1	45.4	36.3	29.1	22.6	19.1
2	6	96.2	92.5	78.5	58.9	47.0	37.5	30.1	23.0	19.2
3	12	95.1	91.2	78.2	60.3	48.9	39.9	32.4	25.0	20.9
4	19	95.9	92.1	77.9	57.8	45.5	35.5	27.3	19.9	16.4
5	25	96.1	92.1	79.9	62.8	51.6	42.1	34.3	26.3	21.5
6	28	97.0	91.5	74.9	54.7	42.4	31.4	23.5	16.8	13.7
7	30	97.6	94.2	81.5	62.7	50.3	40.0	31.6	23.7	19.2
Ave.		95.69	91.73	78.11	59.18	47.30	37.53	29.76	22.47	18.57

Fig. 2

SIZING ANALYSIS OF UNGROUND PHOSPHATIC SANDS

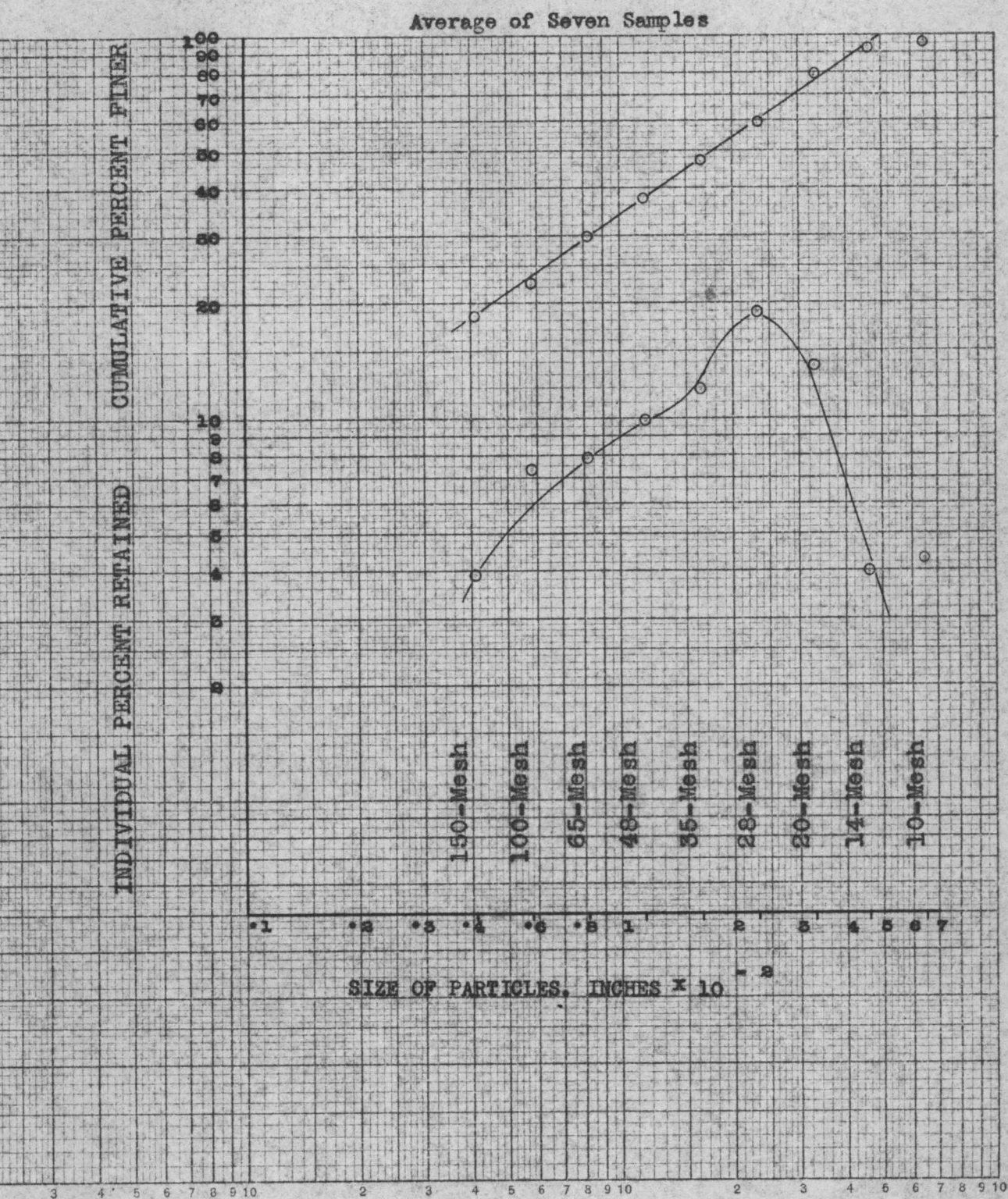
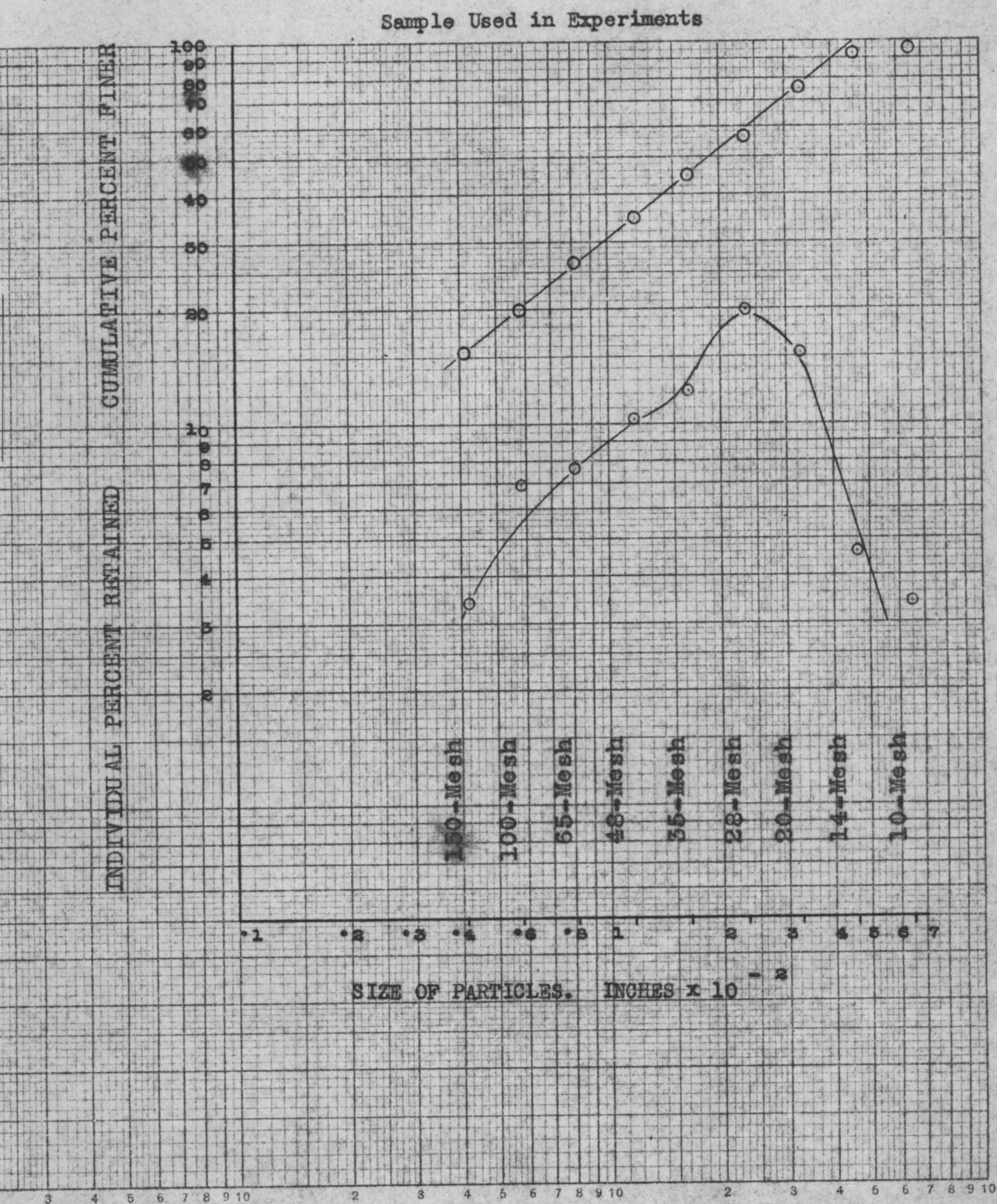


Fig. 3

SIZING ANALYSIS OF UNGROUND PHOSPHATIC SANDS

16



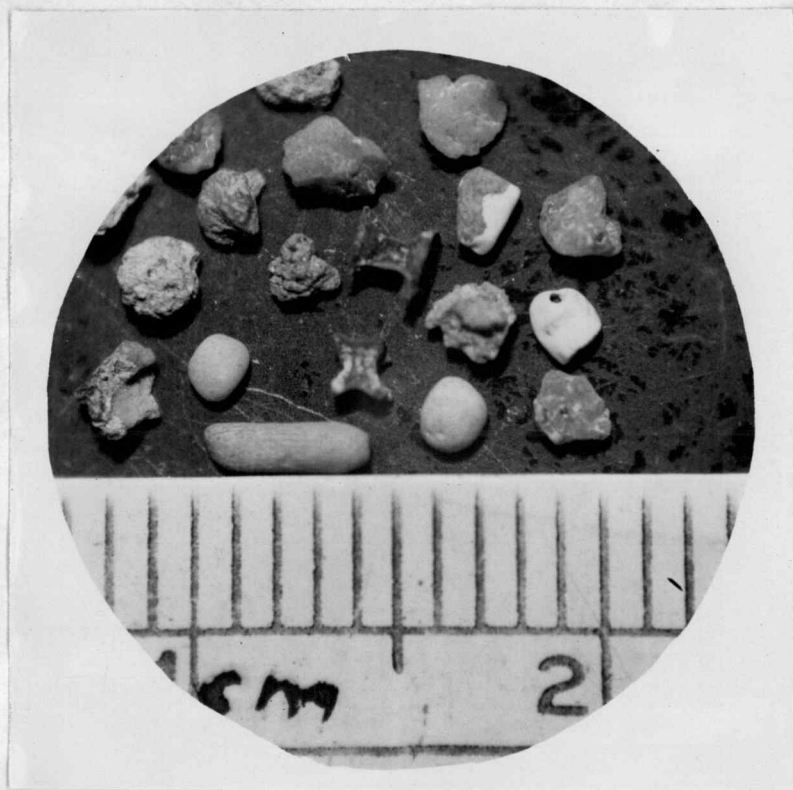


Fig. 4 Unground Phosphatic Sand
10/14 - mesh

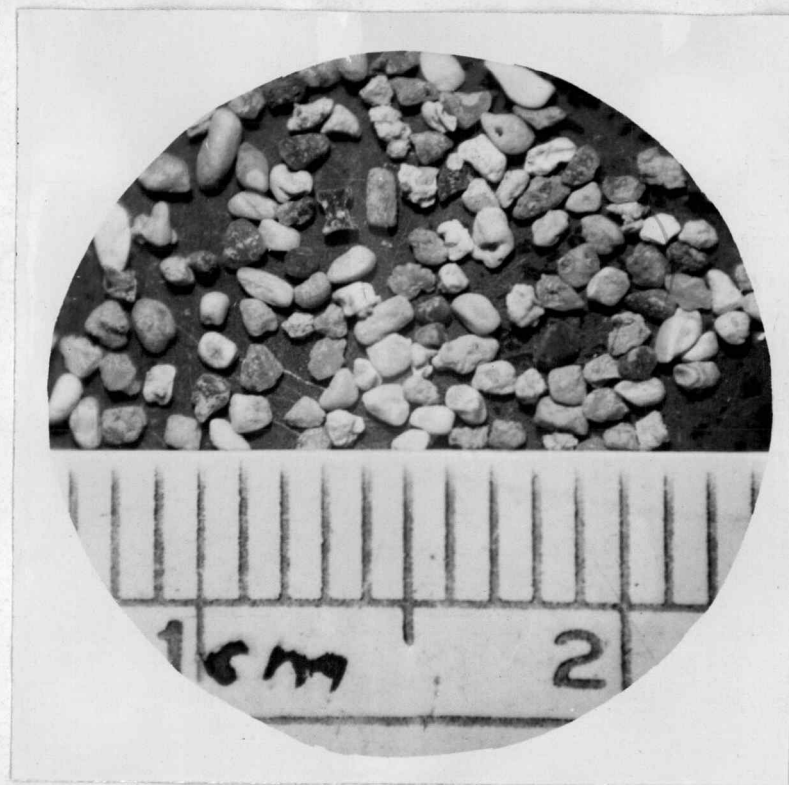


Fig. 5 Unground Phosphatic Sand
20/28 - mesh

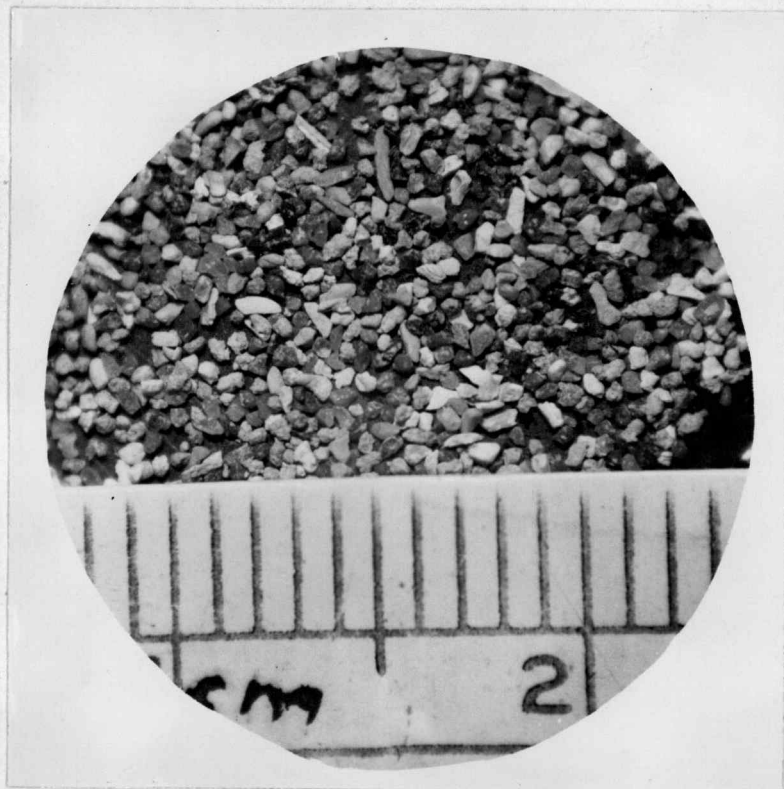


Fig 6 Unground Phosphatic Sand
35/48 - mesh

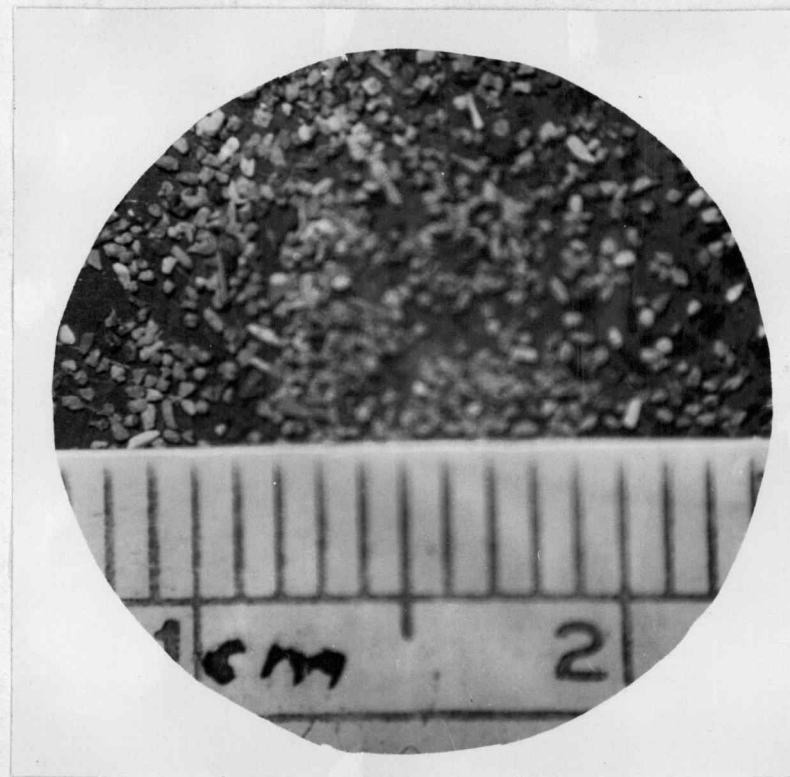


Fig 7 Unground Phosphatic Sand
48/65 - mesh

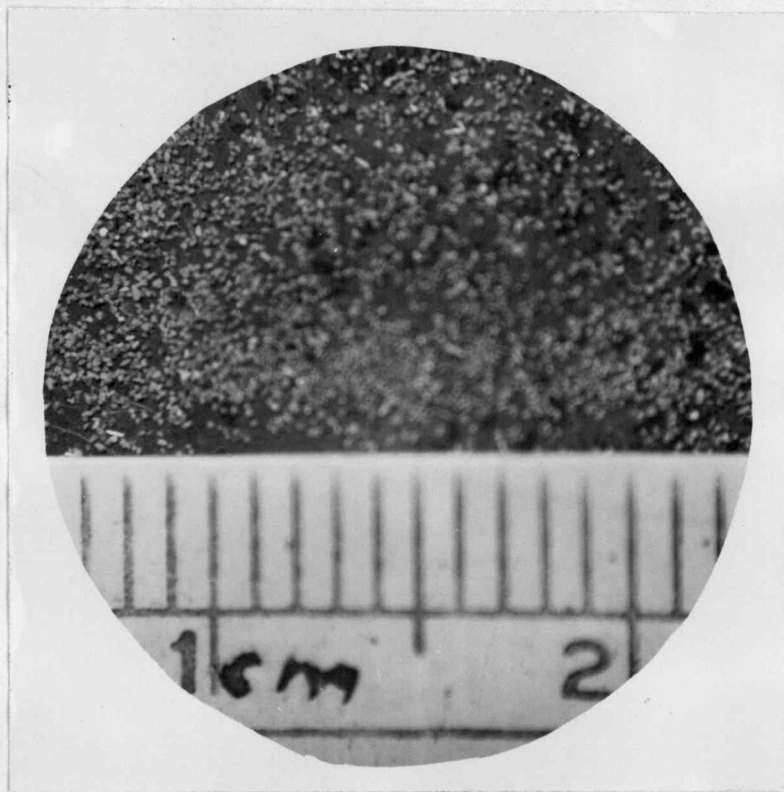


Fig. 8 Unground Phosphatic Sand
100/150 - mesh

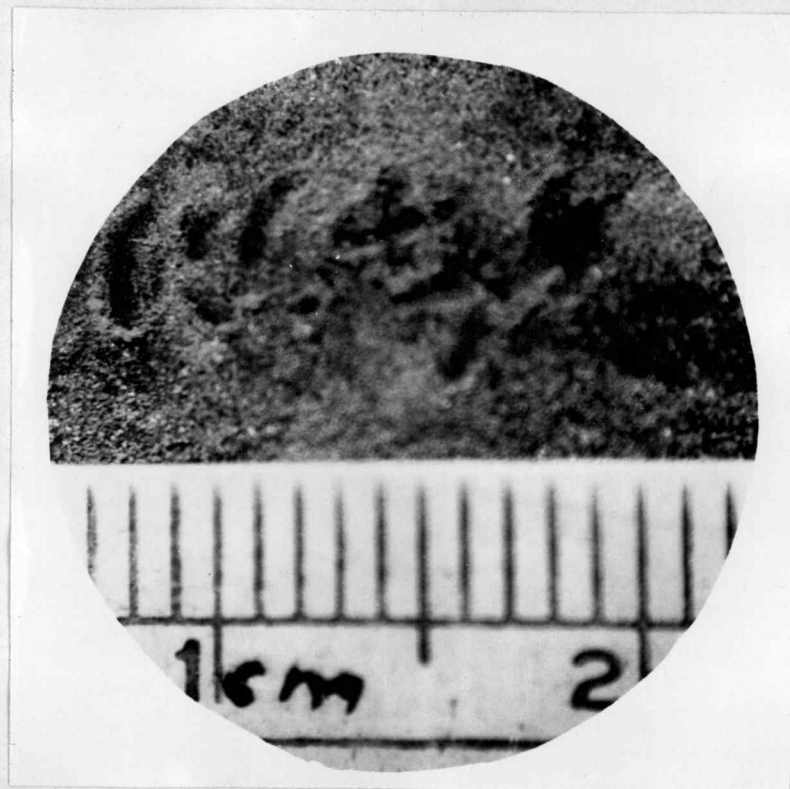


Fig. 9 Unground Phosphatic Sand
Minus 150 mesh

through 150-mesh. Sacks No. 6 and 28 were considered to be exactly representative of Groups A and B, respectively. Aspects of the several fractions of a mixture 1:1 of material from Sacks No. 6 and 28, are shown in Figs. 4-5-6-7-8-9, pages 17, 18, and 19.

Dimensions, in terms of the Tyler Standard Screen-scale, are given below⁽⁶⁾.

Mesh No.	Openings	
	mm.	inches
10	1.651	0.065
14	1.168	0.046
20	0.833	0.0328
28	0.589	0.0232
35	0.417	0.0164
48	0.295	0.0116
65	0.208	0.0082
100	0.147	0.0058
150	0.104	0.0041

All the remarks and results following in the rest of this chapter refer to a sample obtained by mixing ore from Sacks No. 6 and 28 in equal parts. It is thought that the results are applicable to the entire sample as previously indicated.

White, flat, round-edged, shell-like particles abound in the larger sizes. They are the hardest of all components to grind. Some were hand-picked and their chemical composition determined. Fig. 10 is a photograph of these particles. Reference to analytical procedure is made in

further part of present sub-chapter.

Chemical composition of the white, flat, round-edged particles:

Calcium Carbonate	98.1%
Tricalcium Phosphate	0.6
Insoluble in A.R.	1.1
Total	<u>99.8%</u>

Apparently they are marine fossils, parts or shells of mollusks. The presence of small amounts of phosphorus and silica might be explained by contamination.

Crystalline, irregularly broken, light colored grains are also abundant in the coarser fractions. In a fashion similar to that mentioned above, their composition was determined:

Calcium Carbonate	6.4%
Tricalcium Phosphate	2.9
Insoluble in A.R.	90.1
Total	<u>99.4%</u>

Undoubtedly, these grains are free quartz.

Bluish grey, sharp-edged grains are present in the intermediate sizes. When ground, the resultant powder has a brownish grey color. Its chemical composition is:

Calcium Carbonate	11.1%
Tricalcium Phosphate	4.2
Insoluble in A.R.	80.1
Undetermined	4.6
Total	<u>100.0%</u>

These are quartz grains, probably contaminated by or combined with metallic oxides. A picture appears on page 22, Fig. 11.

Two more types of grains are evident in very small



Fig 10 White, shell-like particles



Fig 11 Gray, sharp-edged quartz

quantity. These were separated during the Tabling experiments, and their sizes vary from 0.3 mm. to 0.1 mm. One type is red, floats in Bromoform, and was identified as a variety of clay. The second type is black and strongly magnetic. It sinks in Bromoform, appeared in the upper spigots of the Spiral, and is probably magnetite. No subsequent identification was attempted.

Saline Materials: One portion of the sample was leached with five times its weight of cold water. Color was not affected. Sodium, Potassium and Chlorides were determined in aliquots. Sodium was determined by the Uranyl Acetate method. Potassium was precipitated with Cobalti-nitrite and the precipitate titrated with Cerium Nitrate and Sodium Oxalate. Chlorides were titrated with Silver Nitrate. The following results refer to percentages by weight of original ore:

Sodium Chloride (Na Cl)	3.0%
Potassium Chloride (K Cl)	1.3%

Upon evaporating another aliquot on the steam bath nothing remained but a saline residue.

Organic Matter: A portion of the original sample was leached several times with boiling water. The residual ore had same shade as before. Upon evaporating the bulk of filtrate and washings on the steam bath, a slightly yellow jelly-like residue was obtained. Its odor is exactly the same as that of the original ore. This residue charred

upon heating with a direct flame. It certainly constitutes the organic part of the phosphate-bearing complex. It is insoluble, or almost so, in cold water, but dissolves in hot water. Total Nitrogen was also determined in the original ore. The Kjeldahl modified technique was followed; the distillate was collected in boric acid and titrated with standardized acid. A blank was simultaneously run. The result was:

Nitrogen (N)	0.39%
--------------	-------

Behavior Upon Heating: An electric furnace was used to heat the material to 300°C. for two hours. The color of the sample turned into grey. The sample was further heated for two more hours at 800°C. After reaching a light grey, no other change in color was observed. The losses are reported on Table 2.

Table 2. Loss of Weight by Heating

Heating Conditions	Loss of Weight Referred to Original Sample	
	Individual	Cumulative
	%	%
300°C. for 2 hours	6.5	6.5
800°C. for 2 hours additional	18.6	25.1

The loss of organic matter by a hot thorough leaching did not change the original color. Hence, the discoloration of the rock by direct heating must be due to the loss of other substance. It is believed that a dehydration, and eventually a destruction, of the organic-inorganic complex takes place during the heating; probably this involves the transformation of a brown, hydrated ferric phosphate into a white (practically) anhydrous ferric phosphate. A determination of iron by the Zimmermann-Reinhardt procedure indicated a content of 1.5% by weight in the ore, expressed as the metal Fe. The evolution of carbonic acid from the partial decomposition of the calcium carbonate must also account for part of the total loss of weights; a longer and more intense heating would make the transformation into calcium oxide practically complete.

Total Phosphoric Acid, Calcium Carbonate, and Insoluble

Analytical Determinations: As to total phosphoric acid, the alkalimetric method was selected because of its simplicity and accuracy. The technique followed was that given by the Bureau of Standards⁽³⁾. The alkaline solution was standardized against the Bureau of Standards Standard Acid Potassium Phthalate No. 84⁽⁴⁾. The acid solution was standardized against the alkaline solution. The method was double checked at the start: 1) with the Bureau of Standards Standard Sample No. 56 of Tennessee Phosphate Rock⁽³⁾, and 2) with the Bureau of Standards gravimetric method⁽³⁾. Determinations were always made in duplicate; the average was calculated for consistent results. Otherwise, a third determination was made in a few cases. Results are expressed both in phosphoric acid and in B.P.L. (Bone phosphate of lime) as percentage by weight of original ore.

Concerning the determination of total Calcium, an aliquot of the acid solution of the sample was used. Calcium was precipitated with ammonium oxalate. The calcium oxalate was dissolved in perchloric acid and this solution titrated with standardized Cerium Nitrate. Single determinations were made.

The figures for Calcium Carbonate were computed by subtracting the stoichiometric Calcium Oxide corresponding to the phosphoric acid from the total Calcium Oxide. The resulting difference was then stoichiometrically converted

into Calcium Carbonate.

Insoluble in Aqua Regia was determined after the Bureau of Standards method for Phosphate Rock⁽³⁾. Single determinations were made.

It is not claimed that the figures for B.P.L. and for Calcium Carbonate represent the chemical compounds as present in the ore. Probably the iron is linked to a portion of the phosphoric acid. Likewise, some organic calcium may be present. It is thought that the figures for total phosphoric acid are entirely reliable, and that they serve the best for subsequent computations of recoveries and for judging recovery efficiencies.

Distribution of Phosphoric Acid: In order to obtain useful information as to how the phosphatic content is distributed throughout the ore, determinations of P_2O_5 were made in the several sizes. The results, both individual and cumulative for seven samples, are arranged in Table 3, page 28. Fig.12, page 30, is a plot which shows how the respective averages fluctuate with size.

Proximate Composition: Figures for Insoluble, Tricalcium Phosphate and Calcium Carbonate, are reported on Table 4, page 31. The results for two samples: Sacks No. 6 and 28, are included.

From inspection of Table 3, it is seen that the phosphate-bearing material accumulates in the finer sizes; namely, below -100-mesh. Figures on Table 4 show that the

Table 3 (contd.)
Phosphoric Acid Content According to Ore Sizes

Sample Used in Experiments				
Mesh	Retained Indi- vidual Wt. %	Grade Indi- vidual(*) % P ₂ O ₅	Grade Cumulative(*) Finer % P ₂ O ₅	Individual Distribution of P ₂ O ₅ % of Total
None			6.8	
Plus 10	3.4	2.8	7.0	1.4
Plus 14	4.6	2.0	7.2	1.3
Plus 20	15.3	2.8	8.1	6.3
Plus 28	19.9	2.4	10.1	7.1
Plus 35	12.1	3.1	12.0	5.4
Plus 48	10.3	4.4	14.2	6.6
Plus 65	7.7	5.6	16.7	6.3
Plus 100	6.9	8.4	19.6	8.5
Plus 150	3.4	15.5	20.4	7.8
Minus 150	16.4	20.4		49.3

(*) In the computation of these values the corresponding percentages by weight of the fractions retained have also been taken in account.

Sample Used in Experiments

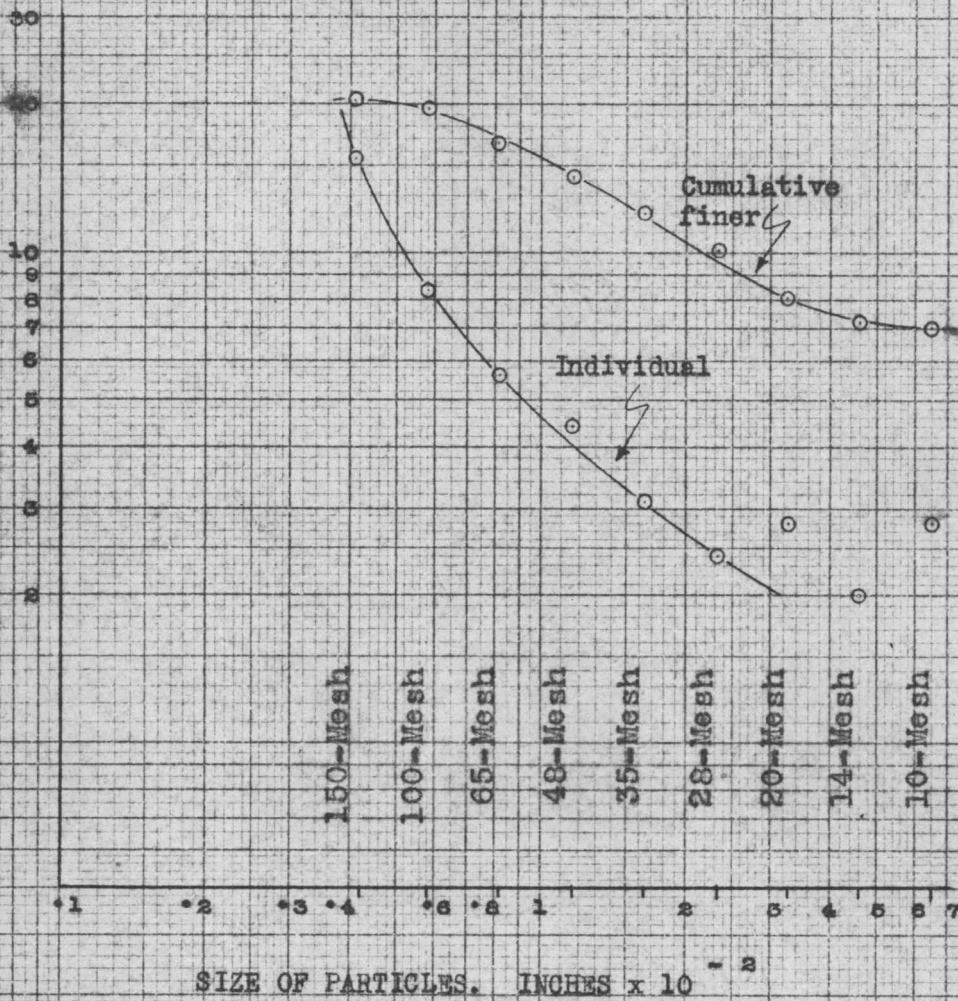
PHOSPHORIC ACID CONTENT. PERCENT P_2O_5 

TABLE 4.- PROXIMATE ANALYSIS OF RAW MATERIAL

Two Samples

Mesh	Insoluble		Ca ₃ (PO ₄) ₂		Ca CO ₃		Undetermined		Total	
	%		%		%		%		%	
	Sack 6	Sack 28	Sack 6	Sack 28	Sack 6	Sack 28	Sack 6	Sack 28	Sack 6	Sack 28
+ 10	19.1	41.8	7.2	4.8	62.3	44.1	11.4	9.3	100.0	100.0
+14	18.9	48.0	6.3	2.8	55.8	24.7	19.0	24.5	100.0	100.0
+ 20	17.1	34.1	6.8	5.2	63.8	59.1	12.3	1.6	100.0	100.0
+ 28	17.9	25.9	5.2	5.5	70.8	65.5	6.1	3.1	100.0	100.0
+ 35	22.5	32.8	7.2	6.3	65.4	58.0	4.9	2.9	100.0	100.0
+ 48	42.3	47.0	10.9	7.5	31.0	30.1	15.8	15.4	100.0	100.0
+ 65	56.7	63.4	14.0	10.5	20.0	19.3	9.3	6.8	100.0	100.0
+100	48.2	55.7	21.2	15.3	12.0	11.4	18.6	17.6	100.0	100.0
+150	25.4	28.7	35.4	27.3	29.4	27.3	9.8	16.7	100.0	100.0
-150	3.0	6.2	49.4	37.8	None	15.8	47.6	40.2	100.0	100.0

quartz predominates in the intermediate fractions, with negligible percentage in the -150-mesh. Calcium Carbonate, however, is the highest in the coarser grains.

"Undetermined" includes organic matter, iron and other minor elements, salts, and water of crystallization. The "undetermined" accounts for almost half of the weight of the -150-mesh fraction. This is an important fact when the upgrading of the ore is considered. Upgrading is the subject of the following chapters.

Specifications Required in the Product

Sizing Analysis

The concentrated material shall pass through Mesh No. 80, (876 meshes per square centimeter). An allowance of 20% of plus 80-mesh material is permitted. In no case shall the fertilizer contain grains larger than 3 millimeters.

Total Acid Phosphoric

The total content of phosphoric acid shall be greater than 18% P_2O_5 .

P_2O_5 Readily Available

The amount of phosphoric acid soluble in acid citric, 2% aqueous solution, is a favorable factor in judging the fertilizer.

Nitrogen and Potash

The presence of Nitrogen and Potash is desirable.

Hydraulic Classification

Sizing Tests on "Dorreo" Fahrenwald Classifier

Statements: The assumptions under which Stokes', Newton's, and Newton-Rittinger's relations hold are far from being met in the case of this ore. Because of the simultaneous presence of small and large particles, neither Newton's or Stokes' formulas apply. As to shape, pictures and screen analyses show that approximately 70% of the material does not have, nor even approximates, the shape of spheres, which is one of the assumptions under which the mentioned relations work. Unless correction factors for sphericity and for crowding conditions were known for this ore, the use of the formulas was considered inadvisable. Moreover, the difference in specific gravities, which is a relevant factor in hydraulic separation, is very small. Because the phosphatic complex contains organic matter, the sp. gr. probably is lower than the 3.14 figure accepted for the pure tricalcium phosphate. If a value of 2.7 is assumed for the sp. gr. of calcium carbonate and of quartz, then there is scarcely a difference of 0.3 in the sp. gr. of the substances which were to be separated.

The considerations for the present case favor an experimental, rather than a strictly theoretical approach of hydraulic classification.

Principles: As hindered-settling is the condition in which separation might take place in commercial scale, a Fahrenwald Sizer was considered suitable for this study. Here, a mixture of grains of different specific gravities, shapes, and sizes, crowded in a thick pulp, is sorted by a rising stream of water. Fines and/or light particles are carried away with the upwards flow, while coarser and/or heavy grains fall. Means are provided to regulate working conditions, and to remove the products of separation.

Purpose: The specific objective of this experiment was to find out whether or not hydraulic classification under hindered-settling conditions was applicable to this ore.

Equipment and Modus Operandi: Main pieces of equipment used were:

- Two Water Meters. No. 1023570.
Buffalo Meter Co., Buffalo, N. Y.
- One "Vibra-flow" Feeder. Type FG. Style 1602.
Ser. No. DF45228. Syntron Co., Homer City,
Pa.
- One Syntron Electric Controller. Type FCO.
Style 4283. Serial No. D2C 45229.
Syntron Co., Homer City, Pa.
- One "Lightnin" Mixer, compressed-air driven.
Model AR 08. Ser. No. 420430.
Mixing Equipment Co., Inc., Rochester N. Y.
- One "Dorreo" Fahrenwald Sizer. 5" x 5" x 12"
sizing column. Ser. No. F-5-D.
The Dorr Co., Inc., New York

One Six-gallons capacity Tank

One Ore Bin

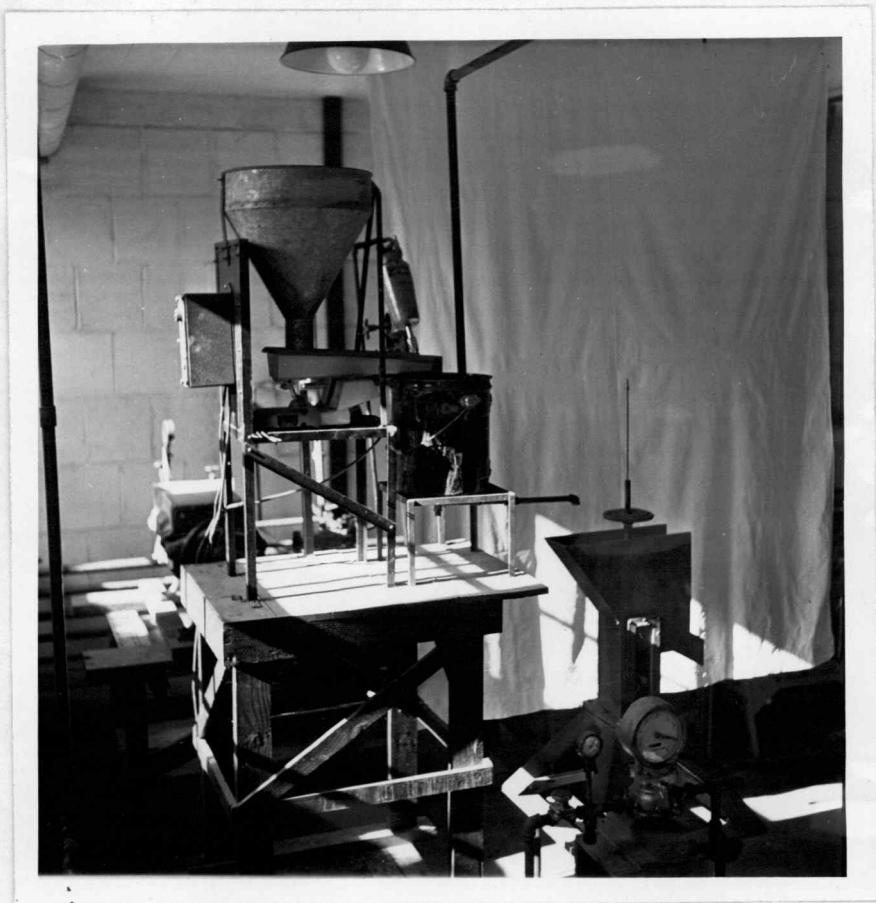
Piping, Fittings, Gages, Stands, Valves, etc.

The equipment was arranged in cascade as shown in Fig. 13, page 37. A Flow-sheet is reproduced in Fig. 14, page 38.

Dry ore was delivered at known rates by Feeder 3 to Mixer 4 where Feed Water was also admitted. Feed water volume was recorded by a Water Meter. From Mixer 4, the pulp went into the Sizer. Here the so-called "Hydraulic Water" entered the system to provide the hydraulic pressure necessary for a rising current of water. While the coarser fractions of the ore settled and were intermittently discharged through the spigot in the bottom, the fines remained in suspension and were separated as the overflow. A teetering effect was maintained in the sorting column.

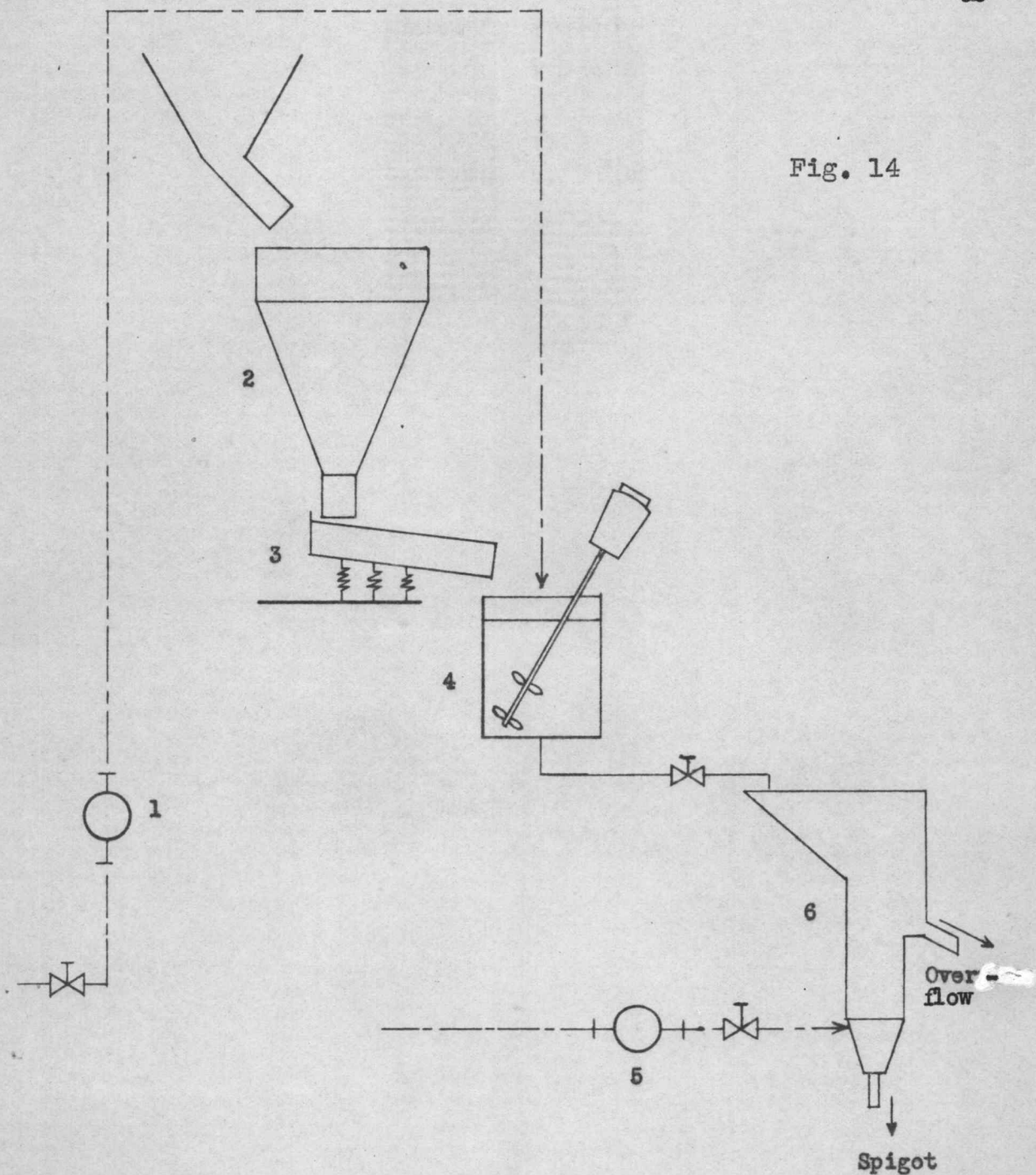
Four tests were made, which will be designated as Runs No. 1, 2, 3, and 4, respectively. As to raw material, everything smaller than 28-mesh was used in Runs 1, 2, and 3. In Run 4, the whole ore was tested. The sample was a mixture of 1:1 of Sacks No. 6 and No. 28. Tap water was used in all cases.

Run No. 1: This test was of a tentative character intended to give approximate information about pulp thickness, rate of feeding ore and water, and range of hydraulic flow. Values compatible with the size of the equipment were desirable and obtained.



**Fig. 13 Arrangement for Hydraulic
Classification**

Fig. 14



1	Feed Water Meter	<p style="text-align: center;">FLOW DIAGRAM FOR EXPERIMENTAL HYDRAULIC SEPARATION OF UNGROUND PHOSPHATIC SANDS</p>
2	Ore Bin	
3	VIBRA-FLOW Feeder	
4	Mixer	
5	Hydraulic Water Meter	
6	Fahrenwald Sizer	

Run No. 2:

Experimental Data:

Size of Ore	-28 mesh	
Pulp Thickness	Ore:Water Ratio	1:4
Ore Fed, in kgs.		3.00
Hydraulic Water, rate of flow, in liters per minute		3.6

Length of Run:

Total of 16 minutes for the whole run,
11 minutes of actual classification.
Operation was continuous and constant.

Fines Recovered (dry), in kgs.	1.32
Spigot Discharged (dry), in kgs.	1.52

The Spigot and Overflow products were separated by decantation and then dried in a steam-heated air-circulating oven at 100°C. for several hours until constant weight. To judge sizing efficiency, sieve analysis were made both on the Spigot and on the Overflow fractions. Results have been tabulated in Table 5, page 40.

Table 5. Sieve Analyses of Feed and Products for Run 2

Mesh No.	Feed(*)		Overflow		Spigot	
	Indi- vidual Retained % by weight	Cumu- lative Finer	Indi- vidual Retained % by weight	Cumu- lative Finer	Indi- vidual Retained % by weight	Cumu- lative Finer
35	21.2	78.8	1.0	99.0	26.9	73.1
35/48	18.1	60.7	2.4	96.6	28.9	44.2
48/65	13.5	47.2	3.4	93.2	21.2	23.0
65/100	12.2	35.0	7.8	85.4	15.8	7.2
100/150	6.0	29.0	16.2	69.2	4.2	3.0
-150	29.0	--	69.2	--	3.0	--
Totals	100.0	--	100.0	--	100.0	--

(*) This column was computed from Table 1, page 13.
 Values for the 80-mesh fraction reported hereon were
 always found by graphical interpolation.

In order to compute the efficiency of separation, the phosphoric acid content was determined in all fractions. Results obtained are reported in Table 6 and Table 7, below.

Table 6. Grades in Overflow

Mesh	Grade Individual		Grade Cumulative Finer		Individual Distribution of Grade
	% P ₂ O ₅	% B.P.L.	% P ₂ O ₅	% B.P.L.	% of Total
None			21.4	46.8	
+48	7.6	16.6	21.9	47.9	1.2
48/65	13.1	28.6	22.2	48.5	2.1
65/100	14.1	30.8	22.9	50.0	5.1
100/150	20.9	45.7	23.4	51.2	15.9
-150	23.4	51.2	--	--	75.7
					<u>100.0</u>

Table 7. Grades in Spigot

Mesh	Grade Individual		Grade Cumulative Finer		Individual Distribution of Grade
	% P ₂ O ₅	% B.P.L.	% P ₂ O ₅	% B.P.L.	% of Total
None			3.3	7.2	
+35	1.7	3.7	4.0	8.7	13.6
35/48	2.1	4.6	5.2	11.4	18.1
48/65	3.0	6.6	7.2	15.7	19.0
65/100	4.4	9.6	13.3	29.1	20.7
100/150	9.3	20.3	19.0	41.5	11.6
-150	19.0	41.5	--	--	17.0
					<u>100.0</u>

In order to examine the distribution of the rest of the main components, determinations of Insoluble and Calcium Carbonate were also made in all fractions. A tabulation of the results appears in Tables No. 8 and No. 9, below.

Table 8. Proximate Composition of Overflow in Run No. 2

Mesh No.	B.P.L. %	Calcium	Insoluble %	Undetermined %	Total %
		Carbonate %			
+48	16.6	34.3	28.5	20.6	100.0
48/65	28.6	5.2	23.6	42.6	100.0
65/100	30.8	None	22.8	46.4	100.0
100/150	45.7	None	8.8	45.5	100.0
-150	51.2	None	3.1	45.7	100.0

Table 9. Proximate Composition of Spigot in Run No. 2

Mesh No.	B.P.L. %	Calcium	Insoluble %	Undetermined %	Total %
		Carbonate %			
+35	3.7	64.6	28.6	3.1	100.0
35/48	4.6	42.0	53.0	0.4	100.0
48/65	6.5	23.4	65.1	5.0	100.0
65/100	9.6	15.9	69.6	4.9	100.0
100/150	20.3	11.8	58.0	9.9	100.0
-150	41.5	None	20.6	37.9	100.0

When considering the Overflow product from the Specifications standpoint, it is seen from Table 5, page 40, that 90.0% goes through 80-mesh. Specifications require a minimum of 80.0%. As -28 mesh ore was used in the experiment, it is obvious that there are no particles larger than 3.0 mm. As to grade, Table 6, page 41, reports a figure of 21.4% P_2O_5 (46.8% B.P.L.). Specifications require at least 18.0% P_2O_5 . References to the Nitrogen, Potash, and Organic Matter contents are made for all of the experiments in a subsequent section.

Throughout this writing, efficiency of separation will be defined as the ratio between the amount of phosphoric acid recovered in the fines and the amount of phosphoric acid put in as feed. Two efficiencies of separation will be distinguished: Operational and Overall. Operational Efficiency will be referred to as based upon the material actually used in the operation concerned. It usually was a -28 or a -20 mesh ore; i.e., the rock was subjected to a preliminary sieve-sorting intended to eventually eliminate large stones or bones. Operational Efficiency will always be computed from experimental data of tests actually performed. On the other hand, Overall Efficiency of Separation will refer to the mine-run ore just as it was received. Overall Efficiency will be calculated from the Operational Efficiency on the basis of mesh fractions and grades previously eliminated. For experiments made on the

whole material, Operational and Overall Efficiencies will be the same.

Operational Efficiency of Separation for Run No. 2:

Data:

Weight of Material Fed, in kgs.	3.00
Grade of Material Fed, in % P_2O_5	10.1
Weight of Fines, in kgs.	1.32
Grade of Fines, in % P_2O_5	21.4

$$\text{Operational Efficiency} = \frac{(1.32)(0.214)}{(3.00)(0.101)} \times 100 = 93.2\%$$

(Operational Efficiency corrected for a Soluble Salts content of 4.3% is 97.4%.)

Overall Efficiency for Run No. 2:

Data:

Percent of + 28 mesh material in the original ore	43.2
Grade of whole original material, % P_2O_5	6.8
Weight of +28 material to be added, in kgs:	

$$\frac{(3.0)(43.2)}{100 - 43.2} = 2.50$$

Overall

$$\text{Efficiency} = \frac{(1.32)(0.214)}{(3.00)(0.101)} + \frac{(2.28)(0.025)}{(3.00)(0.101)} = 78.5\%$$

(Overall Efficiency corrected for Salts effect is 82.0%.)

After writing a "-80 mesh" material-balance, it was found that the weight of -80 mesh fines of overflow and spigots altogether, is greater than the -80 mesh fines in the feed by 14.8%, based on the latter. This increase of fines must be the effect of the thorough stirring with water in the mixer and sizer. This abnormality does not affect the above computations, since they are not based

upon percentage of fines, but upon actual weight of fines.

A P_2O_5 balance was also computed:

P_2O_5 In:

$$(3.00)(0.101) = 0.303$$

0.303 kgs. P_2O_5

P_2O_5 Out:

a) Overflow $(1.32)(0.214) = 0.283$

b) Spigot $(1.52)(0.033) = \underline{0.050}$ 0.333 kgs. P_2O_5

Gain 0.030 kgs. P_2O_5

= 10.0% of the feed

This excess probably has the effect of making the value of efficiencies higher than they actually were. It must have resulted from experimental error. An hypothetical analytical determination of P_2O_5 in the products lower by 0.3, which is admissible within practical limits, would reduce the excess to 6%.

Upon comparing the weight of feed (3.00 kgs.) with the weight of output (1.32 plus 1.52 kgs.) a loss of 0.16 kgs. of material, 5.3% of the feed, is apparent. This must be expected, due to the presence of cold-water-soluble salts in the feed, salts which were not recovered in the output.

Recovery Efficiencies of this Run will be compared with those for Runs 3 and 4 at the end of this section.

Run No. 3:

Experimental Data:

Size of Ore	-28 mesh	
Pulp Thickness	Ore:Water ratio 1:4	
Ore Fed in kgs.		3.00
Hydraulic Water, rate of flow in lts/min		3.6
Length of Run:	Total of 17 minutes for the whole run	
Fines Recovered (dry), in kgs.		1.25
Spigot Discharged (dry), in kgs.		1.63

Table 10. Sieve Analyses of Feed and Products, Run 3

Mesh No.	Feed		Overflow		Spigot	
	Indi- vidual Retained % by weight	Cumu- lative Finer	Indi- vidual Retained % by weight	Cumu- lative Finer	Indi- vidual Retained % by weight	Cumu- lative Finer
35	21.2	78.8	3.1	96.9	24.7	75.3
35/48	18.1	60.7	2.3	94.6	28.8	46.5
48/65	13.5	47.2	5.8	88.8	21.7	24.8
65/100	12.2	35.0	14.7	74.1	17.4	7.4
100/150	6.0	29.0	17.5	56.6	4.7	2.7
-150	29.0	--	56.6	--	2.7	--
Totals	100.0	--	100.0	--	100.0	--
-80		41.5		83.0		15.7

Table 11. Proximate Composition of Overflow and Spigot, Run 3

	P ₂ O ₅	B.P.L.	Calcium Carbonate	In-soluble	Un-determined	Total
	%	%	%	%	%	%
Overflow	22.0	48.1	None	5.2	46.7	100.0
Spigot	3.4	7.4	31.9	52.6	8.1	100.0

From the viewpoint of specifications, it is evident that the fines of the Overflow satisfy the requirements, since the -80 mesh fraction is greater than 80.0%, and since the grade is higher than 18.0% P₂O₅.

Operational Efficiency of Separation for Run No. 3:

Data:

Weight of Material Fed, in kgs.	3.0
Grade of Material Fed, in % P ₂ O ₅	10.1
Weight of Fines, in kgs.	1.25
Grade of Fines, in % P ₂ O ₅	22.0

$$\text{Operational Efficiency} = \frac{(1.25)(0.22)}{(3.00)(0.101)} \times 100 = 90.8\%$$

(Operational Efficiency corrected for Salts effect is 94.9%.)

Overall Efficiency for Run No. 3:

Data:

Weight of 28 mesh material to be added, in kgs.	2.28
Grade of the 28 material, in % P ₂ O ₅	2.50

$$\text{Overall Efficiency} = \frac{(1.25)(0.22)}{(3.00)(0.101)} \quad (2.28)(0.025) = 76.4\%$$

(Overall Efficiency corrected for Salts effect is 80.3%.)

The P_2O_5 balance also showed an output larger than the input, this time by 8.9%.

A loss of 0.12 kgs. of water-soluble materials took place in this run; i.e., 4.0% of the feed.

Run No. 4:

Experimental Data:

Size of Ore	Unscreened stock	
Pulp Thickness	Ore:Water ratio	1.4
Ore Fed, in kgs.		3.00
Hydraulic Water, rate of flow, lts/min		4.7
Length of time, in minutes		16
Fines Recovered (dry), in kgs.		0.42
Spigot Discharged (dry), in kgs.		2.36

Table 12. Sieve Analyses of Feed and Products for Run 4

Mesh No.	Feed		Overflow		Spigot	
	Indi-vidual Retained % by weight	Cumu-lative Finer	Indi-vidual Retained % by weight	Cumu-lative Finer	Indi-vidual Retained % by weight	Cumu-lative Finer
10	3.4	96.6			3.6	96.4
10/14	4.6	92.0			6.7	89.7
14/20	15.3	76.7			20.0	69.7
20/28	19.9	56.8			26.6	43.1
28/35	12.1	44.7	3.6	96.4	17.2	25.9
35/48	10.3	34.4	2.0	94.4	12.0	13.9
48/65	7.7	26.7	3.6	90.8	8.2	5.7
65/100	6.9	19.8	5.3	85.5	4.6	1.1
100/150	3.4	16.4	6.9	78.6	1.0	0.1
-150	16.4	--	78.6	--	0.1	--
Totals	100.0	--	100.0	--	100.0	--
-80		24.0		89.5		3.3

Table 13. Proximate Composition of Overflow and Spigot, Run 4

	P ₂ O ₅ %	B.P.L. %	Calcium Carbonate %	In- soluble %	Un- determined %	Total %
Overflow	22.0	48.1	None	6.9	45.0	100.0
Spigot	3.4	7.4	51.6	36.7	4.3	100.0

This overflow, with 89.5% passing through 80-mesh, and having a grade of 22.0% P₂O₅ (48.1% B.P.L.) meets the established specifications.

(Operational Efficiency of Separation for Run No. 4:)

Data:

Weight of Material Fed, in kgs.	3.0
Grade of Material Fed, in % P ₂ O ₅	6.8
Weight of Fines, in kgs.	0.42
Grade of Fines, in % P ₂ O ₅	22.0

$$\text{Operational Efficiency} = \frac{(0.42)(0.22)}{(3.0)(0.068)} = 45.3\%$$

(Operational Efficiency corrected for Salts effect is 47.3%.)

Overall Efficiency for Run No. 4: Same as Operational.

Overall Efficiency = 45.3%

(Overall Efficiency corrected for Salts effect is 47.3%)

This time the P₂O₅ balance indicated a deficit of 15.4% based upon the feed.

A combined weight of 2.78 kgs. for the products represents a loss of 0.22 kgs. This value is too high when the salts content in the raw material is considered (4.3%). The loss of fines is responsible for the deficit, and, of

course, for the low value of the recovery efficiency.

Before summarizing results for the three runs, the question of the proximate composition of the overflow should be made clear. In general, it can be said that it is very much the same as the composition of the -150 mesh fraction in the original ore. The contents of B.P.L. and Insoluble need not be discussed. As to the "undetermined", amounting to almost half of the composition, it is made up of organic matter, water retained (likely by absorption and/or crystallization) and oxides of iron and other metals.

Of the figures obtained from Runs No. 2, No. 3, and No. 4, the deciding ones are now reported in the form of Table 14.

Table 14. Results from Hydraulic Classification

Run No.	P ₂ O ₅ Balance		Efficiencies of Separation		Overflow		
	Deficit %	Super-avit %	Operational %	Overall %	-80 mesh %	P ₂ O ₅ %	B.P.L. %
2		10.0	93.2	82.0	90.0	21.4	46.8
3		8.9	90.8	80.3	83.0	22.0	48.1
4	15.4		45.3	47.3	89.5	22.0	48.1

In the light of these results, it must be said that hydraulic classification of the ore is possible. Furthermore, a sharp cut can be achieved. In the three experiments, products which meet the specification requirements have been obtained. As to corrected overall efficiency, it is thought that about 80% could be reached, provided there were no irregularities in the P₂O₅ balance, the limitations of which should be kept in mind. It is believed that commercial-scale equipment would afford higher efficiencies of separation for two reasons, chiefly: 1) Operation is steadier and errors minimized; and 2) Separation is performed gradually, in stages, according to the number of spigots, or sorting columns. These are usually from 4 to 6 for each sizer. The laboratory unit used in this experiment has only one spigot. The results for Run No. 4 are significant for an unscreened feed; the recovery efficiencies are far below those obtained when material of finer fractions was used.

Wet Screening

Statement: The known fact that some materials lend themselves to a better separation when suspended in a liquid fluid suggested the wet screening as one possible manner of industrially processing this ore. On the other hand, it is also known that some materials cannot be efficiently handled in such condition. Some of the factors involved are organic matter, shapes of grains, distribution of sizes, openings of the sieve, rate of feed, type of motion, slope.

Principle: To the ordinary screening, stationary or shaking the influence of a moving liquid, commonly water, has been added. The action of the fluid is twofold:

a) Grains reach the sieve in the form of a suspension; b) The stream of water helps washing the "fines" through the screen, and the "coarse" off of the screen.

Purpose: The aim was to secure results of wet-screen-sizing on different sieves.

Equipment and Procedure: One Set of Tyler Standard Screen-scale Sieves Four apertures were tested, namely: 35, 48, 65, and 100 mesh. The material tested was run of mine, a mixture 1:1 of Sacks No. 6 and No. 28. A loss by heating at 105°C. was made on the feed. Value obtained was 4% by weight of original material. A fifty-grams sample of the ore was thoroughly wetted with tap water to a thin paste, then transferred to the corresponding

screen, which was already partially immersed in water. The depth to which the screen remained submerged was such that the ore was always below the water level, and the screen top-rim always above the liquid surface. Four liters of water were held in a suitable size pan. During ten consecutive minutes the sieve was given a shaking movement. Two motions were imparted alternately, up-and-down, and horizontal jerk. Oscillations were approximately one inch, and two inches, respectively. Water was separated by filtration from the fines, and fines as well as oversize were dried at 105°C. constant weight.

Experimental Data:

	Sieves Used			
	35	48	65	100
	<u>Mesh</u>	<u>Mesh</u>	<u>Mesh</u>	<u>Mesh</u>
Weight of Oversize, in grs.	25.4	28.9	33.2	36.4
Weight of Undersize, in grs.	19.7	16.5	12.1	8.8
Weight of Feed, in grs.	50.0	50.0	50.0	50.0

Results: Since the oversize and undersize were dried by heating, the loss by heating should be added to the weights reported; also, loss due to salts dissolved.

Rearranging the Results:

	Sieves Used			
	35	48	65	100
	<u>Mesh</u>	<u>Mesh</u>	<u>Mesh</u>	<u>Mesh</u>
Weight of Oversize, in grs.	25.4	28.9	33.2	36.4
Weight of Undersize, in grs.	19.7	16.5	12.1	8.8
Loss by heating, in grs.	2.0	2.0	2.0	2.0
Loss by salts dissolved, in grs.	2.1	2.1	2.1	2.1
	<u>49.2</u>	<u>49.5</u>	<u>49.4</u>	<u>49.3</u>

Determinations of phosphoric acid were made in the
undersize.

	Undersize			
	35	48	65	100
	<u>Mesh</u>	<u>Mesh</u>	<u>Mesh</u>	<u>Mesh</u>
Phosphoric Acid, % P_2O_5	13.3	16.0	19.1	22.3
B.P.L., %	29.1	35.0	41.8	49.0

From Table 3, the grade of the original material is known to be 6.8% P_2O_5 (14.85% B.P.L.).

Efficiencies of Separation were calculated as the ratio of amount of P_2O_5 , in grams, contained in the Undersize, to P_2O_5 contained in the original fifty-grams sample.

Screen Mesh	35	48	65	100
Separation	77.1	77.6	68.0	57.7

Although the undersize of a cut made at 65-mesh would pass the grade requirement, the percentage retained by 80-mesh would be the limit, about 20%, of what is allowed. This difficulty could be overcome by simply grinding the coarse particles of this undersize until the specification for size was met. Still better recovery could be afforded

if the cut is made at 48-mesh. In this case, the specifications for both grade and size are met. This could be achieved by two operations: Grinding and calcination. The upgrading obtained by heating would be at the expense of the organic matter, which would be lost. From the standpoint of the equipment, a wet screening in the larger sizes is preferable. In order to diminish abrasion and to increase capacity of screens, wet screening should be done in at least two stages: The first at 28-mesh, which would retain about 42% of the feed weight, thus leaving approximately 22% to be retained by the 48-mesh. Further considerations will be discussed in the latter part of this writing.

Tabling

The principles under which the jerking tables work are so well known that they are not described herein. However, let it be known that in the case of this ore, where the valuable material is in the fines, the "tailing side" is not expected to discharge tailings but the concentrate. The "concentrate side" should deliver the valueless coarser fractions. In spite of this difference, and for the sake of preventing confusion, the orthodox convention of naming the four sides will be maintained. The equipment used was a Pilot Table with an attached feeding mechanism. This table, the picture of which appears in Fig. 15, page 57, may be described as:

Pilot Table OVERSTROM UNIVERSAL.
Dimensions: 2 feet by 5 feet.
Straub Mfg. Co., Oakland, Calif.
Actuated by 1/2 h.p. General Electric motor.

The experiment herein described will be called Run No. 7. The feed was the previously described "run of mine" mixture 1:1 of Sacks No. 6 and No. 28. The material was fed as a 1:4 ore to water ratio-pulp, and cleaning water at measured rates was added from the feed side.

The first trial was made with small tension on the spring, which meant the lowest number of strokes per minute. A steep pitch of the table was used. Washing water was added at the rate of 15 liters per minute. Upon visual checking of the performance it was seen that no

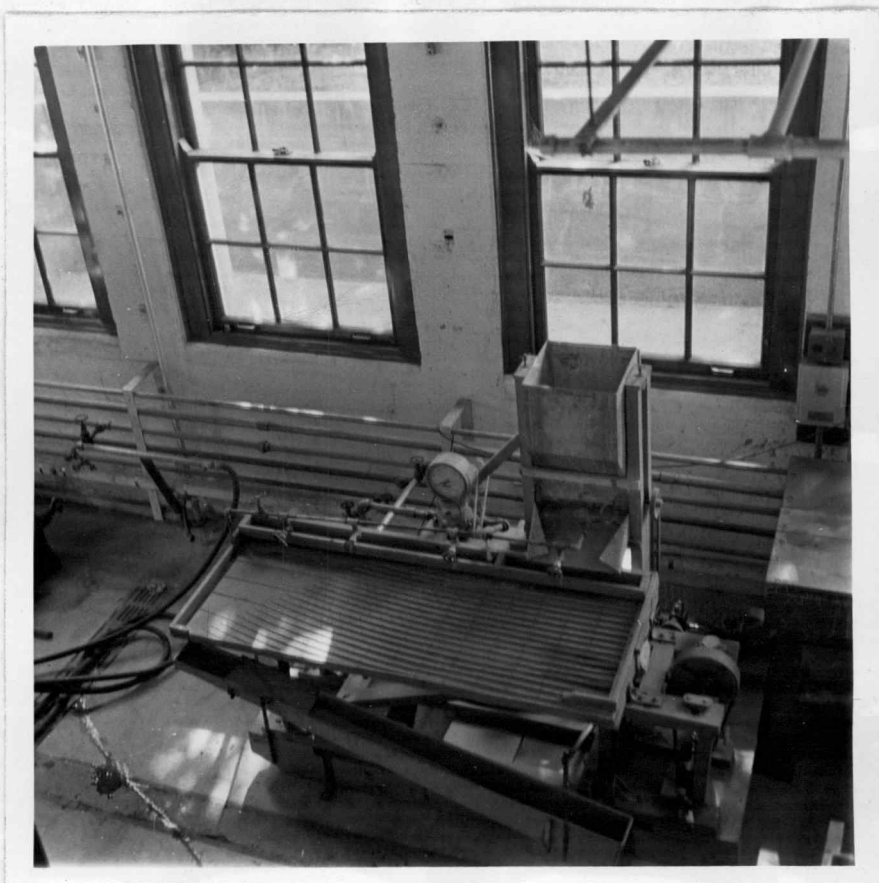


Fig. 15 Pilot Table "Overstrom Universal"

material went to the concentrate end. Practically all of the material remained mixed and went to the tailing side. Neither an increase nor decrease of the rate of cleaning water altered the situation. Upon gradually diminishing the pitch of the table, a very small change was noticed. When the table was in its flattest position, a slightly better separation could be observed. The fines, still mixed with the coarser particles, went to the tailing side, while there was a small amount of coarse discharged from the concentrate end.

The second attempt was made with the strongest tension on the spring. In this way the largest number of strokes per minute was obtained. With the steepest slope, the material did not go to the concentrate end but accumulated itself in the middlings section and in the back end of the table, finally overflowing with the fines. A flatter table produced no better results. The change in rate of washing water throughout this test did not provide any perceptible improvement.

Several intermediate degrees of tension on the table spring were tried at different slopes and at varied rates of cleaning water. In no case was even a fair separation attained and it was quite evident that tabling would not provide an effective means of concentration.

Spiraling

Statement: Because the spiral, besides permitting a gravity separation in a liquid medium, also affords a size classification, it was suggested that its use might permit the separation of the finer particles from the coarse ones.

Theory: According to Gleeson,⁽⁵⁾ separation in the Spiral is the result of two forces acting on the particles suspended in the stream: centrifugal and gravitational. The centrifugal force is tangential to the channel, directly proportional to the square of the velocity of flow and inversely proportional to the radius at which the gravity center of the particle is located. The gravitational force acts downwards. In the case of light particles, namely water and fine, low absolute density grains, the resultant of the forces acts across the channel. As for the heavy elements, the resultant of the two forces pulls down.

"There is, consequently, besides the flow of water down the length of the spiral, a flow of decreasing velocity downward and across the channel to the top, progressively increasing in velocity until friction again enters."

Fig. 16, page 60, is an schematic representation of this crosswise cycle. Fig. 17, page 61, shows a typical, actual distribution of the fractions across the channel. Figs. 18 and 19, pages 62 and 63, respectively, are self-explanatory.

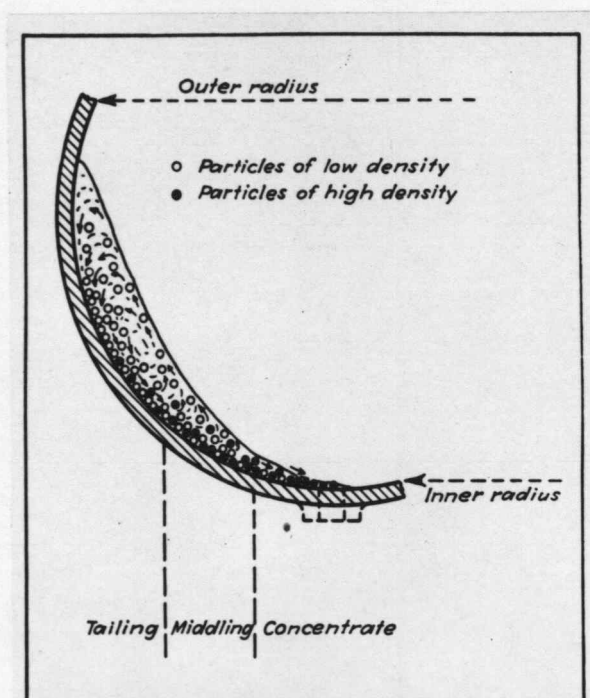
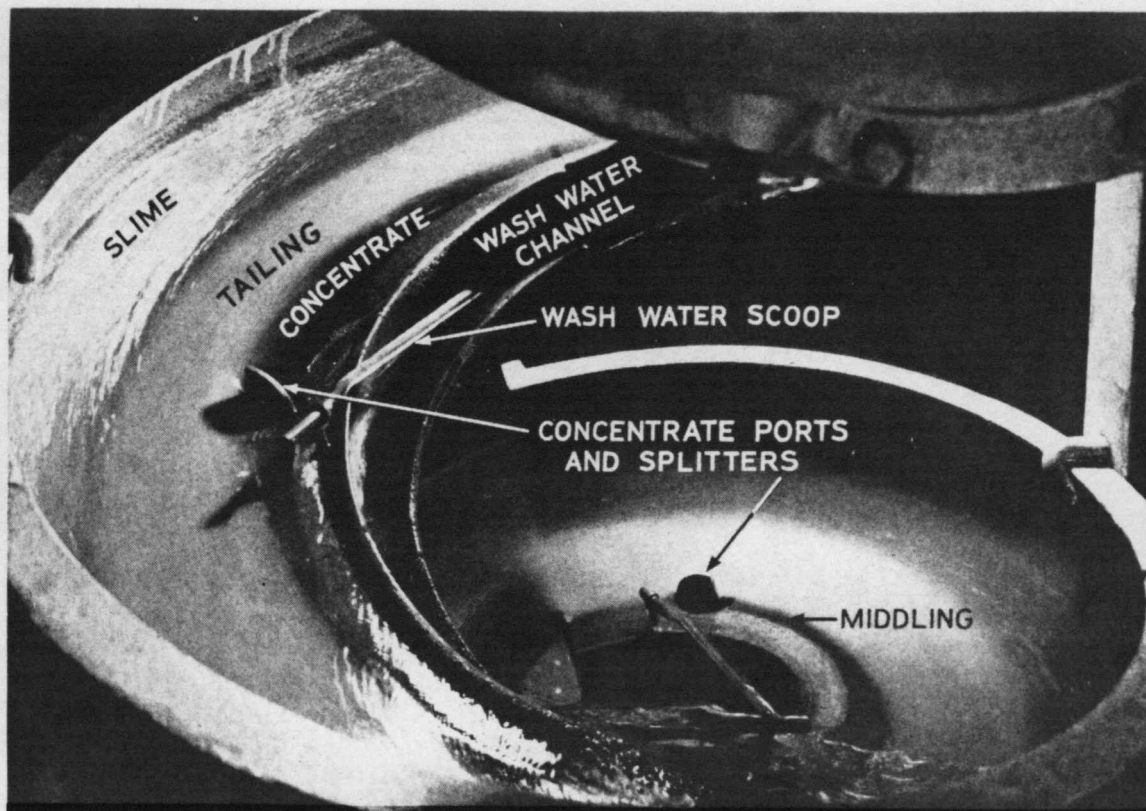


Fig. 16. . Showing how water flow along spiral bottom brings heavy mineral to concentrate outlets; reverse flow above carries lighter mineral up.



CONCENTRATING ACTION of spiral. Note wide black band of concentrate entering upper outlet, which is set for a wide cut; also narrow black band of middling entering lower outlet set for thin cut.

March, 1945—Engineering and Mining Journal

Fig. 17

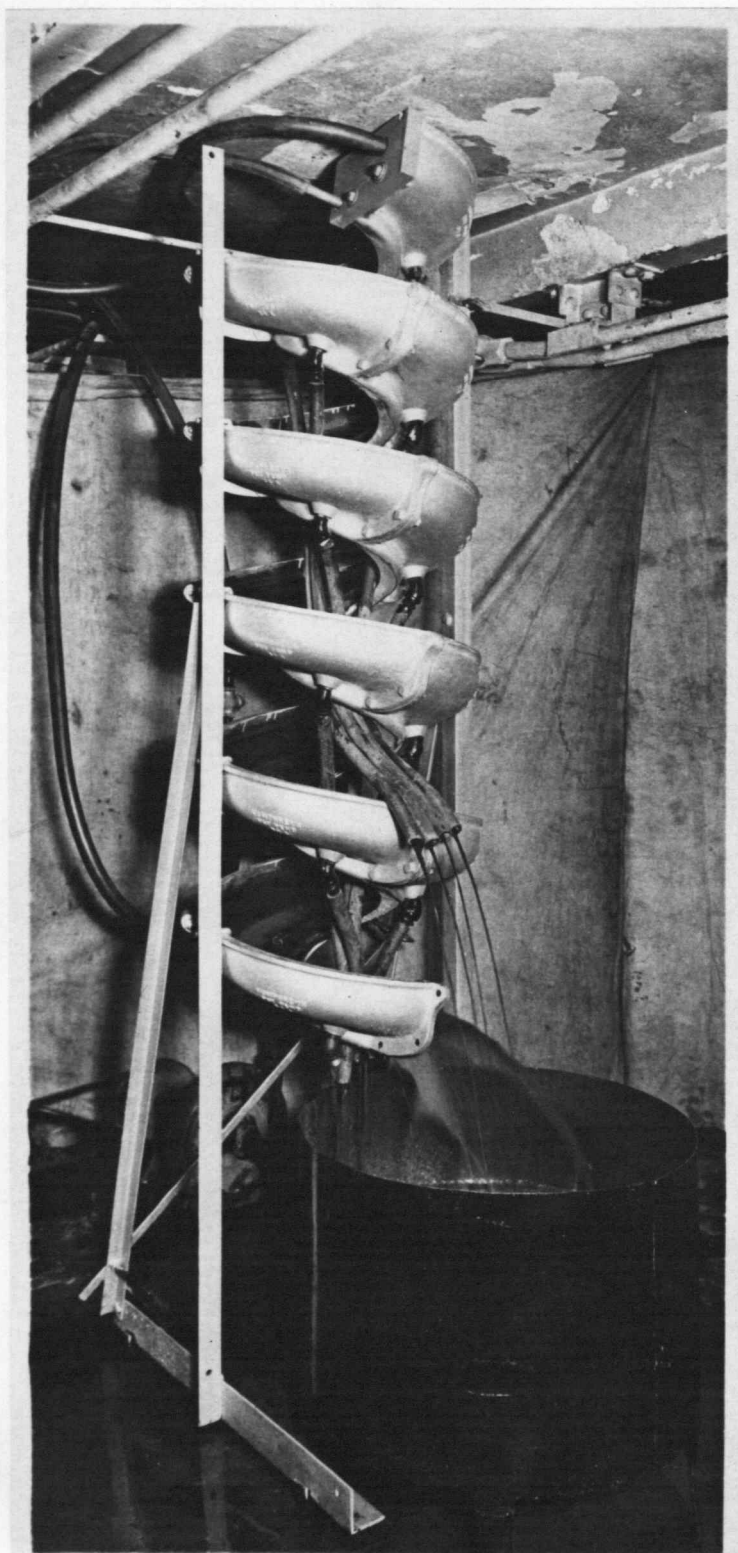


Fig. 18

(over)

No. 19683.

Humphreys Spiral Concentrator - Model 24-A, 5 turn,
in single frame. Laboratory test unit of commercial
size.

June 15, 1944.

The Humphreys Investment Company
910 First National Bank Building
Denver 2, Colorado.

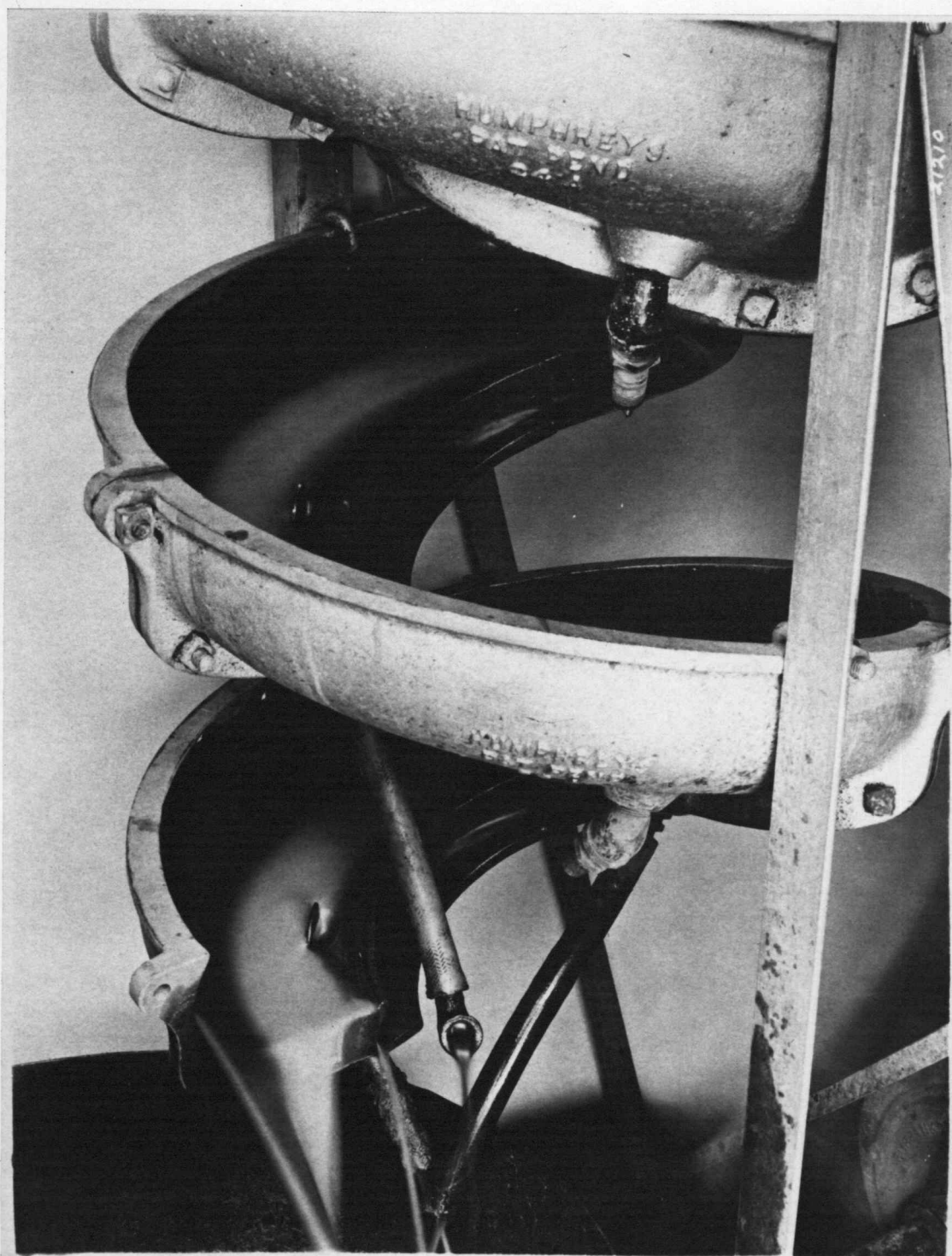


Fig. 19

(over)

No. 21210

Humphreys Spiral Concentrator - Model 24-A, laboratory test unit of commercial size, close-up view.

Two of the adjustable splitters which set into the concentrate outlets are shown. These can be turned to permit a wide or narrow cut of concentrate, to go into the concentrate or middling outlets. Concentrate can be seen in the upper part of the spiral as the dark path toward the inside of the concentrator, while tailing is the lighter colored material running further up into the faster moving stream of water above the concentrate outlet. Wash water is transferred from the small washwater channel on the inside of the spiral by the rod which picks it up from that channel and diverts it to the inside of the main channel. Concentrate is drawn off from the upper outlets, while middling is drawn off from the lower outlets and tailing is discharged at the end of the spiral.

November 25, 1944.

The Humphreys Investment Company
910 First National Bank Building
Denver 2, Colorado.

Equipment and Procedure: A four-turns Humphreys Spiral was used. A centrifugal pump, pipings and tank connected to the Spiral, completed a closed circuit, which is shown in picture 18.

The same sample used in the foregoing experiments was used in this test, except that it was screened through 10-mesh. Tap water was used as the fluid. The splitters were ground to fit. Four runs, designated as No. 8, 9, 10, and 11 were made. Material charged was 3, 4, 5, and 6 kilos, respectively. Pump was maintained at full speed. Samples of both Spigot and Channel products were taken simultaneously once equilibrium in the flowing stream was reached. The samples were decanted, dried, weighted, and analyzed. Results are given in Table 15, page 65.

Considering the behavior of the ore in the spiral from a preliminary, qualitative standpoint, it must be observed in Table 16 that the channel product in all runs contains a high percentage of the 20 mesh grains, i.e., the large, flat particles of calcium carbonate (marine shells). This fact alone makes the channel product of all runs unsatisfactory from the standpoint of size requirements. However, from 20-mesh down, a sudden drop of the quantities of coarse grains could be seen. These values remain consistently low as the sieve apertures decrease.

Referring now to the grades, it is realized from Table 15 that no Run gave a satisfactory product. Apparently the high proportion of 20 mesh particles is responsible for the low grade.

Considering the tests from the viewpoint of the pulp densities, Runs No. 9, 10, and 11, are consistent in that the thicker the pulp the more fines in the channel product. The grades are accordingly higher. This observation confirms the issue that spirals achieve better performance at full load. The problem of improving the channel product is accessible by either one of two ways:

- 1) Feeding the spiral with a -20 mesh charge;

- 2) Subjecting the channel product to further cleaning.

The second method appears to be the most simple and economic.

Hydraulic sizers, or even a 20-mesh screen, would perform the duty with a large capacity rate.

Computation of Efficiencies: Recoveries were calculated upon the basis of separation achieved. The latter was based on the ratio of weights of spigot and channel fractions obtained by sampling. Corrections were included for the salt effect and for the preliminary screening. Results appear in Table 17, below.

Table 17. Efficiencies in the Spiral

Run No.	Charge	$\frac{1}{\text{sp/ch} + 1}$	Grade in Channel	Efficiency of Separation	
	kilos		% P_2O_5	Operational	Overall
				%	%
8	3	0.0968	16.1	21.3	20.6
9	4	0.1704	11.3	26.3	25.4
10	5	0.152	13.6	28.8	27.8
11	6	0.148	15.5	31.3	30.2

The efficiencies of separation are strikingly low. Two immediate reasons are apparent for the low yields: 1) Too much weight of fines in the spigot product and, of course, in the tailings, and 2) The already existent relation between size and grade. A P_2O_5 determination in the -150 mesh fraction of the spigot in Run 11 indicated 18.6%. The possible means of improving the operation would be to

supplement it by retreating the spigot and channel fractions. An hydraulic classifier or even a sand washer would permit recovery of the fines contained in the spigot fraction. On the other hand, an hydraulic classifier or even a screen would eliminate the large flat particles contained in the channel product. From above considerations, it is advisable that further experimentation of the spiral combined with other hydraulic equipment be carried on. Due to the unsatisfactory preliminary results, experimentation with spiral separation was discontinued.

Flotation

Statement: The raw material was considered as a phosphate-silica-calcite ore. Hence, differential concentration by separating a concentrate relatively rich in phosphate and relatively poor in calcite-silica was attempted. The phosphate industry in the U.S.A. is successfully recovering phosphatic material by floating the rejects of the matrix washeries, mainly in Florida, and to a less extent in Tennessee. The problem differs from the Chilean ore in that the phosphatic values to be recovered are distributed in the intermediate sizes (-20/ 65), the finer fractions are undesirable slimes and the percentage of silica and calcite in the U.S. fluophosphates are very much lower.

Because the presence of calcium carbonate, only an alkaline pulp was considered to be feasible for a commercial operation.

Equipment and Procedure: The main pieces of equipment used were:

- 1) One 1000-gram Denver Lab. Flotation Machine, Sub-A, powered by a 1/6 h.p. electric motor, 1 phase, variable speed; Kimble Electric Co., Chicago, Ill.
- 2) One variable speed, electrically-driven mixer, for conditioning.
- 3) One Beckman pH Meter. Industrial Model. Direct Reading. National Technical Laboratories, Pasadena, Calif.
- 4) Micropipettes.

Same mixture as before was used as feed. Eleven tests were made, which will be referred to as Runs No. 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, and 22. In all of the runs but three, the -20 mesh fraction of the original ore was used. The pulp was always prepared and diluted with tap water. Reagents were always added in the conditioner, and the conditioning period was ten minutes. The feed was conditioned as a thick pulp for ten minutes and then transferred to the flotation machine and diluted. pH determinations were made in the pulp after conditioned, in the concentrate, and in the tailings. After flotation, concentrate and tailings were filtered; the solids dried and weighed. Specific conditions for each run will be reported when describing the separate experiments. Reagents used and their intended action are listed below:

Frothers:	Yarmor F Pine Oil
Collector:	Oleic Acid, Orinite, Emulsol x-1, Fuel Oil
Modifiers:	NaOH, Lime Sodium Silicate.

Run No. 12Conditions:

Feed Charge, in grams	1000
Grade of Charge, in % P_2O_5	10.1
Size of Feed	-28 mesh
Pulp Thickness, in % of solids, by wt.	
Conditioning	60
Flotation	25
Flotation period, in minutes	50
pH of pulp, brought with 2% NaOH aq. soln up to	8.0
Yarmor F Pine Oil	0.1 lb/ton
Oleic Acid	1.5 lb/ton
Fuel Oil	3.0 lb/ton

Results: There was so much froth formed that it kept flowing for almost one hour. Weight and grades of concentrate and tailings were:

pH of the tailings 7.7

	Weight (dry) grs.	Grade % by weight P_2O_5	B.P.L.
Concentrate	350	14.9	32.6
Tailings	<u>604</u>	5.3	11.6
Total	954		

Proximate composition was:

	<u>Concentrate</u>	<u>Tailings</u>
Insoluble in A.R.	7.2%	42.9%
Tricalcium Phosphate	32.6	11.6
Calcium Carbonate	6.4	9.1
Undetermined	53.8	36.4
Totals	<u>100.0%</u>	<u>100.0%</u>

Sizing analysis was:

	<u>Concentrate</u>	<u>Tailings</u>
+35 mesh	19.8%	0.0%
35/48	26.9	1.3
48/65	20.6	3.8
65/100	13.9	8.0
100/150	4.9	2.5
-150	13.9	84.4
Totals	<u>100.0%</u>	<u>100.0%</u>

A total output of 954 grams is consistent with the presence of 4.3% of water soluble material in the ore.

Operational Efficiency of Separation:

$$\frac{(350)(0.149)}{(1000)(0.101)} \times 100 = 51.6\%$$

Overall Efficiency of Separation:

Data: % by wt. of finer than 28-mesh, is 56.8

Grade of the overall stock, in
% P_2O_5 , is 6.8

$$\frac{350(0.149)}{(1000/0.568)(0.068)} = 47.0\%$$

Discussion: Recovery is low when compared with hydraulic separation. The concentrate does not pass the grade requirements; the size composition, however, is satisfactory. It is seen that both the siliceous gangue and the calcium carbonate were partially floated. It is also observed that

in spite of the long flotation time and the large amount of foam, an important amount of fines remained unfloated in the tailings.

Run No. 13

Conditions:

Feed Charge, in grams	1000
Grade of Charge, in % P_2O_5	8.1
Size of Feed	-20 mesh
Pulp Thickness, in % of solids, by wt.	
Conditioning	60
Flotation	25
Flotation Period, in minutes	13
Sodium Silicate (dry), in lbs/ton	0.15
Fuel Oil, in lbs/ton	3
pH of pulp (brought up with Sod. Silic.)	8.0

Results: Flotation was run as long as froth kept forming (13 minutes).

pH of the Concentrate	7.1
pH of the Tailings	7.8

	Weight (dry) grs.	Grade % by weight P_2O_5 B.P.L.	
Concentrate	120	20.2	44.1
Tailings	838	5.0	10.9
Total	958		

Proximate composition was:

	<u>Concentrate</u>	<u>Tailings</u>
Insoluble in A.R.	5.5%	33.9%
Tricalcium Phosphate	44.1	10.9
Calcium Carbonate	None	42.1
Undetermined	52.4	13.1
Totals	<u>100.0%</u>	<u>100.0%</u>

Sizing analysis was:

	<u>Tailings</u>
+ 28 mesh	29.2%
28/35	18.3
35/48	16.0
48/65	11.7
65/100	8.5
100/150	2.3
-150	14.0
Total	<u>100.0%</u>

Operational Efficiency of Separation: 29.9%

Overall Efficiency of Separation: 27.3%

Discussion: Sodium Silicate was a good depressant for the calcium carbonate, and was fair for the silica. Sizing analysis of the concentrate was not made because it was visibly evident that the degree of fineness met the specification requirements. In this run, a satisfactory grade was reached. On the other hand, the percentage of -150 fraction in the tailings was the same as in the previous tests; it contained 17.2% of P_2O_5 (37.6% B.P.L.) The absence of a collector was reflected in the very low yield of fines, which was approximately one third of the weight of concentrate produced in Run No. 12. This was the chief reason for such a poor efficiency of separation.

Run No. 14

The object of this test was to find the effect (under conditions of Run 13) of an increase in the amount of the depressant. Everything was the same except that the Sodium Silicate was added at the rate of 0.65 lbs/ton.

Results: The frothing period (flotation time) was only six minutes. The pH of concentrate and of tailings were 7.1 and 7.3, respectively.

	Weight (dry) grs.	Grade % by weight P ₂ O ₅	B.P.L.
Concentrate	115	20.5	44.8
Tailings	801	4.9	10.7

Proximate composition was:

	<u>Concentrate</u>	<u>Tailings</u>
Insoluble in A.R.	3.3%	35.9%
Tricalcium Phosphate	44.8	10.7
Calcium Carbonate	None	42.3
Undetermined	51.9	11.1
Totals	<u>100.0%</u>	<u>100.0%</u>

As to size distribution, the sieve test gave:

	<u>Tailings</u>
+ 28 mesh	33.5%
28/35	17.9
35/48	14.3
48/65	12.0
65/100	6.4
100/150	2.0
-150 mesh	13.9
Total	<u>100.0%</u>

Operational Efficiency of Separation: 29.4%

Overall Efficiency of Separation: 26.6%

Discussion: Efficiencies, although slightly lower, were practically the same as before. Grades were also the same. The silica was not further depressed. Sizing analysis indicated a small decrease in the percentage of the finer grains. Briefly, no practical appreciable change in efficiency or composition was obtained.

Run No. 15

This experiment was made in order to determine the effect that a twofold amount of reagents, compared with Run 14, would have upon the performance. Conditions were the same as in Run 14, except that the Sodium Silicate and the Fuel Oil were used at the rates of 1.3 and 6.0 lbs/ton, respectively. The pH of the pulp was 7.3.

Results: The flotation period was found to be longer and lasted 15 minutes. The pH in the liquid of the concentrate and of the tailings were 7.2 and 7.7, respectively.

	Weight (dry) grs.	Grade % by weight P ₂ O ₅	B.P.L.
Concentrate	149	20.2	44.1
Tailings	782	4.9	10.7

Proximate composition:

	<u>Concentrate</u>	<u>Tailings</u>
Insoluble in A.R.	3.1%	35.4%
Tricalcium Phosphate	44.1	10.7
Calcium Carbonate	None	41.8
Undetermined	52.8	12.1
Totals	<u>100.0%</u>	<u>100.0%</u>

Sizing analysis:

	<u>Tailings</u>
+28 mesh	29.4%
28/35	17.8
35/48	16.3
48/65	10.7
65/100	6.7
100/150	2.0
-150 mesh	17.1
Total	<u>100.0%</u>

Operational Efficiency of Separation:	37.6%
Overall Efficiency of Separation:	33.9%

Discussion: While the grade of the product remained unchanged, the amount of fines collected was larger, this being reflected in the increase in the efficiency of separation. The depression of the silica showed a small increase. The amount of fines left unfloated in the tailings was still high and should be reduced to promote higher efficiencies.

Run No. 16

This run was made at a higher pH in the pulp. The increase in alkalinity was obtained by the addition of Sodium Hydroxide. Unscreened "run of mine" was used as the test sample.

Conditions:

Feed Charge, in grams	1000
Size of Feed	Unscreened
Pulp Thickness, in solids %, by wt.	
Conditioning	60
Flotation	25
Flotation Time, in minutes	13
Sodium Silicate, in lbs/ton	1.3
Sodium Hydroxide	1.3
Fuel Oil	6.0
pH of the Pulp	8.6

Results:

pH in Concentrate	8.1
pH in Tailings	8.3

	Weight (dry) grs.	Grade % by weight P ₂ O ₅	B.P.L.
Concentrate	112	19.4	42.4
Tailings	825	4.6	10.0

Proximate composition:

	<u>Concentrate</u>	<u>Tailings</u>
Insoluble in A.R.	2.1%	35.9%
Tricalcium Phosphate	42.4	10.0
Calcium Carbonate	None	43.5
Undetermined	55.5	10.6
Totals	<u>100.0%</u>	<u>100.0%</u>

Sizing analysis:

	<u>Tailings</u>
+ 28 mesh	38.8%
28/35	14.7
35/48	13.2
48/65	9.3
65/100	6.6
100/150	2.7
-150 mesh	14.7
Total	<u>100.0%</u>

Operational Efficiency of Separation: 32.0%

Overall Efficiency of Separation: 32.0%

Discussion: No appreciable change could be observed except a small additional depression of the silica. The amount of unfloated fines in the tailings remained about the same as in previous runs.

Run No. 17

The possibility of substituting lime for sodium alkali was tried in this experiment.

Conditions:

Feed Charge, in grams	1000
Size of Feed	-20 mesh
Pulp Thickness, in solids %, by wt.	
Conditioning	60
Flotation	25
Flotation Time, in minutes	15
Sodium Silicate, in lbs/ton	0.65
Calcium Oxide (dry)	1.4
Oleic Acid	0.85
Fuel Oil	3.0
pH of the Pulp	7.5

Results:

pH of Tailings were: 7.6

	Weight (dry) grs.	Grade % by weight	
		P ₂ O ₅	B.P.L.
Concentrate	131	20.1	43.9
Tailings	779	4.5	9.8

Proximate composition:

	<u>Concentrate</u>	<u>Tailings</u>
Insoluble in A.R.	3.4%	37.6%
Tricalcium Phosphate	43.9	9.8
Calcium Carbonate	6.8	44.8
Undetermined	45.9	7.8
Totals	100.0%	100.0%

Sizing analysis:

	<u>Tailings</u>
+ 28 mesh	28.3%
28/35	19.1
35/48	16.0
48/65	11.8
65/100	9.2
100/150	3.4
-150 mesh	12.2
Total	<u>100.0%</u>

Operational Efficiency of Separation: 32.5%

Overall Efficiency of Separation: 29.7%

Discussion: The effect of the lime was primarily that of checking the depressing effect of the sodium silicate, at least as far as calcium carbonate was concerned. In spite of this fact, the grade of the fines was still satisfactory. A small increase in the amount of floatation of the fines was indicated.

Run No. 18

This test was made in order to try the "Orinite" as a collector and filmer.

Conditions:

Feed Charge, in grams	1000
Size of Feed	-20 mesh
Pulp Thickness, solids, wt. %	
Conditioning	60
Flotation	25
Flotation Time, minutes	10
Sodium Silicate, lbs/ton	0.65
Lime (dry), lb/ton	0.7
Orinite, lb/ton	6.6
pH of pulp	7.5

Results:

pH of Tailings

7.6

	Weight (dry) grs.	Grade % by weight P ₂ O ₅ B.P.L.	
Concentrate	144	20.5	44.8
Tailings	775	4.0	8.7

Proximate composition:

	<u>Concentrate</u>	<u>Tailings</u>
Insoluble in A.R.	3.2%	38.8%
Tricalcium Phosphate	44.8	8.7
Calcium Carbonate	5.2	46.4
Undetermined	46.8	6.1
Totals	<u>100.0%</u>	<u>100.0%</u>

Sizing analysis:

	<u>Tailings</u>
28 mesh	28.0%
28/35	19.0
35/48	15.2
48/65	11.3
65/100	8.2
100/150	3.5
-150 mesh	14.8
Total	<u>100.0%</u>

Operational Efficiency of Separation: 36.5%

Overall Efficiency of Separation: 33.6%

Discussion: The slightly higher yield of fines and grade were responsible for the increase in efficiencies. The phosphoric content of the tailings was the lowest obtained in any floatation test.

Run No. 19

The feature of this test was the introduction of an emulsifying agent, namely Emulsol X-1. Oleic Acid was used as a collector, and the filmer was purposely neglected.

Conditions:

Feed Charge, in grams	1000
Size of Feed	-20 mesh
Pulp Thickness, solids, wt. %	
Conditioning	60
Flotation	25
Flotation Time, minutes	12
Sodium Silicate, lbs/ton	0.65
Sodium Hydroxide, lb/ton	0.04
Oleic Acid, lb/ton	1.3
Emulsol X-1, lb/ton	0.66

Results:

pH of Tailings:	7.5
-----------------	-----

	Weight (dry) grs.	Grade % by weight P ₂ O ₅	B.P.L.
Concentrate	160	19.9	43.5
Tailings	760	4.6	10.0



Fig. 20 Flotation Tailings from Run 19

Proximate composition:

	<u>Concentrate</u>	<u>Tailings</u>
Insoluble in A.R.	4.2%	36.7%
Tricalcium Phosphate	43.5	10.0
Calcium Carbonate	1.6	44.6
Undetermined	50.7	8.7
Totals	<u>100.0%</u>	<u>100.0%</u>

Sizing analysis:

	<u>Tailings</u>
+ 28 mesh	28.3%
28/35	19.6
35/48	15.7
48/65	11.4
65/100	7.8
100/150	2.7
-150 mesh	14.5
Total	<u>100.0%</u>

Operational Efficiency of Separation: 39.6%

Overall Efficiency of Separation: 35.9%

Discussion: Apparently the emulsifier acted as a promoter. The increase in efficiency was due to a greater collection. The lowering of the calcium carbonate content in the concentrate confirmed the checking action of the lime. The amount of unfloated material was practically unchanged.

Run No. 20

This was a low-capacity test performed at about the same conditions as Run No. 19. The main differences were the reduction of test sample from one kilo down to one half, and the increase in alkalinity.

Conditions:

Feed Charge, in grams	500
Size of Feed	-20 mesh
Pulp Thickness, solids, wt. %	
Conditioning	60
Flotation	14
Flotation Time, in minutes	13
Sodium Silicate, in lbs/ton	0.65
Sodium Hydroxide, lbs/ton	0.48
Oleic Acid, lbs/ton	1.3
Emulsol X-1	0.5
pH of the Pulp	8.5

Results:

pH of Concentrate	7.8
pH of Tailings	7.9

A tough foam was obtained.

	Weight (dry) grs.	Grade % by weight P ₂ O ₅	B.P.L.
Concentrate	89	22.4	48.9
Tailings	372	6.3	13.8

Proximate composition:

	<u>Concentrate</u>	<u>Tailings</u>
Insoluble in A.R.	5.3%	35.9%
Tricalcium Phosphate	48.9	13.8
Calcium Carbonate	None	38.6
Undetermined	45.8	11.7
Totals	<u>100.0%</u>	<u>100.0%</u>

Sizing analysis:

	<u>Tailings</u>
+28 mesh	30.8%
28/35	17.5
35/48	13.6
48/65	11.3
65/100	7.8
100/150	2.7
-150	16.3
Total	<u>100.0%</u>

Operational Efficiency of Separation: 49.2%

Overall Efficiency of Separation: 45.0%

Discussion: The efficiency was larger because of a better recovery of fines and of a higher grade in the concentrate. However, the amount of small-size particles left in the tailings was still appreciable. The improvement in efficiency of separation was not remarkable. From this and the foregoing floatation tests it could be concluded that that no commercial separation is feasible in one stage. The next run is a combined "flotation-separation-flotation" experiment.

Run No. 21Conditions:

Feed Charge, in grams	1000
Size of Feed	-20
Pulp Thickness, solids, wt. %	
Conditioning	60
Flotation	25
Flotation Time, minutes	10
Sodium Silicate, lbs/ton	0.65
Sodium Hydroxide, lbs/ton	0.6
Orinite, lbs/ton	6.6
Yarmor F Pine Oil, lbs/ton	0.13
pH of the Pulp	8.5

The supernatant suspension of unfloated small particles in the tailings was separated from the coarser by decantation. They were later used as feed charge in Run No. 22.

Results:

pH of Concentrate	7.4
-------------------	-----

	Weight (dry) grs.	Grade % by weight P ₂ O ₅ B.P.L.	
Concentrate (No. 1)	136	22.5	49.2
Tailings deprived of fines (No. 1)	640	2.3	5.0

Proximate composition:

	<u>Concentrate</u>	<u>Tailings(*)</u>
Insoluble in A.R.	5.0%	42.0%
Tricalcium Phosphate	49.2	5.0
Calcium Carbonate	None	45.5
Undetermined	45.8	7.4
Totals	<u>100.0%</u>	<u>100.0%</u>

Sizing analysis:

	<u>Tailings(*)</u>
+28 mesh	31.4%
28/35	21.7
35/48	18.2
48/65	13.6
65/100	9.3
100/150	3.1
-150 mesh	2.7
Total	<u>100.0%</u>

Operational Efficiency of Separation: 37.6%

Overall Efficiency of Separation: 34.5%

Discussion: See Run No. 22.

(*) Deprived of the finer particles.

Run No. 22

The feed charge was the unfloated phosphatic fraction from Run No. 21. It was reconditioned as follows:

Feed Charge, dry solids, in grams	15.5
Grade of Feed, in % P_2O_5	17.5
Pulp Thickness, solids, wt. %	
Conditioning	35
Flotation	5
Flotation Time, minutes	13
Sodium Silicate, lbs/ton	0.65
Sodium Hydroxide, lb/ton	0.6
Orinite, lb/ton	6.6
Yarmor F Pine Oil, lb/ton	0.15
pH of the Pulp	8.8

Results:

pH of Concentrate (No. 2)	7.7
pH of Tailings	9.1

	Weight (dry) grs.	Grade % by weight P_2O_5	B.P.L.
Concentrate (No. 2)	71	20.8	45.5
Tailings (No. 2)	71	17.3	37.8

Proximate composition:

	<u>Concentrate (No. 2)</u>	<u>Tailings (No. 2)</u>
Insoluble in A.R.	4.9%	15.9%
Tricalcium Phosphate	45.5	37.8
Calcium Carbonate	0.3	None
Undetermined	49.4	47.3
	<u>100.0%</u>	<u>100.0%</u>

Sizing analysis:

	<u>Tailings (No. 2)</u>
+ 28 mesh	0.0%
28/35	0.0
35/48	0.0
48/65	6.1
65/100	6.9
100/150	6.1
-150 mesh	80.9
Total	<u>100.0%</u>

Operational Efficiency of Separation: 54.4%

Combined Overall Efficiency of Separation 51.2%

Conclusions: The second flotation recovered 54.4% of its phosphatic charge. This figure does not compare very favorably with the first flotation where the efficiency was 49.2%. As to overall efficiency, a second flotation had the effect of a relative increase of 6.2%. This second treatment left unfloated tailings which had 80.9% in the -150 mesh, and assayed 17.3% P_2O_5 (37.8% B.P.L.). These results led to the conclusion that a second flotation is not commercially promising. The experiments involving flotation were terminated.

Pneumatic Concentration

The desirability of a product which would contain all of the potash and organic matter originally present in the mineral, suggested the study of a dry process. A sample was submitted to Federal Classifier Systems, Inc., of Chicago, for classification. This firm reported that the material was fractionated at 80-mesh in the Federal Laboratory Air Classifying Unit B and returned the fraction obtained therefrom.

Principle: In the deflector type of classifiers, cleavage is achieved by opposing a centrifugal current of air against a tangentially projected stream of material. The machine operates under partial vacuum. Each particle is influenced by two forces: A radial force toward a concentric discharge, and a centrifugal force of rotation outward.

Procedure: Three tests designated as Runs 23, 24, and 25 were made, each at different mesh valve opening. Air pressure was kept constant throughout the experiment.

Experimental Data:

Feed Charge, grams	2000
Size of Feed	Unscreened
Grade of Feed, in % P_2O_5	6.8
Air Pressure (arbitrary reading)	20
Valve Openings; dial	13-15-17

Results: The results have been arranged in Tables No. 18, No. 19, and No. 20.

Table No. 18. Results of Pneumatic Concentration

Run	Mesh Valve Opening	Weight of fines grs.	Grade of Fines		Grade of Coarse % P_2O_5	Efficiency of Separation
			% P_2O_5	% B.P.L.		%
23	13	311	16.2	35.4	2.3	37.0
24	15	638	17.1	37.4	2.4	80.2
25	17	450	17.6	38.5	2.1	58.2

Table No. 19. Composition of Pneumatic Concentration Products

	Run 23		Run 24		Run 25	
	Fines %	Coarse %	Fines %	Coarse %	Fines %	Coarse %
Insoluble in A.R.	7.1	29.5	6.1	30.9	5.4	28.8
Tricalcium Phosphate	35.4	5.0	37.4	5.2	38.5	4.6
Calcium Carbonate	8.6	55.0	2.9	52.5	7.7	52.5
Undetermined	<u>48.9</u>	<u>10.5</u>	<u>53.6</u>	<u>11.4</u>	<u>48.4</u>	<u>14.1</u>
Totals	100.0	100.0	100.0	100.0	100.0	100.0

Table No. 20
Sizing Analysis of Pneumatic Concentration Products

	Run 23		Run 24		Run 25	
	Fines	Coarse	Fines	Coarse	Fines	Coarse
	%	%	%	%	%	%
+65 mesh	5.7	80.4	4.9	68.0	6.2	91.1
65/100	4.8	8.4	3.9	11.0	4.2	6.9
-100		11.2		21.0		2.0
100/150	3.8		6.8		6.3	
-150	<u>85.7</u>	_____	<u>84.4</u>	_____	<u>83.3</u>	_____
Totals	100.0	100.0	100.0	100.0	100.0	100.0

Discussion: None of the fines assayed high enough to pass the specification requirements. However, upon looking at the size distribution, it was seen that all samples contained around 90% of -100 grains. It is suggested that a fractionation at 100-mesh would yield fines assaying more than 18% P_2O_5 (39.2% B.P.L.), because of the elimination of the larger, less valued particles. It should be noticed that the phosphoric content of the rejected coarse fractions was among the lowest of all of the experimental tests. The high efficiency of separation obtained in Run No. 24 is indicative of possible, successful commercial application.



Fig. 31 Product from Pneumatic Concentration

Filtration

As most of the recovery methods investigated were wet, a filtration test upon the concentrate was desirable. The knowledge of the characteristic constants of the cake would provide the necessary data for the selection of a full scale filter. Filtration was thought to be the best method for the separation of fines from a liquid suspension. A suspension of fines was filtered at constant pressure at three different pressures.

Theory: The integrated and simplified equation for filtration at constant pressure is expressed as:⁷

$$\frac{P(\theta - \theta_r)A}{V - V_r} = \frac{r'' v}{a} P^s \frac{\mu a (V - V_r - 2V_c)}{2A} + \mu \rho' P^m$$

where

P = pressure differential across cloth and cake, lbs/in²

θ = total time of filtering, min.

θ_r = time of filtering before the desired constant pressure is reached, min.

A = cloth area, in²

V = total weight of filtrate collected, lb.

V_r = weight of filtrate collected during θ_r , lb.

r'' = cake resistivity

v = volume of cake, in $3/1b.$ filtrate

a = concentration of slurry, lb. solid/100 lb. filtrate.

s = cake resistivity exponent

μ = viscosity of filtrate, centipoises

V_c = weight of filtrate trapped in the filtrate line, lb.

ρ' = cloth resistivity

m = cloth resistivity exponent

Equipment, Procedure and Results: Filtration was conducted in a Sperry filter-press of the plate-and-frame type, connected for either reverse or forward washing. Size was 11" x 11" with 6 frames and 7 plates. The slurry was made up by suspending fines, approximately -100 mesh size, in water; concentrations of 7.2 and 11.1 lb. solid/100 lb. of filtrate were used. The concentrate was free-filtering. Filtration tests were made at 5.2 psi; 10.2 psi; and 15.2 psi. Experimental values and results were recorded in Figs. No. 22, 23, 24, and 25.

The value found for s , cake resistivity exponent, was 0.343. Washing rates upon full cakes at different pressures are reproduced in Fig. No. 26. The filter cloth used was a Twill weave, $21/3$ ply warp, $21/3$ ply filling, 20.8 oz./yd². The resistance was determined at two different pressures; the values found for ρ' and m were 8.77 and 0.79, respectively. They appear in Figs. No. 27 and 28.

Fig. 22 EXPERIMENTAL RESULTS FOR FILTRATION OF
PHOSPHATE CONCENTRATES AT CONSTANT PRESSURE

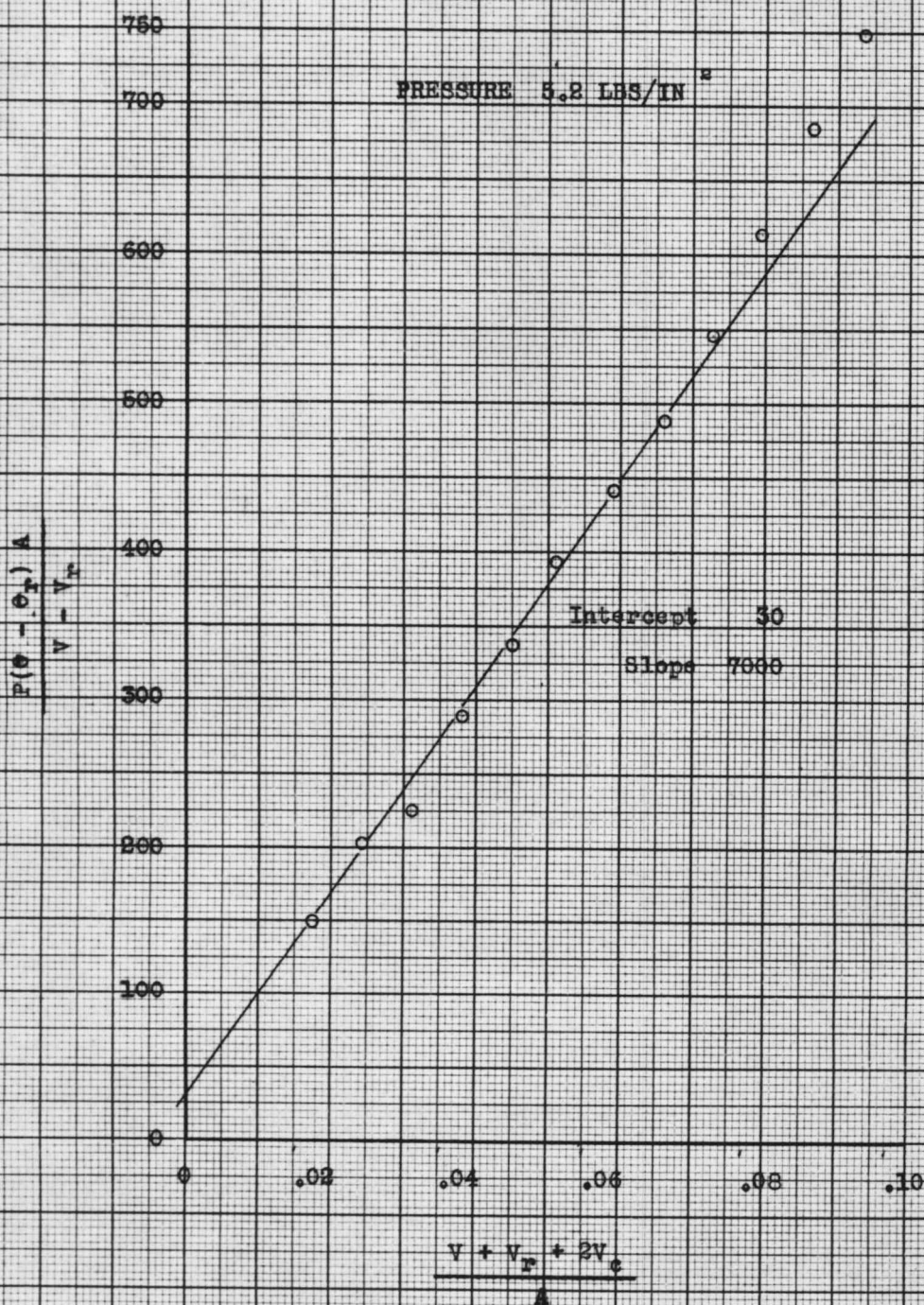


Fig. 23

EXPERIMENTAL RESULTS FOR FILTRATION
OF
PHOSPHATE CONCENTRATES AT CONSTANT PRESSURE

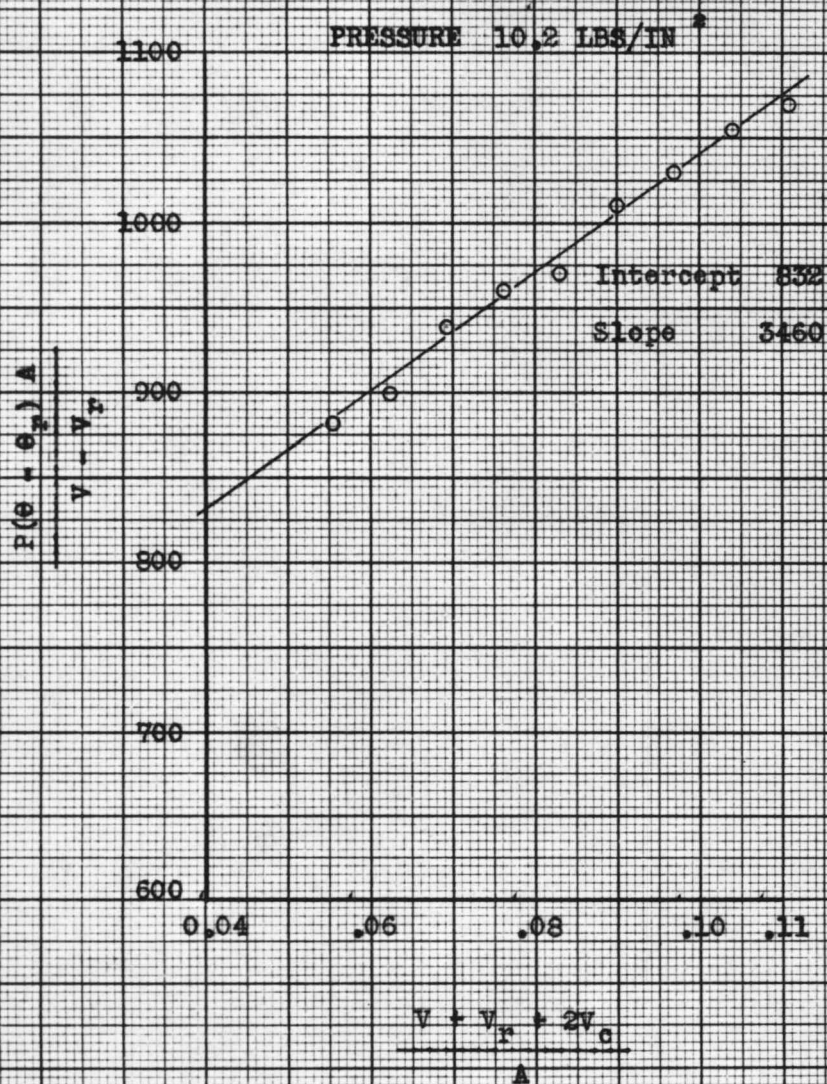
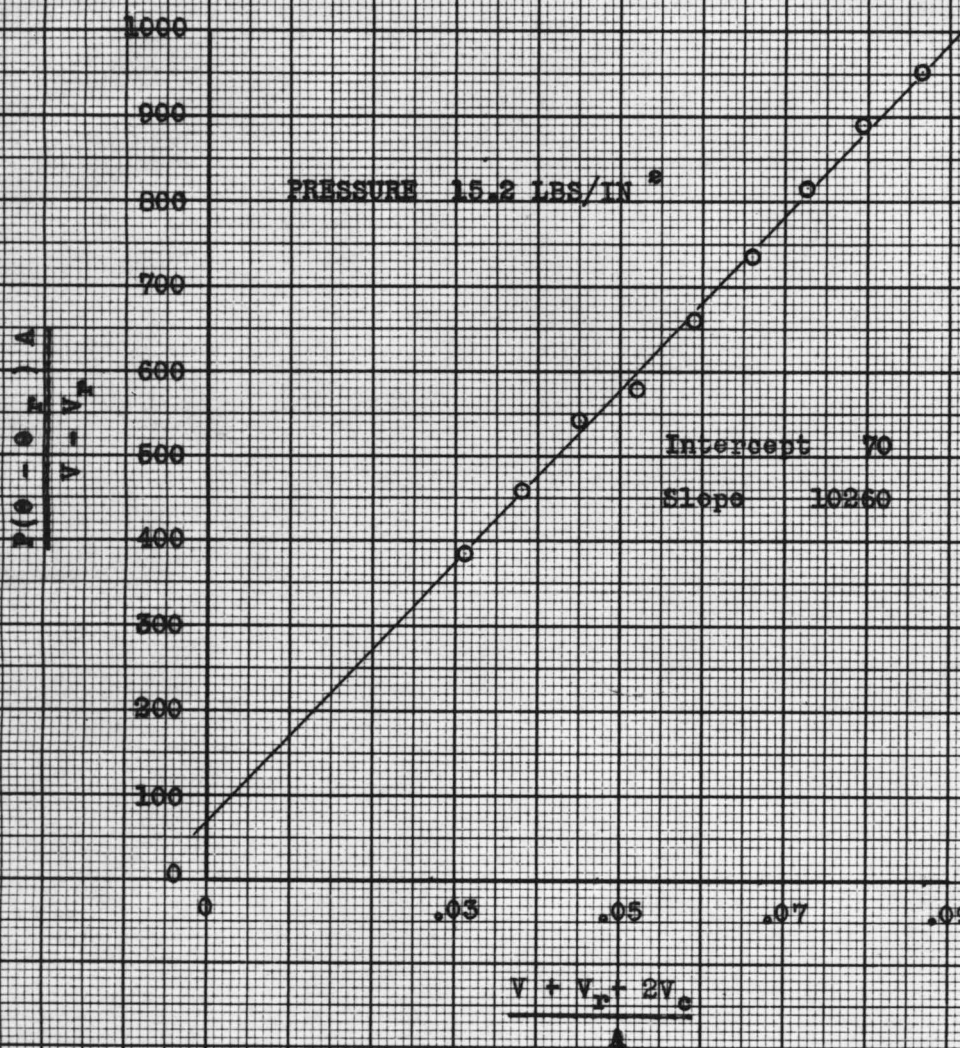
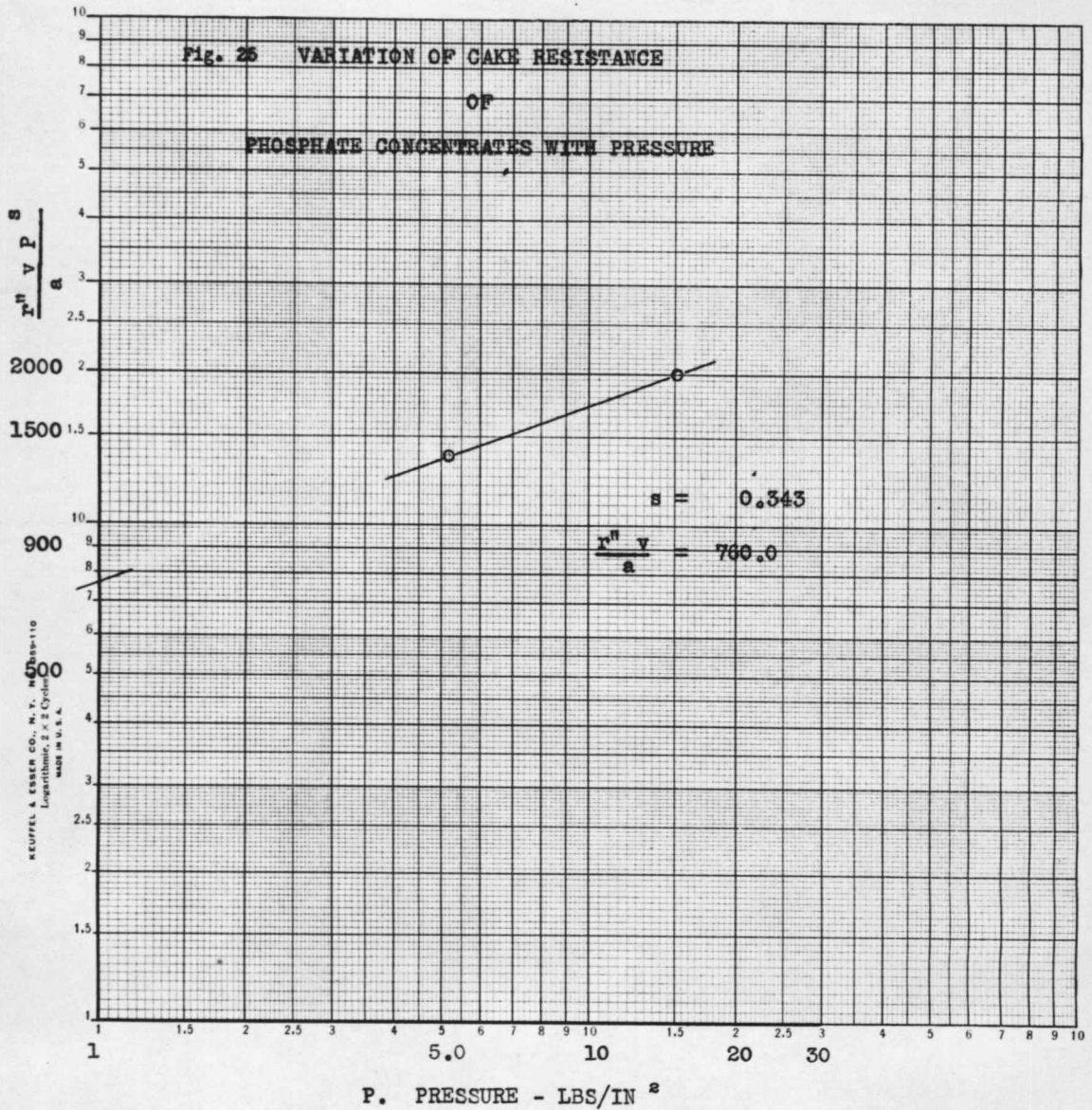


Fig. 24

EXPERIMENTAL RESULTS FOR FILTRATION OF PHOSPHATE CONCENTRATES AT CONSTANT PRESSURE





WASHING RATES. GALS/MIN

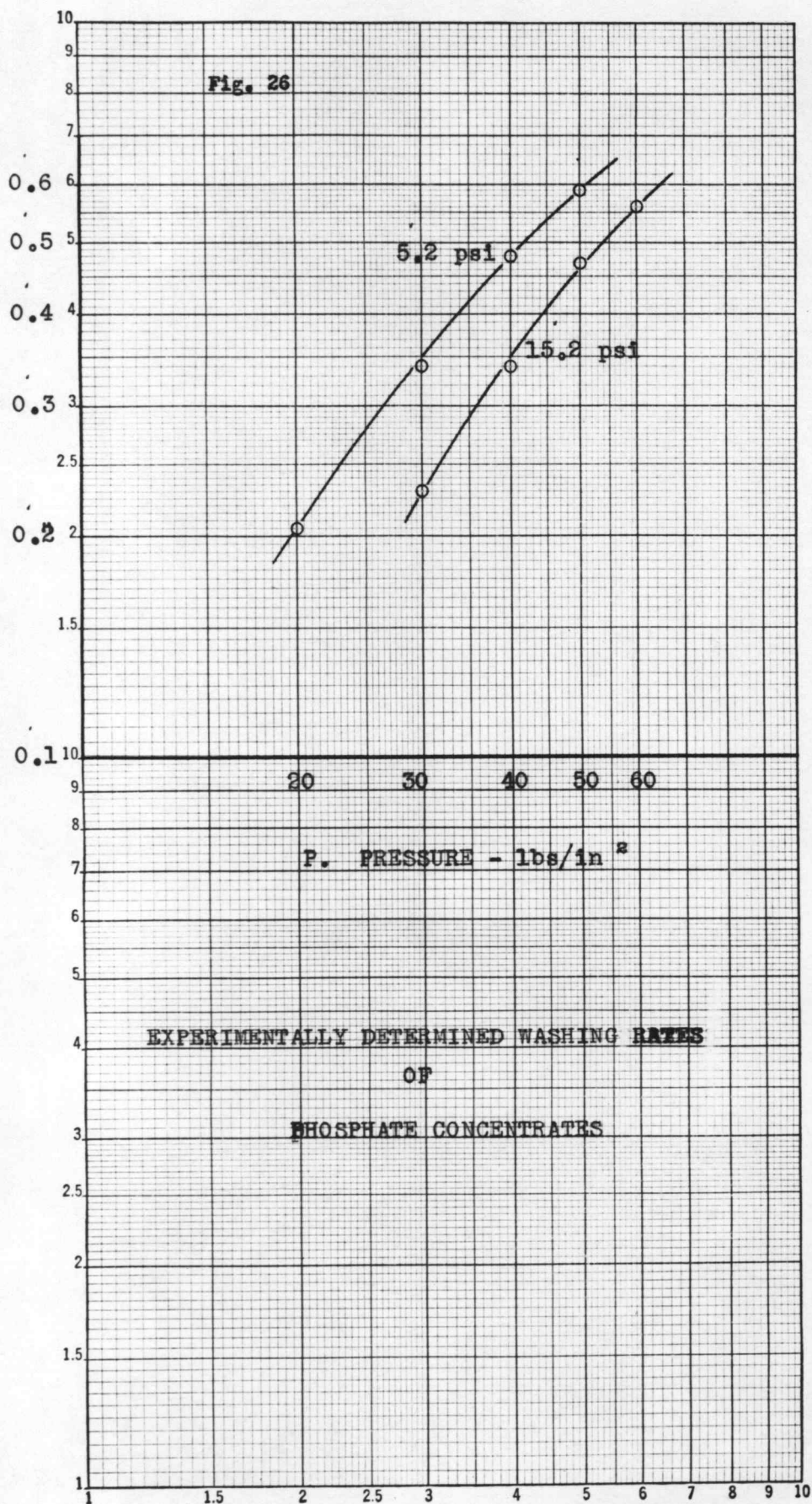


Fig. 27 EXPERIMENTAL RESULTS FOR FILTER CLOTH RESISTANCE

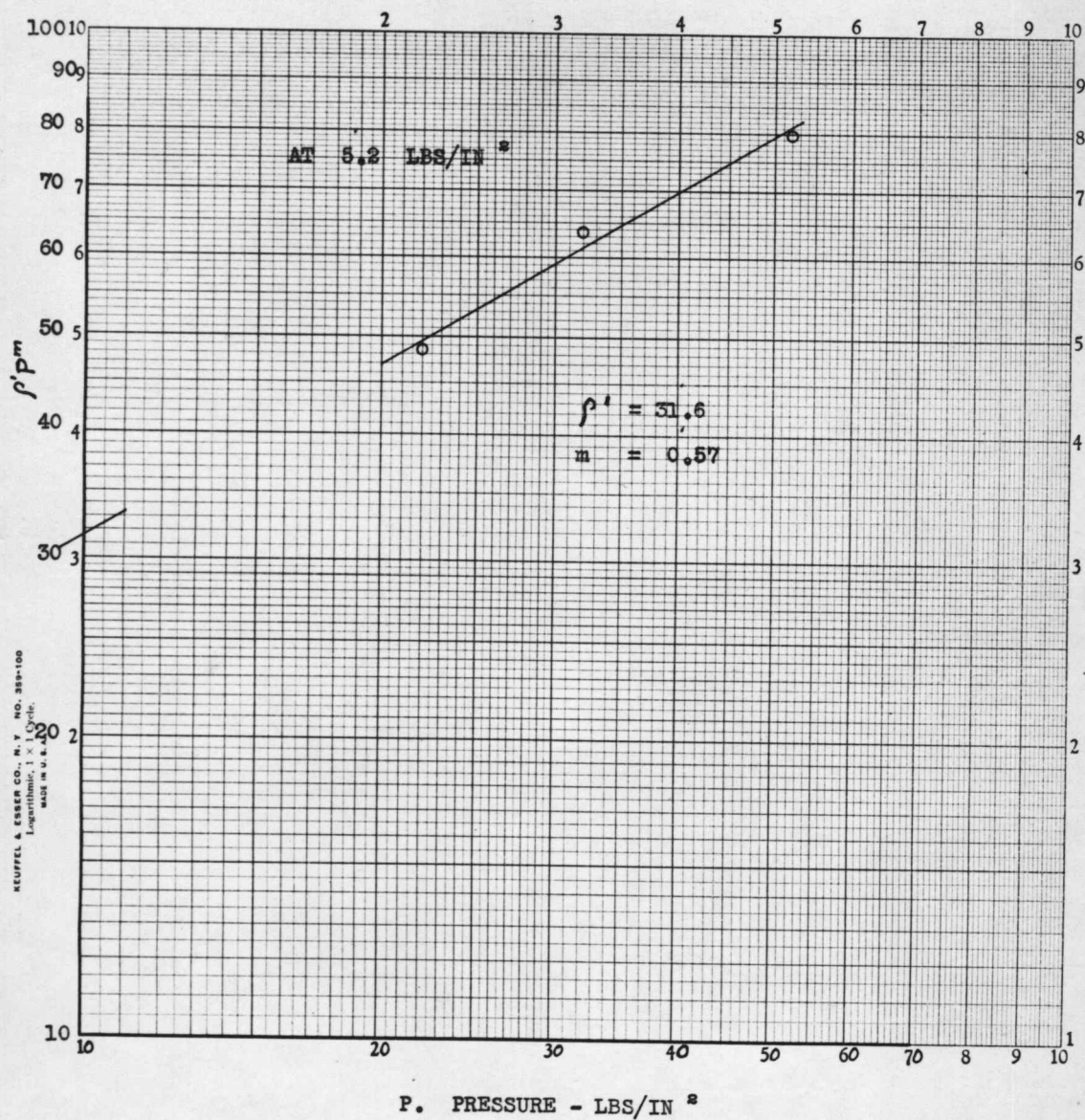
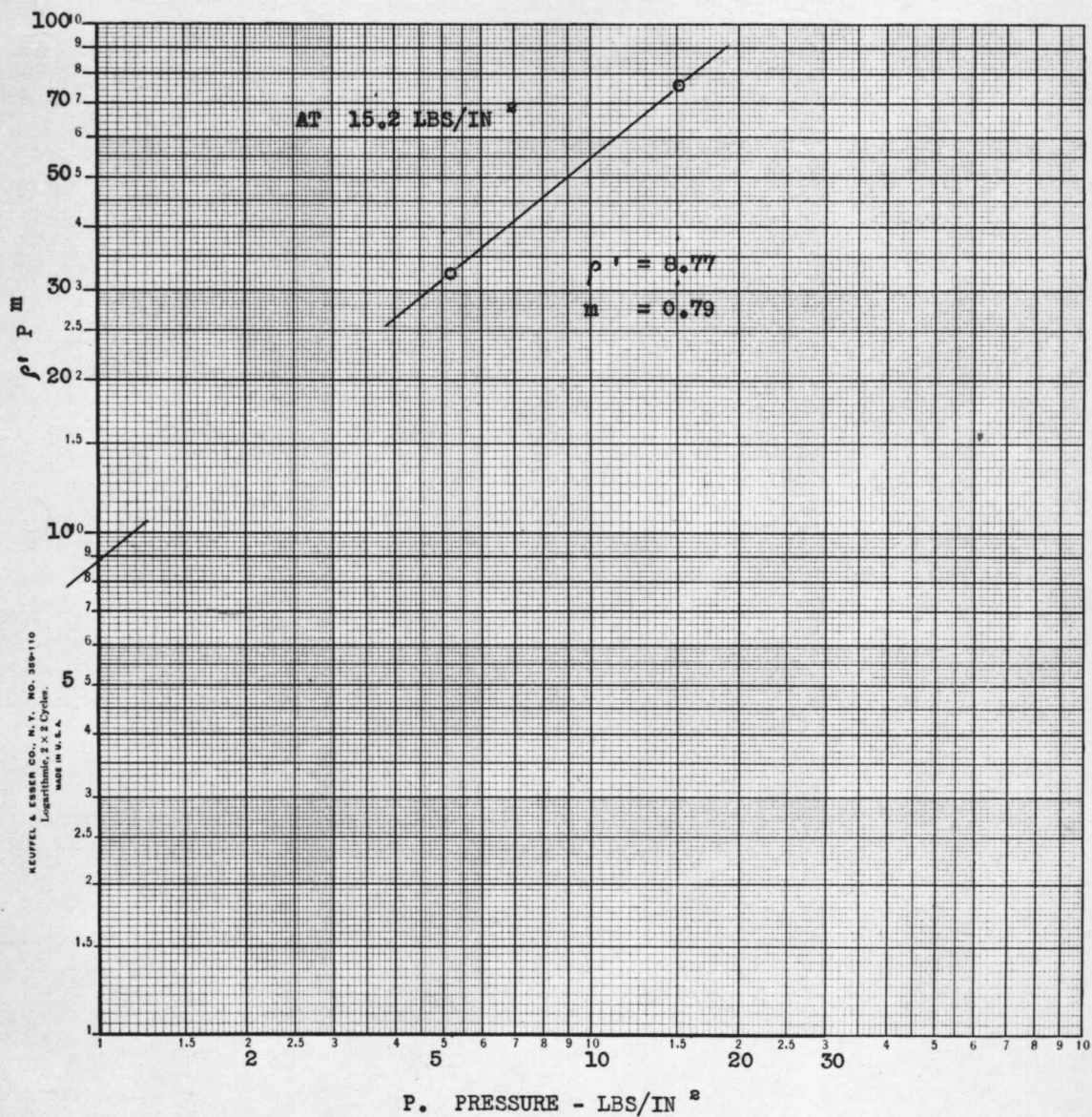


Fig. 28 EXPERIMENTAL RESULTS FOR FILTER CLOTH RESISTANCE



Conclusions and Recommendations

From the results reported, it was concluded that this ore could be concentrated to produce a commercial phosphatic fertilizer. Overall Efficiencies of Separation of 82%, 77%, and 80% were attained by Hydraulic Classification, Wet Screening and Pneumatic Concentration, respectively. The products that were obtained meet the specifications required both as to grade and size composition. Products assaying 22% P_2O_5 (48.1% B.P.L.) were prepared by Hydraulic Classification and Wet Screening; they contained 1% Nitrogen and no potash.

As to the concentrate produced by Pneumatic Separation, and based upon the results for 80-mesh, it could be safely said that a grade of 19% P_2O_5 (41.5% B.P.L.) would be reached should the cleavage be made at 100-mesh. The product contained 1.3% Nitrogen and 4.0% of Potassium Chloride. Regarding the availability of the phosphatic content, it was found that 92% of the phosphoric grade in all the products was soluble in ammonium citrate.²

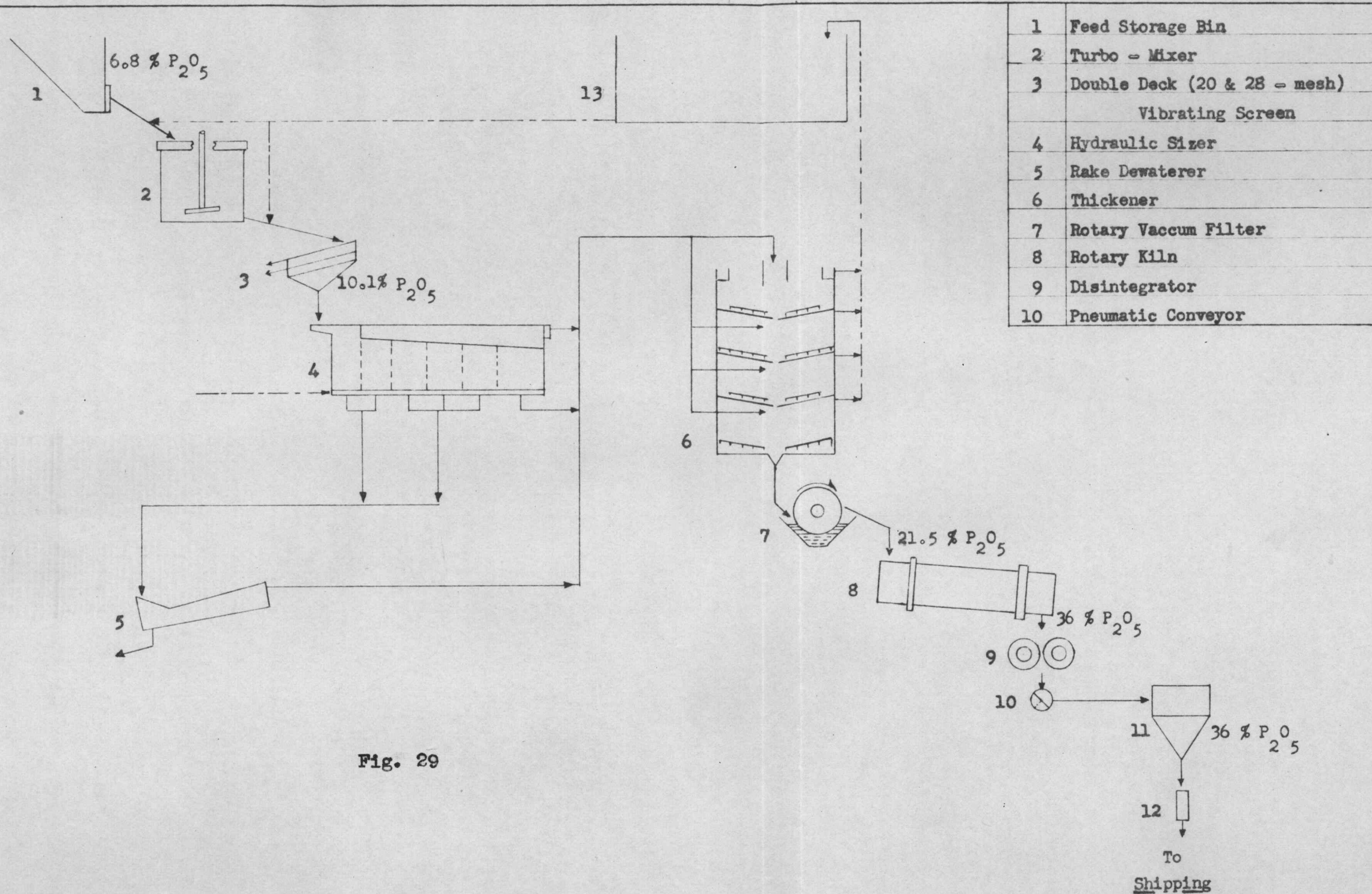
By calcination of the concentrate produced by the wet processes, a fertilizer containing 36% P_2O_5 is obtainable.

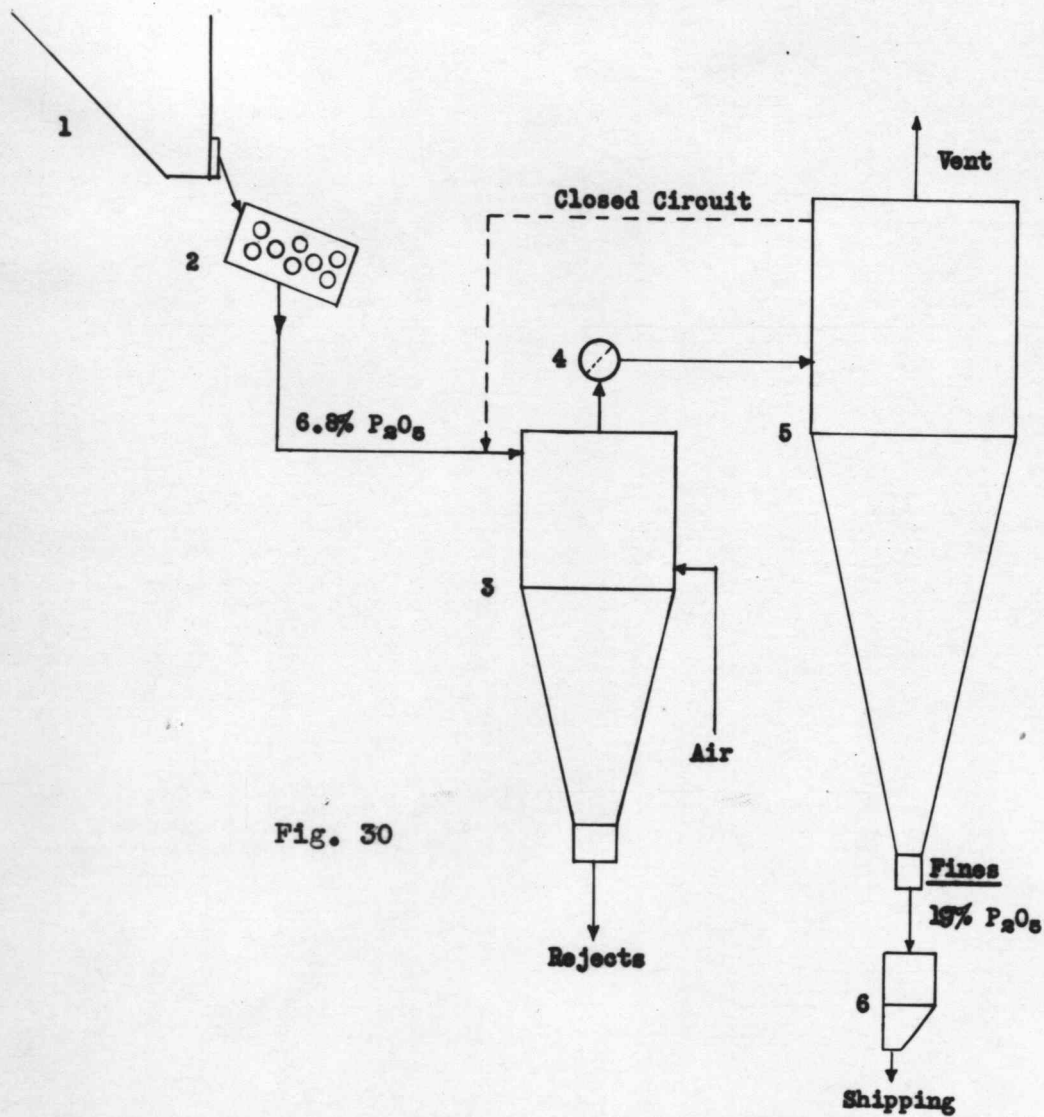
The combined processes of Hydraulic Classification and of Pneumatic Concentration is recommended. The first process, supplemented with calcination, would produce a commodity with a higher grade, thus keeping the shipping and handling costs at a minimum. The pneumatic process would produce a fertilizer which, besides the phosphoric value, contains

organic matter and potash. Figs. No. 29 and No. 30, pages 107 - 108, are flow sheets for the two processes which are recommended. In order to promote efficiency of separation, the raw material should be free of unusually large rocks and deprived of the coarser grains, perhaps 6 mesh. A double-deck, wet, vibrating screen, and a dry trommel would serve the purpose in Hydraulic Classification and in Pneumatic Concentration, respectively.

Operation of the two processes in a Pilot-Plant-scale is strongly recommended before undertaking the construction of any commercial plant. Tests of continuous operation would provide the best data for the design and selection of the final equipment, as well as for optimum operating conditions.

The result of this preliminary research, if carried to a logical conclusion by further work, should constitute a definite contribution to the possible utilization of a natural resource, at present unused. For the circumstances peculiar to Chile, the extension of the processes described in this thesis should contribute to the national welfare. Due to the fact that a specification product can be prepared, and due also to the fact that equipment requirements for the recommended processes are not complicated, there are indications that an industry operating as suggested, should be feasible and economic.





1	Feed Storage Bin	<p>PROPOSED FLOW SHEET for the PNEUMATIC UPGRADING OF UNGROUND PHOSPHATIC SANDS</p>
2	3/16 in. Trommel	
3	Air Classifier	
4	Pneumatic Conveyor	
5	Collector	
6	Bagging Scale	

Items for Further Research

It is suggested that further research be conducted along the following general lines:

1. The exact physical and chemical nature of the brown, phosphate-bearing complex and of the organic matter should be determined.
2. Investigation should be made as to the presence of minor, nutrient elements.
3. An analysis should be made of the nature and properties of the foam produced whenever the material is stirred in water.

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	<u>Page</u>
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