Beneficiation of Idaho Phosphate Rock

By

Newton and Finkelnburg

Idaho Mineral Resources Report No. 3

June, 1947

Issued under grant from the Special Research Fund

School of Mines, University of Idaho

in cooperation with

Idaho Bureau of Mines and Geology

Published by the University of Idaho, Moscow, Idaho

Special Research Program

RECOGNIZING the important part which research can play in the development of the State the 1945 session of the Idaho Legislature made a special appropriation for technical investigations in the fields of agriculture, engineering, forestry, mining, metallurgy and related activities. This special research program was continued and expanded by the 1947 Idaho Legislature. Following the spirit and intent of the appropriation act those problems are considered which are widely applicable throughout the State of Idaho and the results of which it is hoped will contribute directly to the development of Idaho, the utilization of its widespread resources, and have immediate application to its post-war economy.

The Special Research Program is administered by the University of Idaho Research Council. The individual projects are carried on by the various experimental and research divisions of the University. The investigation described in this bulletin is Project No. 6 in the Special Research Program. The work was done by the School of Mines, University of Idaho, in cooperation with the Idaho Bureau of Mines and Geology and under the general supervision of Dean A. W. Fahrenwald.

Beneficiation of Idaho Phosphate Rock

By

JOSEPH NEWTON
Professor of Metallurgy, University of Idaho

and

OSCAR C. FINKELNBURG
Research Fellow, School of Mines, University of Idaho

INTRODUCTION

ONE of the largest reserves of phosphate rock in the world is found in the Phosphoria formation of the Permian age in the western United States and Canada. Figure 1 shows the distribution of the phosphate-bearing beds. Idaho has the largest reserve of high-grade phosphate rock in the United States.

Phosphate Reserves of the United States*
(After Mansfield)

Eastern States	Thousands of Long Tons	Western States	Thousands of Long Tons
Florida	5,081,839	Idaho	5,736,335
Tennessee	194,468	Montana	391,323
South Carolina	8,798	Utah	1,741,480
Kentucky	863	Wyoming	115,754
Arkansas	20,000		
Total	5,305,968	Total	7,984,892
Grand Total	13,290,860		

The Permian phosphate-bearing formation is believed to be of marine origin. The phosphate beds are contained in the phosphate shale member of the Phosphoria formation; this is from 25 to 180 feet thick and consists of various sandstones, limestones, and shales plus one to three beds of high-grade phosphate rock. The high-grade beds are from 4 to 7 feet thick and contain from 32 to 35 per cent P_2O_5; they are black, gray, or brown in color and have an oolitic structure.

At the present time only the high-grade oolitic beds have any commercial value, and the lower-grade portions of the phosphate shale beds are not used. The estimate of Idaho's phosphate reserve is based on the phosphate content of the high-grade beds only.

*Johnson, Bertrand L., Economic Factors in the U. S. Phosphate Industry; Min. and Met., October, 1944.

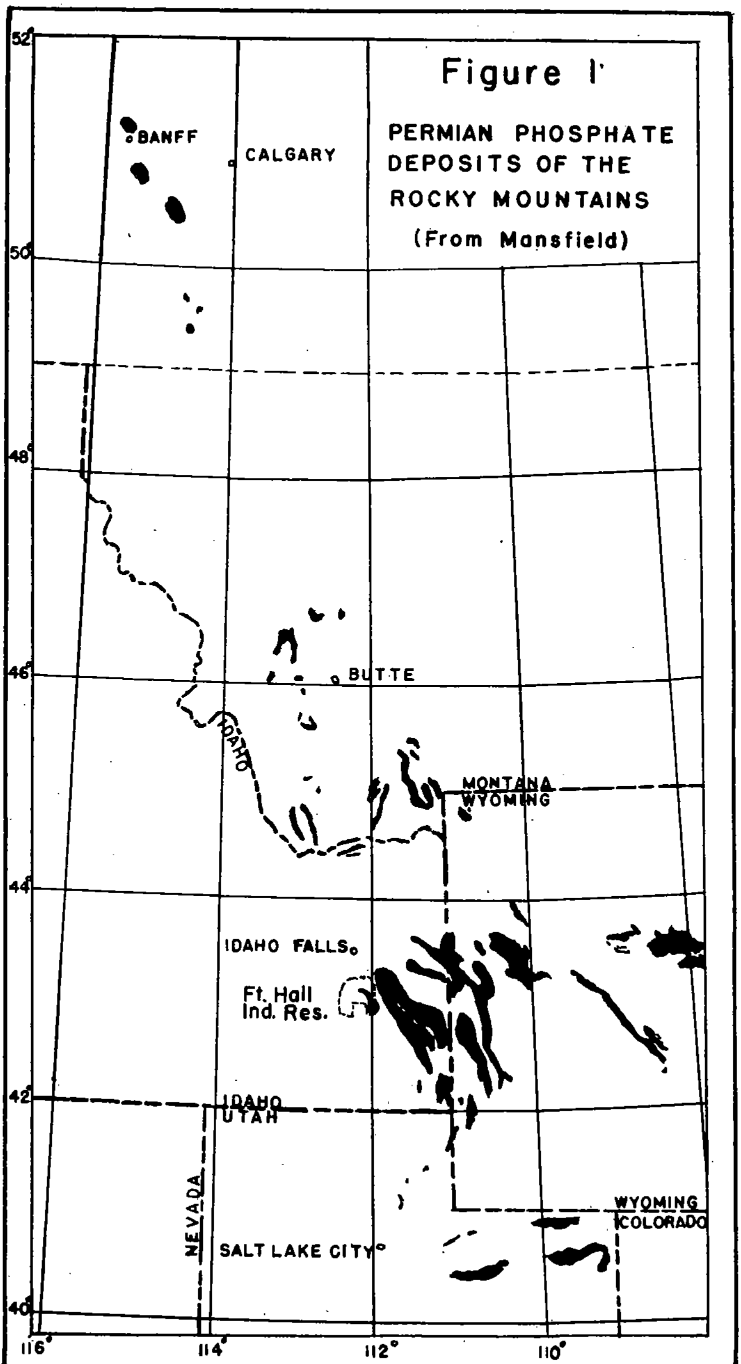
Figure I
PERMIAN PHOSPHATE DEPOSITS OF THE ROCKY MOUNTAINS
(From Mansfield)
BANFF
CALGARY
BUTTE
IDAHO
MONTANA
WYOMING
IDAHO FALLS
Ft. Hall Ind. Res.
IDAHO
UTAH
NEVADA
SALT LAKE CITY
WYOMING
COLORADO
52°
50°
48°
46°
44°
42°
40°
116°
114°
112°
110°

Most of the phosphate rock mined is converted into superphosphate fertilizer by acidulation with sulfuric acid, and for this purpose a minimum of 31.5 per cent P_2O_5 is desired. The purpose of this investigation was to seek a method of beneficiating some of the lower-grade portions of the Phosphoria shale deposits to produce a concentrate of acceptable grade.

Throughout this paper the assays of various products will be reported as per cent of P_2O_5. In many discussions, phosphate concentrations are given as per cent of "bone phosphate of lime" (BPL) which is tricalcium phosphate, $Ca_3(PO_4)_2$. Values of P_2O_5 content can be converted to per cent BPL by multiplying by the factor 2.18; thus 31 per cent P_2O_5 is equivalent to 69 per cent BPL. We shall give the BPL equivalents for most of the important assays in the course of the discussion because this notation is undoubtedly more familiar to many readers.

THE NATURE OF THE PHOSPHATE ROCKS

HIGH GRADE. By high-grade we shall mean the commercial high-grade rock that is suitable for direct acidulation. Figure 2 is a photomicrograph of a thin section of such rock; this is a sample of commercial rock from a deposit on the Fort Hall Indian Reservation near Pocatello, Idaho.

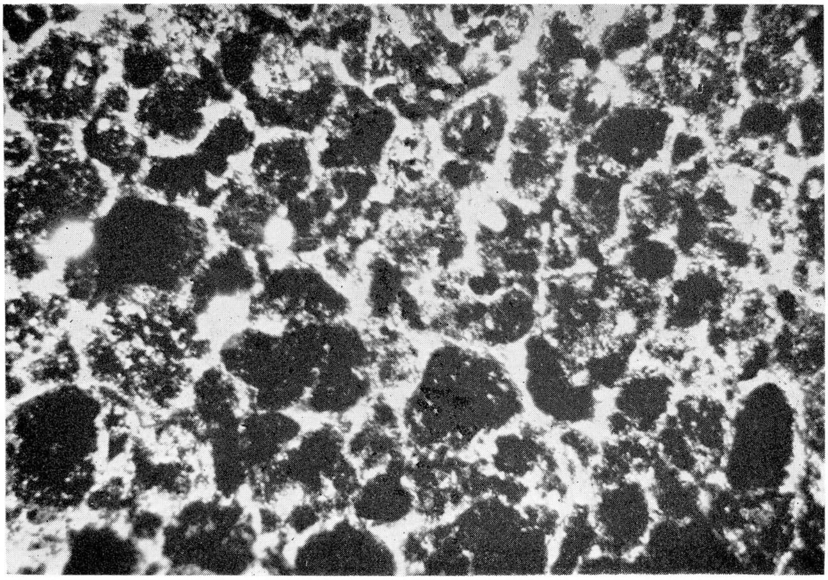

Figure 2—(x50) Fort Hall High-Grade Rock 34.2% P_2O_5

SHALE. By the term "shale" we shall mean the lower-grade phosphate rock as distinguished from the commercial "high-grade" rock. Actually these "shales" are not homogeneous rocks but are composed of shales, mudstones, and limestones interbedded with thin layers of black oolitic high-grade rock.

The shale samples investigated contained about 26 per cent P_2O_5 as an average; certain portions contained as little as 3 per cent P_2O_5 (6.5% BPL).

STRUCTURE. These phosphate rocks (both shales and high-grade) have certain characteristics in common as follows:

1. They are all soft, friable rocks, and offer little resistance to crushing and grinding.

2. The principal phosphate-bearing material is in the form of black oolitic particles cemented together by lighter-colored material. Some coarse oolites were hand-picked from a sample of crushed high-grade rock and assayed; these contained 35.6 per cent P_2O_5 (77.6% BPL) and this represents about the highest grade that could be expected in a concentrate made from these rocks.

3. From the standpoint of concentration methods, the difference between high-grade rock and the lower grade shales is a difference of degree rather than of kind. The lower-grade rocks contain fewer of the black oolites and more of the cementing material. Also, the high-grade beds appear to contain coarser oolites than the shales.

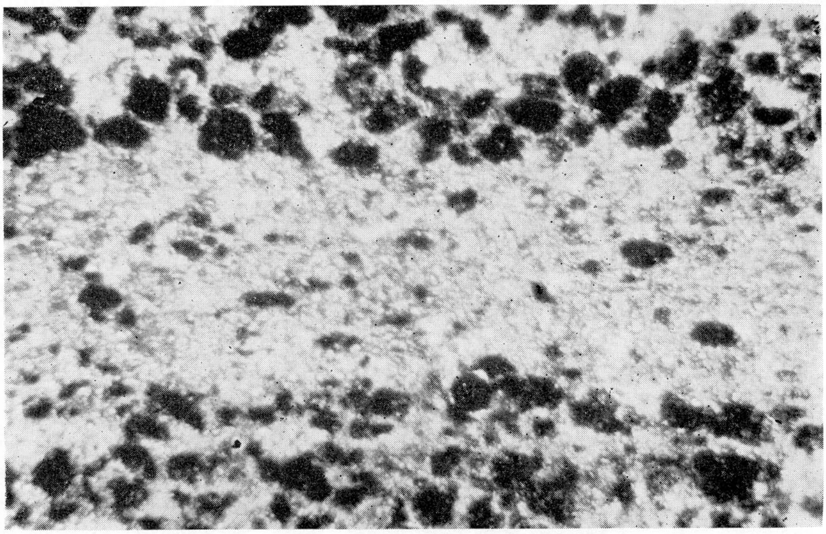

Figure 3—(x50) Fort Hall Shale 19% P_2O_5

Figures 2, 3, 4, 5 and 6 are photomicrographs of thin sections of various phosphate rocks, and it will be noted that the oolites are more abundant in the higher-grade material.

SAMPLES. Most of the work was done on a large sample of phosphatic shale from the mine of the Simplot Fertilizer Company on the Fort Hall Indian Reservation near Pocatello, Idaho. Some work was also done on a shale sample from Montpelier, Idaho, from the mine of the San Francisco Chemical Company. Samples of high-grade rock from both sources were also available.

PRELIMINARY TEST

A sample of the Montpelier shale was placed in a small laboratory ball mill with enough water to cover the rock and allowed to grind for one hour; at the end of this period the material was screen-sized and each size fraction was assayed for P_2O_5. The material was run-of-mine rock and the pieces were about 4 inches in diameter (maximum); no grinding medium was used except the rock itself. The results of this test are given in Tables 1 and 2. The —325 mesh fraction was separated into sands and slimes by decantation.

Table 1

Screen Analysis of Ground Sample of Montpelier Shale. (One-Hour Grind.)

Head Assay = 26.1% P_2O_5

Size (Mesh)	Total Weight Grams	Weight %	Assay % P_2O_5	Recovery % of Total P_2O_5
+3	1003	41.1	27.5	43.3
3/4	98	4.0	26.8	4.1
4/6	46	1.9	28.3	2.0
6/8	38	1.6	28.7	1.7
8/10	15	0.6	27.9	0.6
10/14	5	0.2	27.6	0.2
14/20	2	0.1	27.2	0.1
20/28	1	0.05	27.0	0.05
28/35	2	0.1	26.2	0.1
35/48	5	0.2	29.8	0.2
48/65	11	0.4	32.2	0.6
65/100	40	1.6	33.7	2.1
100/150	97	4.0	33.2	5.0
150/200	215	8.8	32.3	10.9
200/325	170	7.0	29.4	7.8
—325 sand	242	9.9	22.6	8.6
—325 slime	450	18.4	17.6	12.4
Total	2440			

Table 2

Screen Analysis, Assays, and Recoveries of —48 Mesh Material from One-Hour Grind (Table 1).

Calculated Head Assay = 24.7% P_2O_5

Size (Mesh)	Weight Grams	Weight %	Assay % P_2O_5	Recovery % of Total P_2O_5
48/65	11	0.9	32.2	1.1
65/100	40	3.3	33.7	4.5
100/150	97	7.9	33.2	10.6
150/200	215	17.5	32.3	23.0
200/325	170	13.9	29.4	16.5
—325 sand	242	19.7	22.6	18.1
—325 slime	450	36.8	17.6	26.2
Total	1225	100.0		100.0

The large amount of +3 mesh material in Table 1 represents the coarse rocks that remained in the mill. The data in Table 2 is based on the assumption that the —48 mesh material may be considered the "finished product" of this grind, and the table shows the distribution of P_2O_5 in the —48 mesh products.

Tests on the individual pieces of low-grade shale showed that they had a chalky texture and were quite soft; when a piece is crushed with a hammer it tends to disintegrate into an impalpable powder. This fact together with the data in Tables 1 and 2 led to the following observations.

1. The screen analysis shows relatively few particles in the size ranges between 10 mesh and 48 mesh, but shows considerable amounts in the sizes finer than 48 mesh. This abnormal size distribution is characteristic of a non-homogeneous rock containing hard and soft portions; when such a rock is crushed it tends to show relatively large amounts of material in the range of the "natural grain size" of the harder minerals.

2. The material between 48 mesh and 200 mesh was black in color and contained about 33 per cent P_2O_5 (72% BPL) which is well above the minimum limit desired for acidulation (31.5% P_2O_5).

3. The slime washed from the —325 mesh material was brown in color and contained only 17.6 per cent P_2O_5 (38.4% BPL). The weight of this slime was relatively large.

4. It appeared that the phosphate oolites, therefore, were more resistant to grinding than the soft chalky material between the oolites. The low-grade material formed a very fine brown slime; the high-grade material remained as a black sand.

Slimes are usually very difficult to handle in a concentrating operation, and it seemed likely that it would be necessary to deslime the ground rock as a preliminary to almost any concentrating process. In these rocks, however, it is possible to reduce most of the "gangue" to slime, and it seemed that the most promising method for concentration would consist of controlled grinding and desliming of the ground product. This line of attack was followed throughout most of the investigation.

ROCK MILLING

GRINDING. The rock milling tests were made in a small cylindrical mill 1 foot in diameter and 4 inches long; it was mounted on driving rolls and turned at a speed of 50 RPM (65 per cent of the critical speed). The mill was charged and discharged by removing one side of the mill, and it was completely closed while running; each grind was, therefore, a batch test. The mill had a smooth lining, and the combination of smooth lining and low speed of rotation gave a very gentle grinding action.

THE CHARGE. The material charged to the mill was the run-of-mine rock just as it came from the sample sacks. The only preparation given the rock was to break up pieces with the hammer when they were too large to fit in the mill.

The shales are quite soft and they break up along the bedding planes so that the majority of the pieces are in the form of flat slabs. All the pieces become uniformly covered with a brown dust when they are exposed to the atmosphere, and in the samples as they are received there is no apparent difference between the high-grade and low-grade portions of the rock.

A charge of rock was added to the mill together with enough water to completely cover the rock. No balls, pebbles, or other grinding media were added; the rock was simply allowed to grind itself. After charging, the side of the mill was bolted in place, the mill placed on the driving rolls and allowed to run for a definite interval; when the run was complete, the side of the mill was removed and the contents discharged into a tub.

EFFECT OF THE GRIND. After a charge of rock had been allowed to grind in the mill for a definite time (usually 15 to 30 minutes) it displayed a considerably altered appearance. The water would be charged with a very fine brown slime which could be quite easily separated from the sand by two or three decantations. The fine sand would be black in color and coarser particles would be rounded off like water-worn pebbles. In the case of the coarser pieces it was now quite easy to distinguish between the high-grade and low-grade rocks.

The mill made very little noise while running and this, together with the appearance of the ground product, makes it appear that the grinding is largely the result of attrition caused by the pieces rubbing together. There probably is not much crushing or grinding by the cascading and impact of the larger pieces.

Almost all the rocks found in these shale members are quite soft and become rounded off after a short period of grinding. The only exception is a black chert which is found in small quantities; this chert resists the grinding almost completely, and the pieces retain their sharp edges and angular shapes even after prolonged grinding.

TREATMENT OF THE GROUND PRODUCT. After a sample of rock had been ground for the required time, the mill was opened and discharged through a screen into a tub. This screen had ⅝-inch openings and served to retain the coarser products which were washed and returned to the mill. The material remaining in the tub was then wet-screened on a finer screen (20 mesh in some tests, 48 mesh in others) and the material passing this fine screen was considered the "finished product" of the grind. The oversize was returned to the mill with the +⅝-inch rock.

The "finished product" was then separated into "sand" and "slime" by repeated decantations. It was agitated with water, allowed to stand for about 30 seconds, and then the water containing the brown slime was poured off. Three or four decantations usually sufficed to separate the brown slime from the black sand. Sand and slime samples were then dried, weighed, and assayed for P_2O_5. In some tests a portion of the ground rock was "deslimed" by screening on a 325 mesh screen and the —325 mesh material was considered the "slime" (tailing).

LOCKED-GRIND TESTS. The rock-mill tests were conducted in a series of locked-grind tests designed to approximate a continuous grinding operation. After a single grind (cycle) the "finished product" was screened out and enough new rock added to the mill to equal (approximately) the weight of the finished product removed; the mill was then run for the second cycle, and so on. Each test, therefore, consisted of several grinding cycles.

The sand and slime from each cycle was weighed and assayed separately and the results totaled. Table 3 summarizes the results of these rock mill tests. The recoveries are all calculated on the basis of the total amount of P_2O_5 in the finished product.

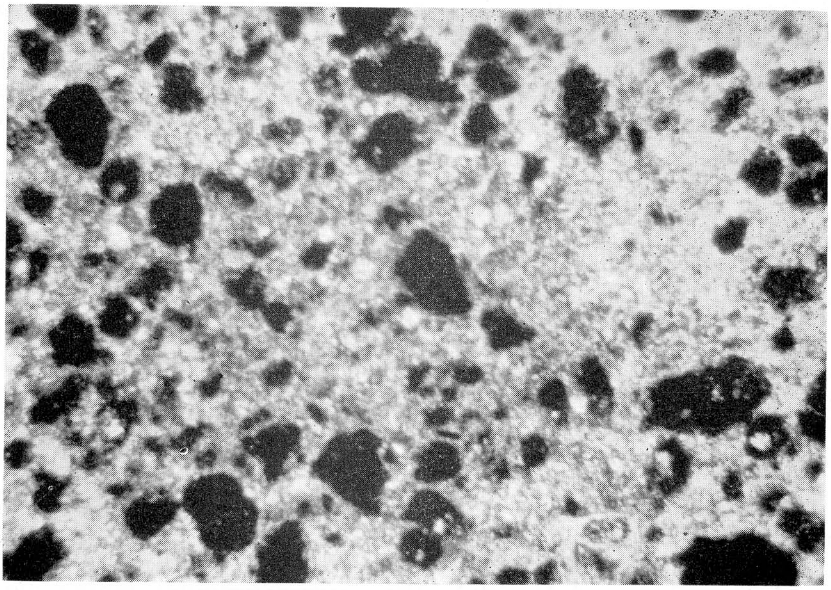

Figure 4—(x50) Fort Hall Shale 16.9% P_2O_5

RESULTS OF ROCK MILLING TESTS. The results summarized in Tables 3 and 4 show that it is possible to produce a satisfactory concentrate from some of these shales by rock milling and desliming. Several points should be noted in connection with these data.

(1) The second test on the Fort Hall shale is a decided improvement on the first test, both with respect to recovery and grade of concentrate. The second test utilized more and shorter grinding cycles and there was more rock in the mill. This suggests that the

Table 3

Results of Rock Mill Tests

Material Ground	Head Assay % P_2O_5	Weight of Rock Initially in Mill, Grams	No. of Cycles	Time per Cycle, Minutes	Mesh of Separation of Finished Product	Residue Left in Mill		Sands (Total)			Slimes (Total)	
						Grams	% P_2O_5	Grams	% P_2O_5	Recovery of P_2O_5 %	Grams	% P_2O_5
Fort Hall Shale	23.7	2000	6	30	48	1325	23.5	3092	29.6	83.5	1533	11.8
Fort Hall Shale	24.0	3000	11	15	20	1935	13.5	8001	30.2	85.9	2898	13.7
Fort Hall High-Grade	32.7	3000	5	15	20	4360	34.0	5786	34.85	94.4	2009	14.0
Montpelier Shale	25.0	3000	11	15	20	2091	23.9	5137	28.9	76.3	2560	17.9

Table 4

Effect of Desliming by Decantation as Compared with Screening on 325 Mesh

(Tests as given in Table 3)

Sample	Desliming by Decantation		Removal of All —325 Mesh Material	
	Grade of Conc. (sand) % P_2O_5	Recovery of P_2O_5 %	Grade of Conc. % P_2O_5	Recovery of P_2O_5 %
Fort Hall Shale	29.6	83.5	......	......
Fort Hall Shale	30.2	85.9	32.3	79.5
Fort Hall High-Grade	34.85	94.4	......	......
Montpelier Shale	28.9	76.3	31.1	59.7

grinding should be done as rapidly as possible and probably means that a quick-discharge mill should be used.

(2) The results on the Montpelier shale are decidedly inferior to those on the Fort Hall shale. This appears to result from the fact that the oolites are smaller in the Montpelier shale; consequently the slime assays are higher and the recovery in the sands is lower. Figure 7 shows screen analyses of composite samples of three of the sands from these rock mill tests; note that the Montpelier shale shows the smallest particle size and that the sand grains from the high-grade rock are much coarser than those from either of the shales.

(3) The grade of concentrate that can be made from these shales depends upon where the "cut" is made in desliming the material. By taking a coarser product off in the slime it is possible to improve the grade of the sand; this, of course, makes a corresponding decrease in recovery as the data in Table 4 show.

(4) In the case of the Fort Hall shale it is possible to produce a sand containing 32.3 per cent P_2O_5 (70.5% BPL) with a recovery of 79.5 per cent of the total P_2O_5. With the Montpelier shale, "desliming" at 325 mesh yielded a sand containing only 31.1 per cent P_2O_5 (67.3% BPL) with a recovery somewhat under 60 per cent. The grade of this sand from the Montpelier rock could be raised by cutting out coarser material (see Table 1) but this would mean a further sacrifice of recovery.

(5) The 5-cycle test on the sample of high-grade (commercial) rock was made to see if it would be possible to improve the grade of the commercial rock by grinding and desliming. The test showed that this could be accomplished with a high recovery. The oolites in the commercial rock are larger than those in the shales, and the "point of separation" could have been made at about 10 mesh instead of 20 mesh without lowering the grade of the sand produced; this would have increased both the recovery and the grinding capacity of the mill.

(6) It is of interest to compare the grinding capacities of the four tests shown in Table 3. Taken in the same order, the capacities are:

Sample	Weight of Finished Product
Fort Hall Shale	3.41 pounds per hour
Fort Hall Shale	8.00 pounds per hour
Fort Hall High-Grade	11.68 pounds per hour
Montpelier Shale	4.19 pounds per hour

The second test showed greater capacity than the first test because there was more rock in the mill and because the separation of the "finished product" was made at a coarser size. The fourth test (on the Montpelier shale) shows only about half the capacity of the second test; this appears to be due primarily to the finer size of the oolites in the Montpelier shale. Because of this the rocks are smoother and do not wear down as rapidly by attrition. The Fort Hall high-grade, which contains the largest oolites, also shows the largest grinding capacity.

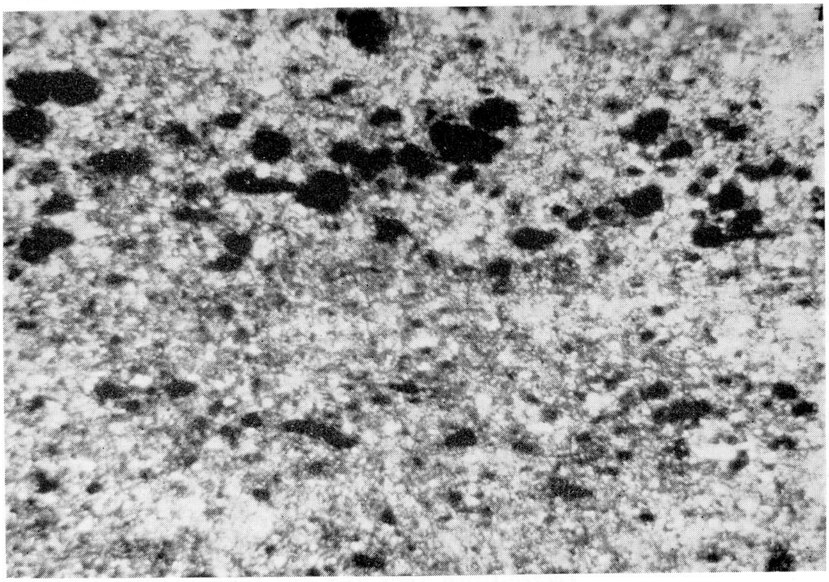

Figure 5—(x50) Fort Hall Shale 3% P_2O_5

ROD MILL GRINDING

This series of tests was designed to study the effect of desliming a product ground in a rod mill.

MILL USED. The mill used was a laboratory rod mill 9 inches in diameter and 12 inches long. The rod charge consisted of 35 steel rods ⅞ inch in diameter and 12 inches long. Mill speed was 59 RPM which is about 65 per cent of the critical speed.

THE CHARGE. Only the Fort Hall shale was used in the rod mill tests. A large sample of this shale was crushed in a cone crusher to pass a 10 mesh screen, and this was used as the rod mill feed. The grinding was done in a pulp with a liquid—solid ratio of 1:1 and 1470 grams of dry rock constituted a charge. This gave enough pulp to just fill the void spaces in the rod charge.

BATCH TESTS. A series of batch tests were made for varying time intervals. The ground product was deslimed in the same manner as the rock mill products. Results of these tests are summarized in Table 5.

Table 5

Results of Batch Tests with Rod Mill

Feed: —10 Mesh Fort Hall Shale

Time of Grind, Minutes	Calculated Head Assay % P_2O_5	Assay of Sand % P_2O_5	Assay of Slime % P_2O_5	Recovery of P_2O_5 in Sand %
1	26.0	28.79	12.10	91.4
2	26.5	29.65	12.98	90.1
3	26.2	29.46	15.01	87.0
5	22.6	26.96	15.65	74.2
10	23.2	27.80	17.66	65.6
15	21.6	27.57	19.22	37.4

The results of these batch tests indicate that it is a very easy matter to overgrind this rock. From the standpoint of grade of concentrate and recovery the 2-minute grind gives about the best results. For longer grinding periods both the grade of concentrate and the recovery decrease steadily.

LOCKED-GRIND TESTS. Two locked-grind tests were made in the rod mill using the same charge as in the batch tests. At the end of each cycle the "finished product" was removed by screening and replaced by an equal amount of new feed. The results are given in Table 6.

Table 6

Locked-Grind Tests with Rod Mill

Feed: —10 Mesh Fort Hall Shale

Calc. Head Assay % P_2O_5	No. of Cycles	Time of Grind Per Cycle Minutes	Point of Separation of Finished Product	Assay of Sand % P_2O_5	Assay of Slime % P_2O_5	Recovery of P_2O_5 in Sand %
24.6	5	5	100 mesh	29.9	15.7	76.6
25.0	6	2	65 mesh	29.0	13.2	85.4

The second of these locked-grind tests shows a higher recovery and lower slime assay although the assay of the sand is slightly lower in the second case. These effects are the results of using shorter grinding periods and a coarser separation of the finished product.

If we compare the results shown in Tables 5 and 6 with the results given in Table 3 for the Fort Hall shales it can be seen that there is no significant difference. For the particular conditions that were investigated the results of rock milling are slightly superior, but it is quite likely that if somewhat different choices of time of grind and point of separation were made in the rod mill grinding the results would be as good as those of rock milling. Also, the grade of the sands produced in rod milling could be improved by desliming at a coarser size (as in Table 4 for the rock milling tests).

DRY GRINDING AND AIR CLASSIFICATION

A series of tests were made to see if it would be possible to "deslime" dry ground material by air classification. A sample of the —10 mesh Fort Hall shale was dry ground in the rod mill until it all passed 48 mesh, and samples of this dry-ground rock were then air classified in a column to remove the dust or "slime."

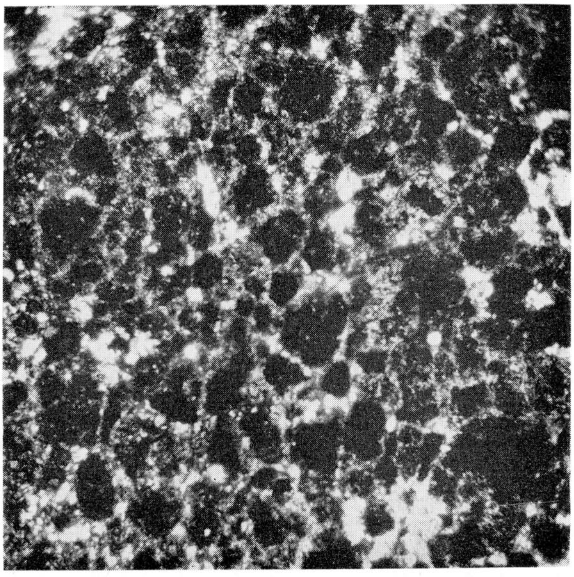

Figure 6—(x50) Montpelier Shale

AIR CLASSIFIER. The classifying column used in these tests was designed after an air analyzer used by the U. S. Bureau of Standards.* This consisted of a pipe 60 inches long and 2½ inches

*Pearson, J. C., and W. H. Sligh: An Air Analyzer for Determining the Fineness of Cement; U. S. Bureau of Standards Technologic Paper No. 48, 1915.

in diameter. The pipe was suspended vertically and a bulb was attached to the bottom of the pipe to hold the sample. The stream of air entered the bulb through a nozzle and was directed downward upon the sample; the air agitated the dust and sand and the fine dust was carried away by the rising current of air in the tube. A sample of about 45 grams was used and the blowing was continued until the sand remaining in the bulb came to constant weight. Weights and assays of the dust were determined by difference. Results of several of these tests are given in Table 7.

Table 7

Results of Dry Grinding and Air Separation

Test No.	Head Assay % P_2O_5	Air Pressure Psi	Nozzle Diameter mm.	Assay of Sand % P_2O_5	Assay of Dust % P_2O_5	Recovery of P_2O_5 in Sand %
3	25.6	1.0	2.00	31.28	12.47	85.4
5	28.4	1.0	2.00	31.21	12.44	86.7
6	25.0	1.2	1.00	27.59	11.42	92.7
7	24.4	1.2	1.75	30.92	12.64	81.6
8	25.2	1.2	2.75	31.80	15.50	74.3

Test 5 (Table 7) is the same as Test 3 except that the sample used in Test 5 was oven-dried for three days. This made practically no difference in the results.

The results of dry grinding and air separation are quite good and they compare very favorably with the results obtained by the wet grinding and desliming tests. Note that it is possible to prepare sand containing over 31 per cent P_2O_5 (67.7% BPL) with recoveries of over 85 per cent. As the air velocity is decreased (Test 6) the recovery increases but the grade of the sand decreases; the opposite results are obtained when the air velocity is increased (Test 8).

GRINDING WITH RUBBER-COVERED RODS

It was decided to investigate the effect of grinding with rubber-covered rods which might be expected to provide a gentler grinding action than bare steel rods. The same rod mill employed in the previous test was used, but the grinding media consisted of 24 steel rods ½ inch in diameter by 12 inches long; these rods were covered by stretching rubber tubing over them. The mill was operated at 74 RPM, and, as before, the amount of material charged was enough 1:1 pulp to fill the void spaces in the rod charge. The —10 mesh sample of Fort Hall shale was used for mill feed.

Four batch tests were made with the rubber-covered rods, and four tests were also made with the bare steel rods (the same rods with the rubber coverings removed). The ground rock was deslimed by decantation as in previous tests. Results are in Table 8.

Table 8

Results Obtained by Grinding with Rubber-Covered Rods and Bare Steel Rods

Feed: —10 Mesh Fort Hall Shale

Head Assay, 24.6% P_2O_5; Pulp Ratio, 1:1

Rods Used	Time of Grind, Minutes	Assay of Sand % P_2O_5	Assay of Slime % P_2O_5	Recovery of of P_2O_5 in Sand, %
Rubber-covered	3	29.2	10.9	89.4
Rubber-covered	6	29.6	11.1	87.4
Rubber-covered	10	30.2	12.7	82.6
Rubber-covered	15	29.9	16.5	76.2
Bare Steel	3	30.3	11.4	87.2
Bare Steel	6	30.2	13.6	82.7
Bare Steel	10	29.6	16.7	75.0
Bare Steel	15	29.9	18.1	66.4

The results obtained by grinding with rubber-covered rods indicated that there was no particular advantage to be gained by this method; about the only difference to be noted is that the rubber-covered rods grind more slowly. The results of the 3-minute grind with bare steel rods are almost identical with the results obtained in a 6-minute grind with rubber-covered rods. Likewise the 10-minute grind with bare steel rods gives practically the same values as the 15-minute grind using the rubber covered rods (Table 8).

WILFLEY TABLE TESTS

Four tests were made on a small laboratory Wilfley table with a 12-inch by 30-inch deck. The results are given in Table 9.

Table 9

Results of Wilfley Table Tests

Feed—Fort Hall Shale; Head Assay—25.0% P_2O_5

Feed Size	Product	Assay % P_2O_5	Recovery of P_2O_5 %	Remarks
—10 Mesh	Concentrate	29.70	52.25	
	Middling	22.35	44.20	
	Tailing	14.03	3.62	
—10 Mesh	Concentrate	29.32	61.70	Table Products Deslimed
	Middling	21.60	34.80	
	Tailing	13.98	3.51	
—48 Mesh	Concentrate	31.42	69.20	
	Middling	18.23	24.55	
	Tailing	14.10	6.30	
—48 Mesh	Concentrate	32.48	37.60	Table Feed Deslimed
	Middling	28.95	60.00	
	Tailing	21.98	2.40	

There is a small difference in specific gravity between the high-grade and low-grade portions of these shales; the high-grade has a specific gravity of about 2.93 and the low-grade about 2.75.

The table tests were made primarily to see if gravity concentration would produce a higher grade product than desliming. The results of these tests (Table 9) indicated that a concentration could be made, but neither the grade of concentrate nor the recovery was high enough to encourage further study of table concentration.

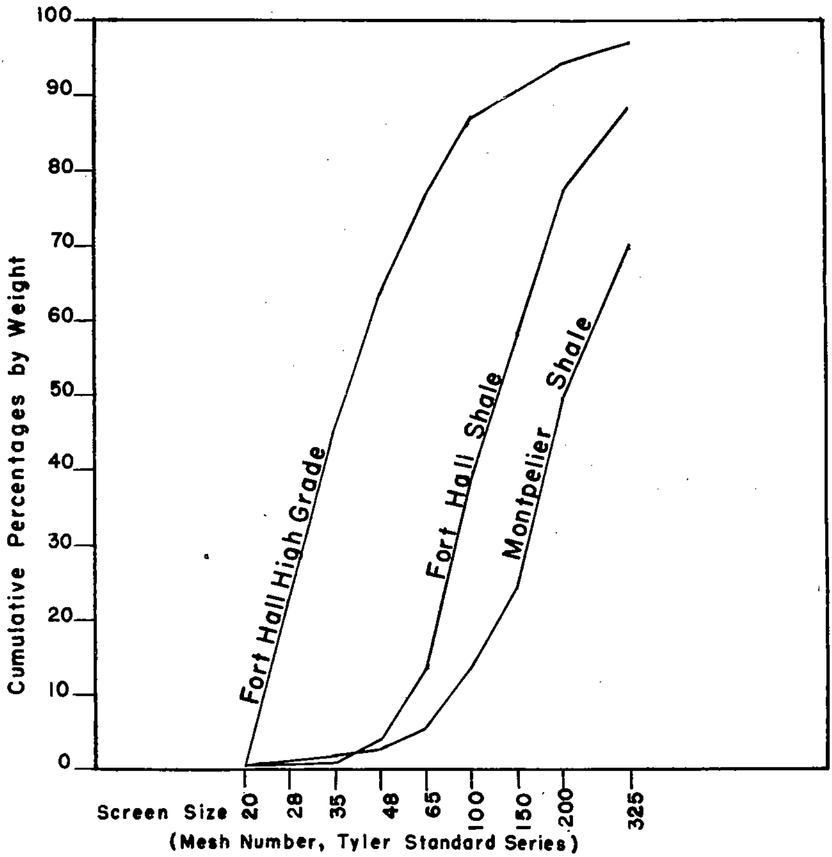

Figure 7—Screen Analyses —20 Mesh Sands

SUMMARY OF EXPERIMENTAL WORK

1. The phosphatic shales consist of black oolites of high-grade phosphate mineral cemented in a matrix of lower-grade, chalky material.

2. When these rocks are ground, the low-grade portion tends to form a brown slime; the phosphate oolites tend to remain as a black, high-grade sand.

3. The shales can be concentrated by a combination of selective grinding and desliming.

4. Various methods of grinding were investigated and, of course, there are many other methods that might work equally well. The principal requirement of the grinding method is that it shall promote the disintegration ("sliming") of the low-grade matrix and avoid overgrinding the phosphate oolites as far as possible.

5. The "desliming" can be done either with water or with air; therefore the grinding may be either wet or dry grinding.

6. In general, the grade (P_2O_5 content) of any ground product can be improved by making a "coarser cut" when removing the slime. This, of course, lowers the recovery of P_2O_5. Within limits the operator should be able to make any grade of concentrate desired (up to about 32 per cent P_2O_5 or 70 per cent BPL) by making the requisite sacrifice in recovery.

SUITABILITY OF THE PRODUCT FOR ACIDULATION

Because the primary purpose of this study was to investigate the possibility of making a product suitable for direct acidulation, it is necessary to consider the suitability of these products for this purpose.

In the present method of manufacturing superphosphate the high-grade rock is usually ground dry and then mixed with the proper amount of diluted sulfuric acid. The acid is diluted about 1:1 with water.

For acidulation the rock should be ground to 90-95 per cent —100 mesh and 72-76 per cent —200 mesh. These requirements would make it necessary to employ two stages of grinding if the method of grinding and desliming was used for concentration. For the best recovery and grade of concentrate the initial grinding should be so conducted that the oolites are liberated at their "natural grain size" as nearly as possible. When such a product is deslimed the resulting concentrate (sand) is too coarse for direct acidulation and would have to be reground.

If the concentrating method involved some type of dry grinding and air classification, the concentrate produced would be dry and fine grinding and acidulation could proceed by the same method that is now used. However, if wet grinding and water classification were used it would be necessary to dry the material or use another method of acidulation.

It might be possible to acidulate wet rock with strong (undiluted) sulfuric acid provided that the rock did not contain too much moisture; for this method the maximum moisture content that could be permitted would be about 16 per cent.

The sands produced by grinding and desliming settle very rapidly. Simple decantation of the water from a pan usually leaves less than 30 per cent moisture, and a filter would readily bring this down to about 10 per cent. Of course the moisture content and ease of filtration of these coarse sands is not the important consideration—

what is of interest is the moisture content that could be obtained in a reground product ready for acidulation.

The regrinding would have to be done either wet or dry; it would not be possible to grind a coarse sand containing 10 or 15 per cent moisture and have the ground product contain the same percentage. Either the sand would have to be completely dried (for dry grinding) or more water would have to be used (in wet grinding).

In order to determine what could be expected of this finely-ground material, a sample of concentrate (sand) was reground for 15 minutes and then filtered on a small pressure filter. The material filtered readily and the filter cake contained only 13.6 per cent moisture. This particular sample contained 68.6 per cent of —200 mesh which means that it was somewhat coarser than the size desired for acidulation (72-76% —200 mesh), but the experiment indicates that it should not be a difficult matter to dewater these reground sands sufficiently for direct acidulation (below 16% moisture content).

SUMMARY

Following are brief discussions of some of the facts brought out by the experimental work, and of some of the factors that would have to be considered in commercial concentration of these phosphatic shales.

APPLICATION. A successful method of concentrating the low grade phosphatic shales would have two principal benefits. In the first place it would increase the phosphate reserve—possibly it would multiply the estimated reserve tonnage by a factor of 3 or more. Of more immediate interest is the fact that it might be of direct economic benefit to those who are now mining phosphate rock. If it is necessary for the operator to mine large quantities of shale in order to get at the high-grade commercial rock then it might be advantageous to be able to recover a portion of the P_2O_5 in the shale in the form of a high-grade concentrate.

To take a concrete example, suppose that in a particular deposit there is a 6-foot bed of high-grade with a 20-foot bed of shale above it. If the shale contains 24 per cent P_2O_5 and it is possible to make an acceptable concentrate with 80 per cent recovery, then this concentrate produced would be the equivalent of a 12-foot bed of high-grade.

COST. Any method involving only grinding and desliming should be quite low in cost. Rough estimates on the capacity of the laboratory rock mill indicated that the treatment cost should be on the order of 10 cents per ton, and it is possible that other methods of grinding might be even less expensive.

Two locked-grind tests were made with a small ball mill to determine the relative grindability of phosphatic shale and quartz. Both samples were —10 mesh and the grindability was expressed as the amount of —100 mesh material made per minute. On this basis the grindability of phosphate shale was 2.3 times that of the quartz rock, and this is probably on the conservative side because grinding through 100 mesh means that some of the harder oolites were being ground up.

One of the most attractive features of concentration by simple grinding and desliming is the potential low cost of the process. The value contained in the rocks is low, and the expensive and complicated treatment methods would not be warranted.

CHOICE OF METHOD. Several alternative methods of grinding and desliming have been investigated, and there are others that might be equally effective. The costs should be quite low for almost any method chosen, and it would be difficult to make a choice until larger-scale tests had been made.

Desliming might be done by either air or water classification depending upon whether dry or wet grinding is used. Several factors would have to be considered in making this choice—relative costs of the two methods, disposal of the tailing (dust or slime), method used for regrinding the sand concentrate, and the removal of moisture from a wet-ground product. In either case it would also be necessary to decide where the "cut" should be made in desliming—how much recovery should be sacrificed in order to improve the grade of the concentrate.

ROCK MILLING. The method of rock milling would be cheap because it would not be necessary to purchase any grinding media and the power required to operate a rock mill would only be about half that required for a ball mill of the same size. It is quite likely that a good part of the rock should be crushed to about ¼-inch diameter before feeding it to the rock mill because the larger rocks wear away quite slowly.

ROD MILLING. Although a rock mill would have a reasonably large capacity on grinding these shales, it is almost certain that a rod mill (or ball mill) would have several times the capacity of a rock mill of equal size. Also the cost of grinding would be low—probably lower than the cost of rock milling.

It must be remembered, however, that this preliminary grinding is essentially a concentrating operation and must have a selective action. The experiments show that it is very easy to overgrind these shales in a rod mill unless the rock can be circulated through the mill very rapidly. In selecting a method of grinding, therefore, two factors must be considered—the selective action and the grinding capacity.

DESLIMING. The low-grade "slimes" or dust can be removed either by air or water classification. Air classification would require that the rock be ground dry, and it would present a problem in dust recovery and disposal. The principal virtue of dry grinding and air classification is that it would produce a dry sand concentrate.

Larger-scale tests would be required to determine the relative costs of wet- and dry-grinding methods.

TAILING DISPOSAL. The disposal of the tailing (slime or dust) would be an important problem in any case, and this might well be the factor that would decide which method should be employed.

APPLICATION TO OTHER DEPOSITS. Most of the work was done on the Fort Hall shale, and, of course, the application of these methods to other shales would depend upon how nearly their structure resembled the Fort Hall shale. A few tests were made on a shale from Montpelier, and these results were definitely inferior because the Montpelier shale contains finer oolites than the Fort Hall shale and it is not possible to make as good a recovery. Laboratory tests would have to be made on the shale from any particular region to determine its characteristics.

The samples investigated all contained between 24 and 26 per cent P_2O_5 (52 to 57% BPL), but there are other shale deposits that have a lower average grade. Norris* states that at Conda the phosphate shales are 160 feet thick and average about 12 per cent P_2O_5 (26% BPL) over their entire thickness.

It is probable that these lower-grade shales could be treated economically if their structure resembles that of the Fort Hall and Montpelier shales. There are two possibilities that should be considered in this connection.

1. In some cases the shales contain beds that are relatively barren (1 to 8 per cent P_2O_5) and these might be segregated from the higher-grade shales by selective mining.
2. It is possible that some of the low-grade material could be removed by a rock-milling process. Note (Table 3) that in the second test on Fort Hall shale the residue remaining in the mill after 11 cycles contained only 13.5 per cent P_2O_5. In this case the lower grade material appears to be the most resistant to grinding, and where this is true the rock-milling method would permit the separation of a portion of the low-grade material as large pebbles or boulders.

ACKNOWLEDGMENTS

To A. W. Fahrenwald, Dean of the School of Mines and Director of the Idaho Bureau of Mines and Geology, who suggested the problem in 1938, and who directed the work here reported; to Dr. L. C. Cady, Executive Secretary of the University Research Council; to Professor W. W. Staley, who assisted in the field; to Mr. Harry Marsh, Secretary of the Idaho Mining Association for helpful assistance; to the Simplot Fertilizer Company and their staff; and to the San Francisco Chemical Company, the authors wish to express their thanks for valuable assistance and cooperation.

Especial credit is due the junior author of this report, who competently performed most of the experimental work and from whose master's thesis much of the data for this report were taken.

*Norris, E. M.: Underground Mining of Phosphate Rock at Conda, Idaho; Mining and Metallurgy, October, 1944.