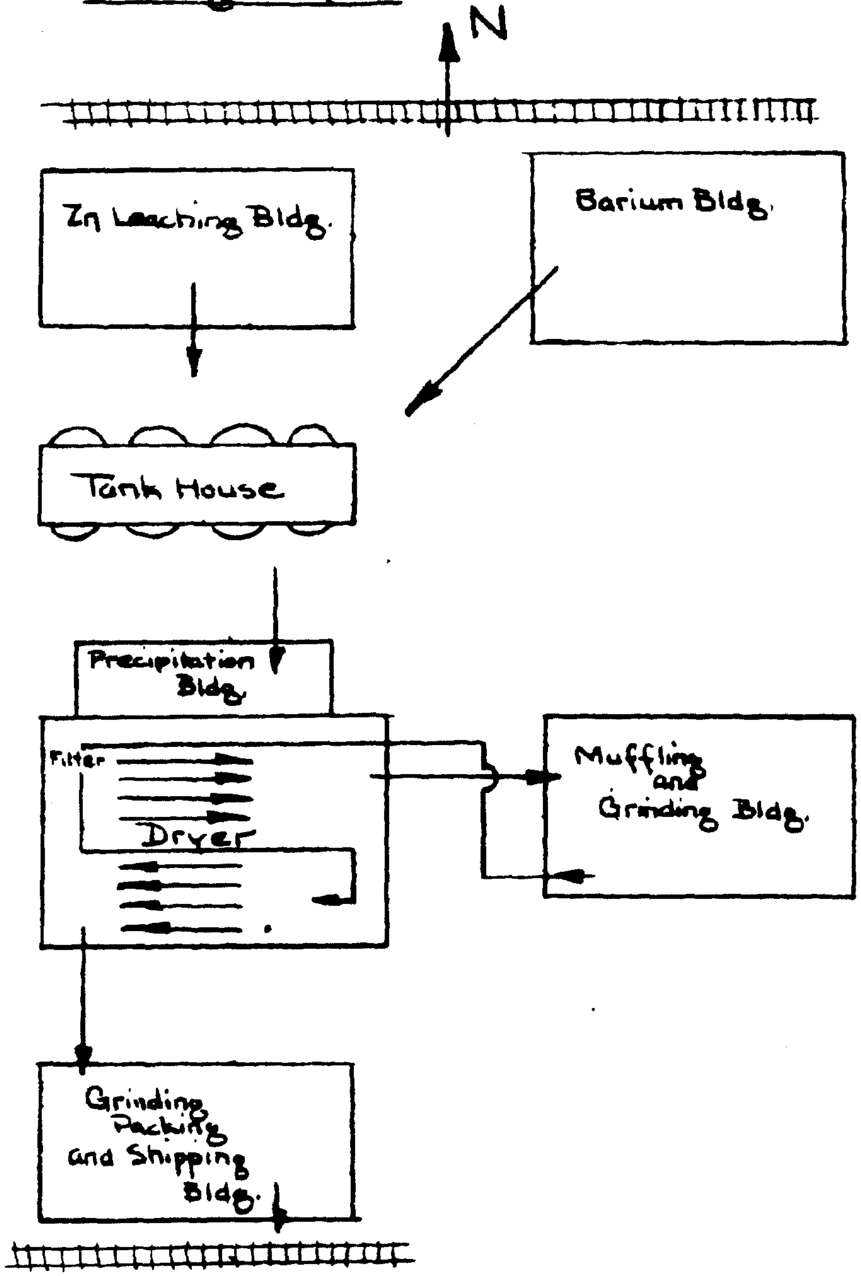
Sheet A
Nov. 20, 1997

LITHOPONE DEP'T

Building Layout



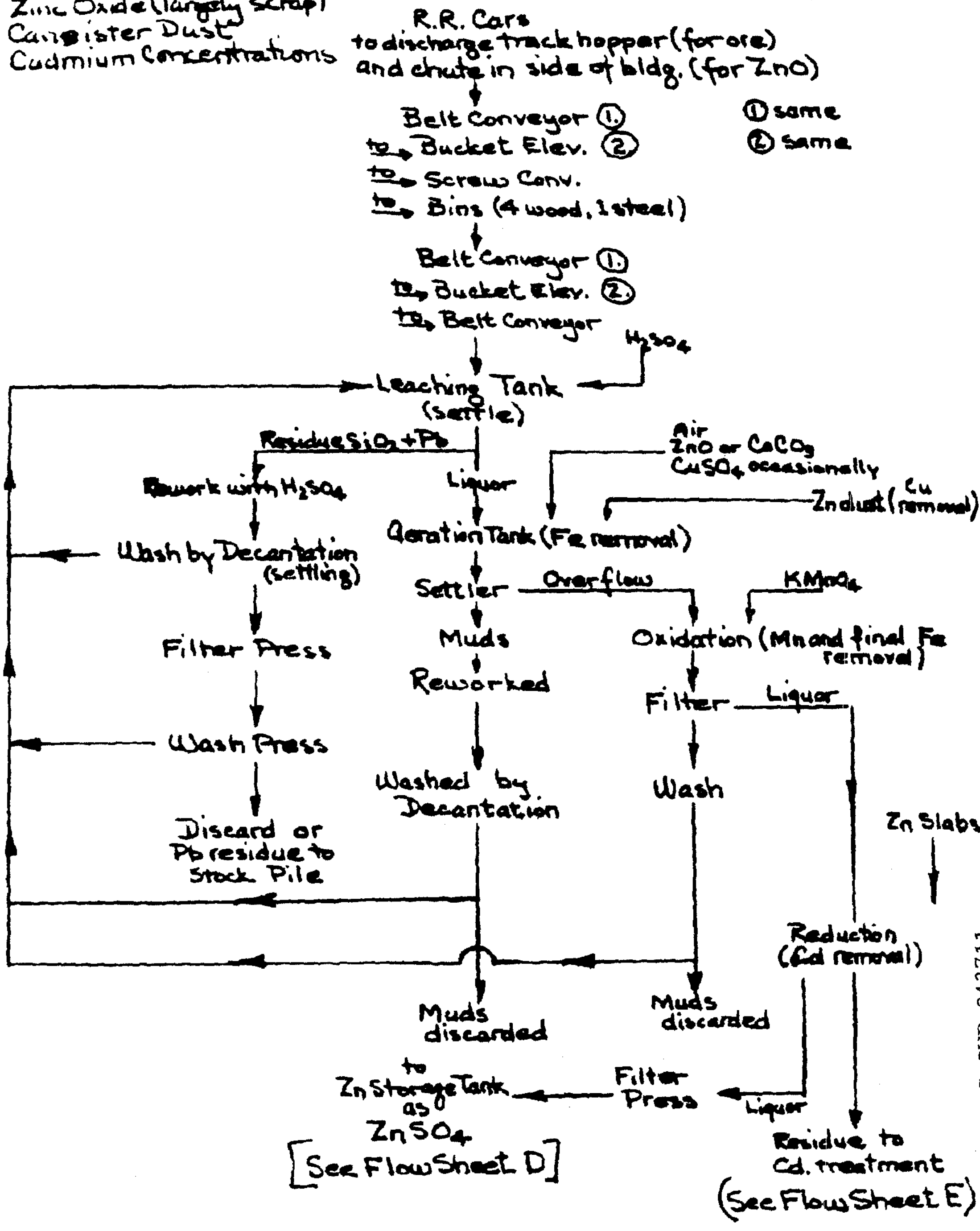
0007-SWP-043710

N23238

LITHOPONE DEP'T
Zinc Leaching and Purification

Sheet B
Nov. 20, 1952

Raw Materials:
- Roasted Hanover Ores
- Zinc Oxide (largely scrap)
- Canister Dust
- Cadmium Concentrations

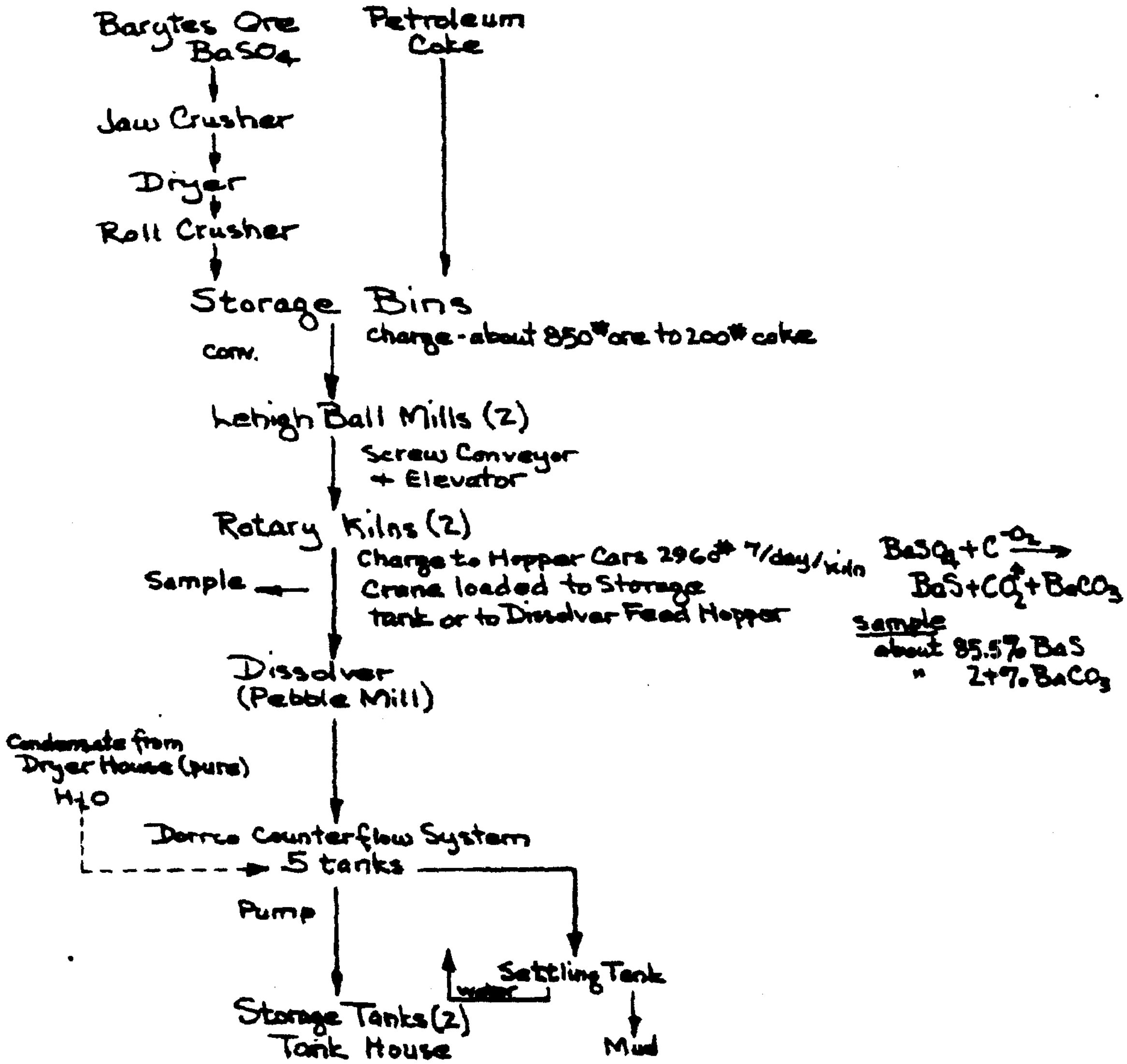


0007-SWP-043711

LITHOPONE DEP'T

Barium Bldg.

Sheet C
Nov. 21, 1952



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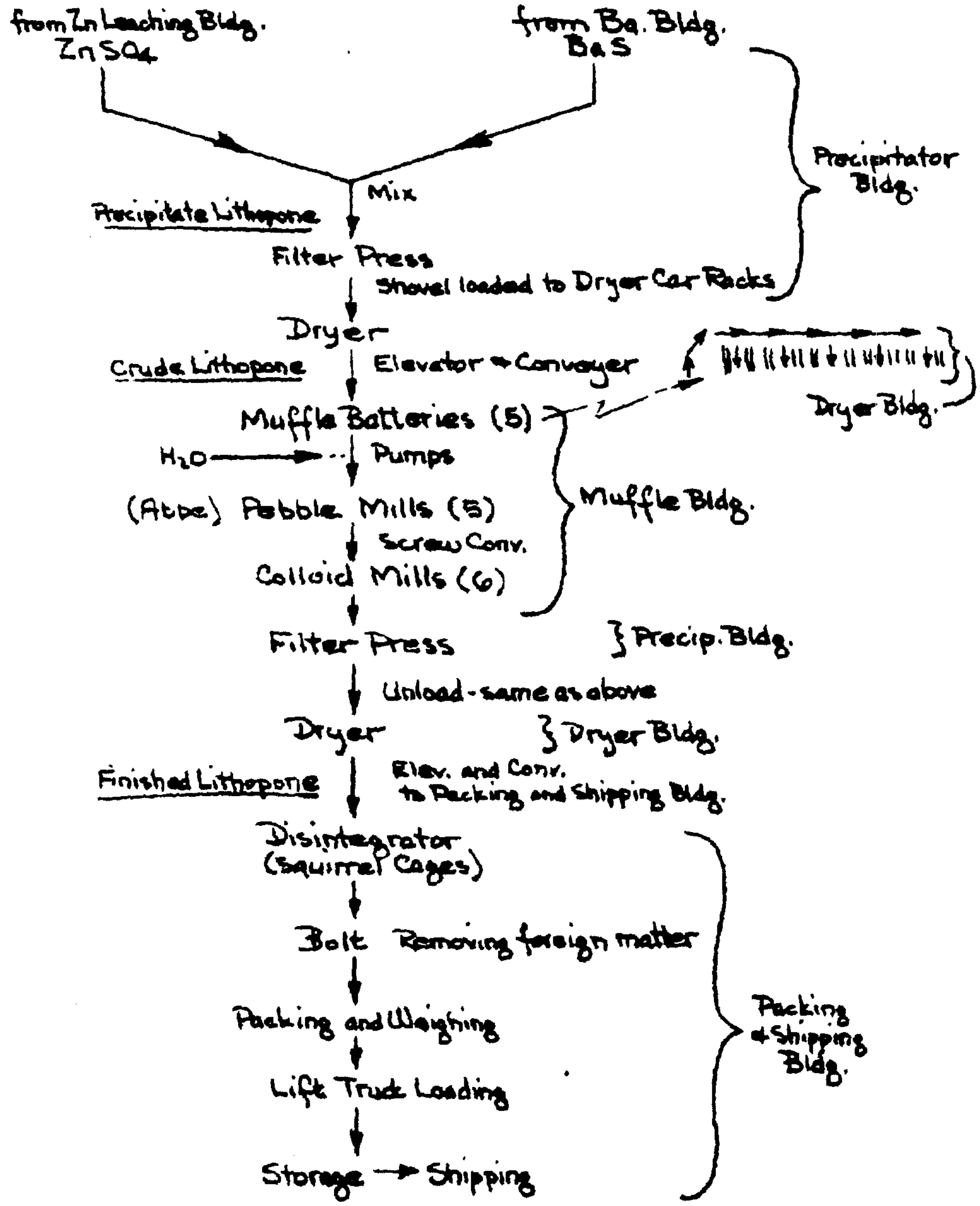
LITHOPONE DEP'T

Sheet D
Nov. 21, 1952

Finish Dep't.



LITHOPONE PRECIPITATION

SULPHUR ADDITION TABLE

All Grades, except 362 base
(Revised 10-24-'52)

Press Filtrate cc N/10 I. 25 cc Sample	Pounds Sulphur per 5 tons Lithopone: 1 x	2 x	3 x	4 x	5 x	6 x
7.6 - 8.0	5	5	5	5	5	5
7.1 - 7.5	5	5	5	5	5	5
6.6 - 7.0	5	5	5	5	5	5
6.1 - 6.5	5	5	5	5	5	5
5.6 - 6.0	5	5	5	5	5	6
5.1 - 5.5	5	5	5	5	7	8
4.6 - 5.0	5	5	5	7	8	10
4.1 - 4.5	5	5	6	8	10	12
3.6 - 4.0	5	5	7	9	11	14
3.1 - 3.5	5	5	8	10	13	16
2.6 - 3.0	5	7	11	15	18	22
2.1 - 2.5	5	9	14	19	23	28
1.6 - 2.0	5	13	20	27	33	40
1.1 - 1.5	7	21	32	43	53	64
0.6 - 1.0	11	33	50	67	83	100
0 - 0.5	24	48	72	96	120	144

The sulphur addition to the precipitate is based on the relation of the SH content of the crude press filtrate and also on the ZnO content of the finished product.

Normal use 1 x table for Albalith 10 and 90 grades.

Normal use 3 x table for 11, 13, 14, 73, 806 and 807.

The sulphur table used will vary with the increase or decrease in the desired ZnO content of the finished material.

CWO-7-27-56

0007-SWP-043714

0007-SWP-000127064

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Mineral Products Laboratory

October 25, 1955

447 - LITHOPONE, GENERAL RESEARCH

SPECIAL REPORT

GRINDING OF LITHOPONE IN A HOCHBERG MILL

Summary:

Preliminary tests have demonstrated that "quench box" lithopone (discharge from calciner) can be ground in water slurry by a Hochberg Mill. Limited tests did not indicate that this technique will produce a lithopone of superior texture; however, there do appear to be advantages in cost and size of equipment. Considerably more work needs to be done to establish optimum processing conditions and proper evaluation of product with respect to color degradation, texture, opacity, etc.

Description of Mill:

The mill used in the work under discussion was a laboratory unit designed by the Dispersion Laboratory after the principles of the Hochberg patent U.S. 2,581,414. The work likewise was done with the assistance and co-operation of the staff of the Dispersion Laboratory.

The grinding chamber of the mill consists of a vertical cylinder, 4 inches in diameter and 15 inches deep. The lowest 2 inches of the cylinder wall consists of a removable 40 mesh screen; the bottom is solid. Projecting down through the center of the cylinder and extending nearly to the bottom is a motor-driven shaft carrying five evenly spaced disks or rotors. These rotors are 3 inches in diameter, 1/2 inch thick, and 2 inches apart. The first rotor extends to within one inch of the bottom of the chamber and the last to within 3 1/2 inches of the top.

In operation, the mill is loaded about 3/4 capacity with Ottawa Sand, minus 20 plus 30 mesh. The rotors are driven at 3450 RPM, or 2700 ft./min. peripheral speed, and the grinding is accomplished by the motion of the sand grains. Unground slurry is fed in continuously at the top of the mill and discharged out through the screen at the base of the chamber.

The grinding chamber is so mounted that the lower third is contained in a closed receiving chamber. The receiving chamber is fitted with a sloping bottom for good drainage and is discharged through a pipe and gate valve. Inasmuch as the screen area is fixed in this mill, the retention time in the mill, and hence the amount of grinding, is controlled by regulating the discharge rate through the valve.

The following size and capacity figures for the mill (2 inch free-board) are pertinent:

	Cu. In.	Quarts
Total Volume	163	2.82
Working Volume (rotors inserted)	138	2.39
Apparent Volume of Sand Charge	115	2.0
Solid Volume of Sand	73	1.25
Volume of Slurry	65	1.14

Experimental Work:

The particular in-process material with which this work is concerned is "quench-box" lithopone, or calciner discharge product. The sample submitted, AURL 3613, was taken from regular production. This material is relatively coarse; much of the aggregate being in excess of 40 mesh.

All of the grinding trials were run at a 30% solids concentration. As a basis for comparison, a portion of the sample was ground in a laboratory pebble mill.

Capacity of Mill	1.00 gal.
Volume Pebbles (apparent)	0.50
Volume Slurry	0.29
Total Volume	0.60
Weight Slurry	1432
Weight Lithopone (dry)	429

The progress of the milling was followed by selecting small samples at periodic intervals and checking for grind and residue.

Time	Hegman Grind*	325 Mesh Residue
1/2 hr.	$\frac{2\ 1/4}{0}$	6.4%
1	$\frac{3\ 1/8}{2}$	0.80
1 1/2	4	0.17
2	5	0.03
3	5 3/4	Less Than 0.01
4	6 1/8	Less Than 0.01
4 1/2	6 1/2	Less Than 0.01

*Run directly on mill slurry.

$\frac{2\ 1/4}{0}$ indicates dirt to 0 grind.

At the completion of the run, the slurry was de-watered and dried at 110°C. for further evaluation.

In operating the Hochberg Mill, the discharge valve on the receiver was closed and sufficient water added to the milling chamber to bring the level just above the charge of sand. The rotors were then started, feeding begun, and the discharge valve opened just sufficiently to maintain the working level in the mill. For several reasons, it was desirable to use a feed of 30% solids concentration on all runs. Several preliminary trials were made which, while indicative, were not too satisfactory because of the non-uniformity of the feed rate and solids concentration. This problem (which would not be encountered in plant operation) was due to the rapidity with which the very coarse solids settled out of suspension, together with the physical arrangement for feeding the mill.

With the exception of the last run, one gallon (4900 grams) of 30% slurry was used in all the trials. The method of feeding the mill was to add the wet lithopone "sands" and water separately in small increments and in the proper proportions, as nearly as could be estimated. Since the mill was loaded with water at the start of the run, the first discharge was necessarily very dilute; hence about half of the feed was run through before retaining a sample of the product. After certain tests on the wet slurry, a portion was de-watered and dried at 110°C. for special texture tests.

Run No. 1:

The lithopone solids were reduced to 60% concentration to obtain a "pourable" slurry, which was then fed to the mill with additional water as required. There was no discharge of lithopone from the mill and after a short period of time, the feed had to be discontinued because the level in the milling chamber did not recede. The rotors were continued until it was obvious that the discharge screen was blocked and would not be relieved by additional grinding.

Run No. 2:

In this run, 0.2% tetrasodium pyrophosphate (lithopone basis) was added as a dispersing agent. The lithopone solids were added as a wet sludge containing 30% moisture, and the additional water carrying the dispersing agent added separately as before. This time there did not appear to be any hold-up in the lithopone discharge, although the solids concentration of the retained sample was only 23% lithopone. It was noted that there was considerable discoloration, indicating abrasion, during the early part of the run.

Run No. 3:

Another attempt was made to run without a dispersing agent. It was felt that in the first run the solids concentration was too high in the milling chamber, thus blocking the screen.

More care was taken to add sufficient water with the feed. There was a good discharge of lithopone during most of the run, but the mill appeared to block up just at its completion. Discharge ceased and water could not be flushed through until a heavy charge of dispersing agent was added. The fact that a dispersing agent relieved the block strongly indicates that there is a tendency for the lithopone solids to bridge across the screen as a "cake" under certain conditions.

Run No. 4:

This run was basically a repetition of the second run, using pyrophosphate to produce a dispersed system. An attempt was made to increase the feed rate with only indifferent success because of the nature of the feed. While there was a continuous discharge of slurry, the yield of lithopone appeared to be low. Flushing out of the mill further indicated that a considerable build-up had occurred.

Run No. 5:

The behavior of the first four runs indicated a marked tendency for lithopone to build up in the mill and gradually block the discharge screen. However, it was felt that this may have been due to poor feed techniques, which resulted in variable and, at times, excessive solids content and low through-put rates. In order to correct the situation, a quantity of the quench-box material was given a very light pebble milling; 15 minutes at 35% solids. This slurry, then reduced to 30% solids, was still relatively coarse with a grind of 0, but could be readily kept in suspension by occasional stirring.

In this run, no dispersing agent was used and the nature of the feed enabled a uniform charge to the mill at about twice the rate used in the previous trials. Immediately following the slurry charge, flushing water was run through the mill to wash out the bulk of the lithopone retained in the milling and receiving chambers. Accordingly, the effluent was collected as three separate portions; the dilute pre-run effluent, the product effluent, and the wash effluent, as outlined below.

Feed:	Lithopone Slurry (Lithopone, dry	6390 gms. (1.3 gals.) 1900 gms.)
	Flushing Water	2850 gms. (0.75 gals.)
Time to Charge Slurry:		9 min.
Rate:		8.7 gal./hr.
Discharge:		
1. Prerun Effluent		1495 gms. (0.35 gal.)
Solids conc.		14.0%
Lithopone yield		210 gms.
Grind		7 H.

2.	Product Effluent	5055 gms. (1.08 gal.)
	Solids conc.	25.0 %
	Lithopone yield	1265 gms.
	Grind	6 1/4 H. / 4
3.	Wash Effluent	3214 gms. (0.79 gal.)
	Solids conc.	8.6 %
	Lithopone yield	276 gms.
	Grind	7 H / 5 1/2

There did not appear to be any inclination for the mill to choke up in this run. While there was a shortage of 150 gms. of lithopone in the combined effluents, this can be at least partially accounted for by the tendency of the lithopone to settle out in the receiving chamber of the mill.

After evaluation of the separate fractions of effluent, they were combined and a portion of the composite de-watered and dried. Later examination (see results below) indicated that a classification for removal of oversize was necessary. Consequently, a sample of the composite slurry was dispersed with 0.25% tetrasodium pyrophosphate and elutriated through a single chamber of an inverted cone-type soil elutriator. The classified slurry was then flocculated with a small amount of alum, de-watered, and dried at 110°C.

Run	Effluent Slurry		Dry Pigment		
			Hoover Muller Texture		
	Grind	Residue	25	100	400 Rev.
Permolith 40M, Standard	—	—	4	5	5 1/2
Quench Box, Unprocessed	—	—	0	1 1/4 / 0	4 1/2 / 3
Ball Milled, 6 1/2 hours	6 1/2	l.t. 0.01	6	6 1/4	6 1/4
Run #1	No Product				
Run #2, (disp.agent)	6 3/4 / 4	0.059	4 1/4 / 3 1/2	4 1/2	5 1/8
Run #3	0	1.05	2 1/4 / 1	3 1/2 / 2 3/4	5 1/4
Run #4, (disp.agent)	6 3/4 / 0	0.099	3 / 1/2	4 1/2 / 3 1/4	5 1/4
Run #5, composite	6 1/4 / 4	0.20	3 1/2 / 2	3 3/4	6 / 5 1/4
Run #5, classified	7+	l.t. 0.01	6	6 1/4	—

All samples from the runs in the Hochberg mill showed a definite gray discoloration, indicating abrasion in the mill. The product of Run #5 showed the least discoloration. An analysis of this product showed that it had picked up 0.08% Fe; almost certainly from abrasion of the mill by the sand.

Discussion and Conclusions:

There appears to be no question that the Hochberg Mill is capable of grinding quench-box lithopone to satisfactory fineness. It is also apparent that such a process would require a classification system in conjunction with it, just as is used with a ball-milling circuit.

It has not been definitely established whether the Hochberg unit will produce a lithopone of improved texture and dispersion characteristics.

The products from all the runs exhibited considerable color degradation because of mill abrasion. The staff of the Dispersion Laboratory feels that this abrasion could probably be corrected by replacing the present mild steel rotors with a hard alloy. On the other hand, it must be recognized that the Hochberg mill has been primarily intended for dispersion of pigments in oil media. This essentially provides a lubricant that minimizes the abrasive action of the sand, whereas water slurries have little lubricating effect. Hence, non-metals such as ceramics, glass coatings, rubber, or plastics might provide a better solution to this problem.

The important advantages of the Hochberg mill appear to be the small size of equipment required and the low power consumption. If we use Run No. 5 as a basis and assume a direct size factor, a 35 ton per day lithopone unit would require a mill having the following approximate dimensions:

Height	6 1/4 ft.
Diameter	1 1/2 ft.
Capacity	11 1/2 cu.ft. (85 gals.)
Sand Charge	725 lbs.
Rotor Speed	700 RPM. (2700 ft./min.)

This, of course, refers only to the grinding chamber and does not include the receiver and auxiliary equipment.

The conclusions drawn here are only tentative. Additional work must be done to establish optimum conditions and longer runs should be made to obtain definite evaluations, both as to the application of the Hochberg process to the grinding of quench-box lithopone and to the quality of the pigment produced.

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Auxiliaries Mailing List

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