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Stabilizing Agglomerated Slimes for Cyanide Leaching

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THE leaching method that was first widely used with the cyanide process consisted of percolation leaching of crushed ore in vats or leaching tanks. It was frequently necessary to separate the sand for cyanide treatment and discard the slimes, because only granular material allows the necessary amount of solution to percolate through it in a reasonable time. Later a slime process consisting of agitation followed by thickening and filtration was developed, and is extensively used to the present day.

Even though the agitation-leaching slime process is considered satisfactory, the cost of plant and of operation are both greater than for a percolation-leaching plant of equal capacity. This situation has been recognized in the hydrometallurgical treatment of copper-bearing slimes; and a considerable amount of work has been done at the Southwest Experiment Station of the U. S. Bureau of Mines in an effort to develop a percolation method of treating slimes. Experimental work on the copper-leaching problem was started by H. E. Keyes¹, and later tests were made by J. D. Sullivan and A. P. Towne². The Bureau of Mines work resulted in the development of a process by which slimes were moisture-agglomerated with sand and crushed ore, to form a permeable mass through which solution could be trickled without breaking down the glomerules. The agglomeration trickle-leaching method was developed on a small scale and finally tried out in a 12-ton vat. Successful results were obtained as long as the leach solution was added slowly, so that the agglomerated charge was never flooded. If the leach solution flooded the charge, the glomerules broke down and the charge became impervious.

There are two important difficulties in the application of the agglomeration trickle-leaching method to the cyanide process for gold and silver ores:

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1. Moisture-agglomerated particles are held together by the surface tension of the film of water around them. Whenever more solution is added than will trickle down in a thin film, the surface forces no longer hold the particles together and the porous glomerules slump to a runny mud. In this condition many finely divided ores are practically impervious to leach solutions. Consequently, in order to prevent flooding and the resulting loss of a leaching-tank charge, the rate of adding solution must be controlled with such extreme care that the process appears impractical.

2. The moisture-agglomeration trickle-leaching process requires the incorporation of a considerable amount of sandy and granular material in the glomerules in order to give them sufficient stability. In the Bureau of Mines 12-ton test, the mixture agglomerated contained only 27 per cent of minus 200-mesh material and 32 per cent was larger than 10 mesh. Only very rarely will gold and silver ores yield a good extraction when such comparatively coarse rock is leached.

This paper describes the development of a method of stabilizing agglomerated slimes by the formation of a cementing substance in the glomerules. When glomerules are stabilized in this way the necessity for incorporating coarse material with the slimes is eliminated, and the glomerules do not break down when flooded. For leaching with cyanide solutions, a stabilization of agglomerated slimes by the precipitation of calcium carbonate in the glomerules is recommended.

AGGLOMERATION

Two samples of quartz, which had been ground dry in a laboratory ball mill, were used in a preliminary study of moisture agglomeration. The particle-size distribution of the two samples is shown in Table 1.

TABLE 1.—*Screen Analysis of Quartz Samples*

Mesh (Tyler Standard)	Sample No. 1, Per Cent	Sample No. 2, Per Cent
- 42 + 60	8.2	0.0
- 60 + 100	24.6	0.0
-100 + 150	13.4	0.0
-150 + 175	5.6	0.0
-175 + 200	7.8	0.0
-200	40.4	100.0

Neither equipment nor ore sufficient to make large-scale tests was available. Consequently, many of the tests were made in small glass tubes. It was thought that agglomerated material would not pack in small-diameter tubes as it does in large leaching tanks, because of the supporting action of the side walls on the charge. Furthermore, some

means of controlling the amount of packing was desired for the sake of duplication. After numerous endeavors to determine a means of artificial packing that would serve the purpose, vibration was tried, and gave as uniform a compaction as was desired. This was carried out by placing the charged glass tubes, with a rubber stopper in the bottom, on the vibration strip of a Tyler Hum-mer screen.

Moisture agglomeration was obtained by adding the desired amount of solution slowly from a burette to the dry sample in a pan while the sample was stirred and shaken. On a larger scale, Sullivan and Towne obtained satisfactory agglomeration by tumbling the material in a cement mixer during moistening.

In the first test, 90 grams of sample No. 2 was agglomerated with 10 grams of water, charged into a glass tube $1\frac{1}{2}$ in. in diameter on top of a bed of coarse quartz, vibrated for one minute and then compared with 90 grams of the same sample that was dry and not agglomerated. The agglomerated sample occupied about twice the volume of the untreated material. This shows that agglomeration produces a very large increase in porosity.

Porosity itself is not necessarily a measure of permeability, because the voids in the bed may not be interconnecting. Therefore, an apparatus was constructed to measure permeability by means of the rate of flow of air through a bed of the material. By this means the effect of vibration, moisture used in agglomeration, and particle size on the permeability was investigated. The effect of vibration on the permeability of sample No. 1, when agglomerated with different amounts of water, is shown in Fig. 1. The effect on the permeability of the amount of water used in agglomeration with the two samples in which particle-size distribution differed are shown in Fig. 2. In this work the air flow was converted to darcys. One darcy is the permeability that will permit a rate of flow of one centimeter per second through a cross section of one square centimeter, and a length of one centimeter under a pressure differential of one atmosphere with a fluid of one centipoise viscosity.

Conclusions based upon agglomeration tests are as follow:

1. The porosity and permeability of finely ground ores can be greatly increased by moisture agglomeration.
2. Variations in the amount of solution used in agglomeration causes a noticeable difference in the amount of permeability. The maximum permeability is reached between 10 and 18 per cent moisture.
3. A point of saturation occurs when too much solution is added, causing the glomerules to break down to a runny mud. Beyond 22 per cent moisture, none of the materials tested had a measurable permeability by the method used.
4. The amount of solution necessary to give maximum permeability by agglomeration increases with the fineness of the particles.

5. The permeability of loose beds of agglomerated material decreases with packing, but the permeability decrease becomes less as packing progresses.

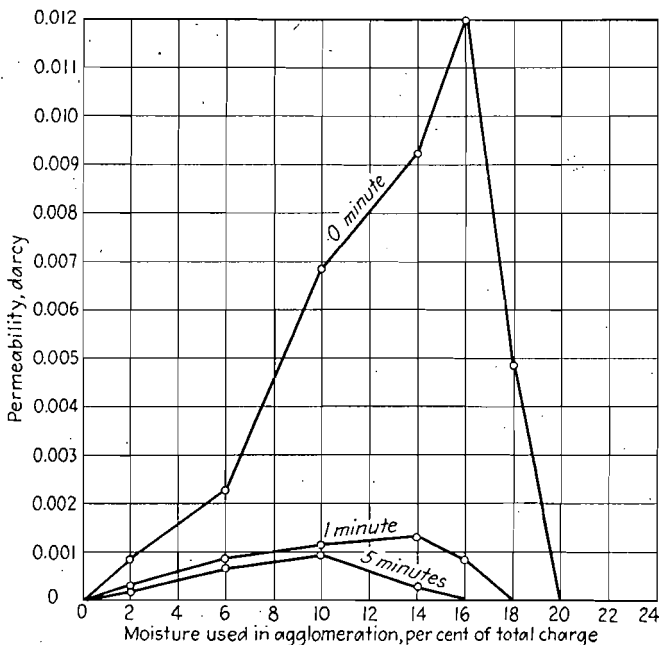


FIG. 1.—PERMEABILITY OF MOISTURE AGGLOMERATED SAMPLE NO. 1. PACKED BY VIBRATION FOR 0, 1 AND 5 MINUTES.

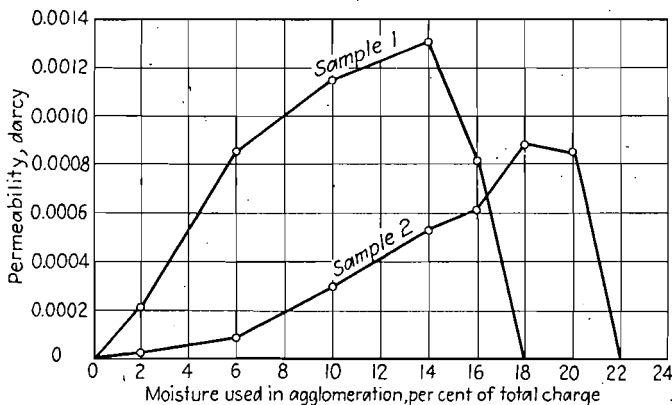


FIG. 2.—PERMEABILITY OF SAMPLES 1 AND 2. MOISTURE AGGLOMERATED AND PACKED BY VIBRATION FOR ONE MINUTE.

STABILITY TESTS

The resistance of agglomerated material to slumping, when wet with solution, is considered to be its stability. The following test was devised in order to measure the stability of agglomerated materials.

A glass tube $1\frac{1}{2}$ in. in diameter was fitted with a screen to support a layer of coarse quartz. The charge, consisting of 100 grams of agglomerated material to be tested, was packed on top of the quartz by a one-minute period of vibration. After packing the charge, a filter paper was placed on top and solution was added slowly until the charge was just covered. Then the charge was allowed to drain. When solution ceased to drip from the ore, the charge was subjected to another minute of vibration. If the ore column was not stable it subsided during the second vibration period. The agglomerated materials, which did not subside enough so that further flow of solution was impossible, were then flooded to a depth of 10 cm. and the rate of passage of solution was measured in seconds per cubic centimeter of efflux from the bottom of the column of material. This value was converted to inches of decrease of solution on top of the charge per hour, and in that form was used to compare the effectiveness of stabilizing treatments.

Series A. Stability Tests with Sample No. 1

Section I. Standard Test. No Means of Stabilization Used.—In order to obtain a standard against which to work, a stability test was made on agglomerated material that had not been stabilized. This material became practically impervious unless solution was added slowly until efflux started to discharge from the bottom. Therefore the first tests were made with carefully controlled solution.

Test 1. Conditions: Sample No. 1 agglomerated with 14 per cent water. No stabilizing agent. Solution added slowly until first efflux. Results: Efflux not clear; showing fines carried by solution. Rate of flow 1.56 in. per hour.

Section II. Lime as a Stabilizing Agent.—Tests were made with lime alone as a stabilizing agent, to determine whether lime, because of its flocculating effect, would increase the stability of glomerules. The desired amount of hydrated lime, containing 70 per cent CaO, was mixed with the sample before moisture agglomeration.

General conditions: Sample No. 1 agglomerated with 14 per cent water. Solution added slowly until first efflux. Stabilizing agent, lime in varied amounts. Results presented in Table 2.

Discussion: The tests with lime show that lime alone has a tendency to increase the stability of moisture-agglomerated quartz particles. The flocculating effect of lime undoubtedly aids the formation of fluffy glomerules during moisture agglomeration.

Section III. Lime and Carbon Dioxide; Lime Varied.—Tests were made to determine the stabilizing effect of calcium carbonate precipitated from lime in the glomerules by the action of carbon dioxide. Hydrated lime containing 70 per cent CaO was added dry before agglomeration.

After moisture agglomeration, charging, and one minute of vibration, carbon dioxide gas was passed through the ore column. The gas was introduced under a pressure of 1 in. of water, and the length of time during which it was introduced was controlled to give the amount desired. For this purpose, the amount of gas that would flow into the agglomerated bed in a given time was determined in a separate test. In the tests of section III a constant amount of carbon dioxide was used and the amount of lime was varied.

General conditions: Sample No. 1 agglomerated with 14 per cent water. Solution added slowly until first efflux. Stabilizing agents, lime and carbon dioxide; lime in varied amounts, carbon dioxide approximately 6 per cent of the charge by weight. Time allowed for CaCO_3 formation and setting before solution added, 3 minutes. Results presented in Table 2.

Discussion: The precipitation of calcium carbonate in the glomerules by the action of lime and carbon dioxide increases the stability of the glomerules. This is shown by: (1) a decrease in subsidence of the bed during the second period of vibration; (2) a lack of fines in the efflux; (3) an increase in the percolation rate; (4) a noticeable rigidity in the mass of agglomerated material removed from the tube after the test.

Increased stability is to be expected with increased amounts of calcium carbonate formed. In these tests a true value of the stability caused by the smaller amounts of lime is not shown, because an excess of carbon dioxide was later found to be harmful.

Section IV. Lime and Carbon Dioxide. Rapid Flooding.—In the previous tests, solution was slowly added to the agglomerated column until efflux started to discharge from the bottom. This was done in order to obtain results in the standard test on glomerules which had not been stabilized. The test in this section was made to find whether glomerules stabilized with calcium carbonate would stand rapid flooding.

Test 9. Conditions: Sample No. 1 agglomerated with 14 per cent water. Rapid flooding with solution. Stabilizing agents, lime and carbon dioxide; lime, 1 per cent; carbon dioxide, 6 per cent. Time allowed before solution added, 3 minutes. Results: Efflux clear. Rate of flow 6.0 in. per hour.

Discussion: The rate of flow in this test with rapid flooding is the same as in test No. 6, in which solution was added slowly until first efflux. This shows that glomerules stabilized with calcium carbonate are strong enough to stand rapid flooding. Rapid flooding was used in all of the following tests.

Section V. Lime and Carbon Dioxide. Water Used in Agglomeration Varied.—Tests were made to find the effect of varying the amount of water used in agglomerating sample No. 1 when lime and carbon dioxide were used to stabilize the glomerules.

General conditions: Sample No. 1 agglomerated with varying amounts of water. Stabilizing agents, lime and carbon dioxide; lime, 1 per cent, carbon dioxide, 6 per cent. Results presented in Table 2.

Discussion: According to the tests in this section, 10 per cent water should be used in agglomerating sample No. 1. In all of the following tests with sample No. 1, the water used in agglomeration amounted to 10 per cent of the charge.

Section VI. Lime and Carbon Dioxide; Carbon Dioxide Varied.—Tests were made to find the correct amount of carbon dioxide to use with 1 per cent lime.

General conditions: Sample No. 1 agglomerated with 10 per cent water. Stabilizing agents, lime and carbon dioxide; lime, 1 per cent, carbon dioxide varied. Time allowed before solution added, 3 min. Results presented in Table 2.

Discussion: About the theoretical amount of carbon dioxide to form calcium carbonate with lime gives the best result. An excess of carbon dioxide appears to reduce the cementing effect of calcium carbonate.

Section VII. Lime and Carbon Dioxide; Time Allowed for CaCO₃ Formation Varied.—In the previous tests with lime and carbon dioxide, a period of 3 min. was allowed between the addition of carbon dioxide and the second flooding. The tests in this section were designed to find the effect of varying this time allowed for formation and setting of the calcium carbonate.

General conditions: Sample No. 1 agglomerated with 10 per cent water. Stabilizing agents, lime and carbon dioxide; lime, 1 per cent, carbon dioxide, 1 per cent. Time allowed for CaCO₃ formation varied. Results presented in Table 2.

Discussion: The formation of calcium carbonate is evidently concurrent with the passage of carbon dioxide. A period of setting is thus unnecessary in order to obtain the maximum benefit from the lime and carbon dioxide used.

Section VIII. Lime and Sodium Carbonate, Preliminary Tests.—It was thought that in some cases it might be more convenient to form calcium carbonate from the reaction between lime and sodium carbonate. This would have the additional advantage of supplying sodium hydroxide for alkalinity in the cyanide process. In these tests hydrated lime containing 70 per cent CaO was mixed into the dry material, which was subsequently agglomerated with a sodium carbonate solution.

General conditions: Sample No. 1 agglomerated with 10 per cent of solution. Stabilizing agents, lime and sodium carbonate. Time allowed for CaCO₃ formation and setting, only that required in manipulation of the test. Results presented in Table 2.

Discussion: These preliminary tests show that effective stabilization of glomerules may be brought about by the use of lime and sodium carbonate.

TABLE 2.—*Stability Tests with Sample No. 1*

Test No.	Variable	Efflux	Rate of Flow. In. per Hr.
SECTION II. TESTS WITH LIME			
Standard	LIME, PER CENT		
	None	Hazy	1.56
2	1	Slightly hazy	2.5
3	2	Slightly hazy	2.7
4	3	Slightly hazy	2.8
SECTION III. TESTS WITH LIME AND CARBON DIOXIDE			
	LIME, PER CENT		
5	0.25	Clear	3.3
6	1	Clear	6.0
7	2	Clear	11.4
8	8	Clear	Too rapid to measure
SECTION V. TESTS WITH LIME AND CARBON DIOXIDE			
	WATER IN AGGLOMERATION, PER CENT		
10	6	Slightly hazy	7.0
11	10	Clear	8.9
12	14	Clear	6.0
13	18	Clear	3.7
SECTION VI. TESTS WITH LIME AND CARBON DIOXIDE			
	CARBON DIOXIDE, PER CENT		
14	0.3	Hazy	2.7
15	1	Clear	9.1
16	3	Clear	9.0
17	6	Clear	8.9
18	13	Clear	7.6
19	26	Clear	6.3
SECTION VII. TESTS WITH LIME AND CARBON DIOXIDE			
	TIME FOR CaCO_3 FORMATION, MIN.		
20	None	Clear	18.1
21	0.5	Clear	11.2
22	1	Clear	10.0
23	6	Clear	9.5
SECTION VIII. TESTS WITH LIME AND SODIUM CARBONATE			
	LIME, PER CENT	SODIUM CARBONATE, PER CENT	
24	0.7	1	Slightly hazy 5.5
25	1	1	Clear 11.0
26	1	0.5	Clear 6.0
SECTION IX. TESTS WITH LIME AND SODIUM CARBONATE			
	TIME FOR CaCO_3 FORMATION, MIN.		
28	10	Clear	9.1
29	30	Clear	13.0
30	45	Clear	26.7

Section IX. Lime and Sodium Carbonate; Time Allowed for CaCO₃ Formation Varied.—It was thought that in order to obtain best results, the formation of cementing calcium carbonate from lime and sodium carbonate would require a period of time. In the tests in this section, varying amounts of time were allowed after agglomeration, which marks the start of calcium carbonate precipitation, and before the charge was flooded.

General conditions: Sample No. 1 agglomerated with 10 per cent of solution. Stabilizing agents, lime and sodium carbonate; lime, 1 per cent, sodium carbonate, 1 per cent. Time allowed for CaCO₃ formation varied. Results presented in Table 2.

Discussion: The efficiency of lime and sodium carbonate increases as the time allowed for calcium carbonate formation is increased. Consequently, a period of time should be allowed for the reaction to become complete, and for the calcium carbonate to set before solution percolation is started.

Series B. Stability Tests with Sample No. 2

All of the previous tests were made with sample No. 1, containing 40.4 per cent of minus 200-mesh material. The tests in this series were made to find whether or not extremely fine material, such as sample No. 2, could be agglomerated and stabilized for percolation leaching.

Section I. Standard Test. No Means of Stabilization Used.—Test 31. Conditions: Sample No. 2 agglomerated with 10 per cent water. No stabilizing agents.

Results: As soon as the ore was flooded for the first time, it subsided and packed tight, so that solution would not percolate.

Discussion: With sample No. 2, which had been ground so that it would all pass through a 200-mesh sieve, the glomerules collapse very readily upon flooding. To preserve the permeability formed by agglomeration, the glomerules must be stabilized before flooding.

Section II. Lime and Sodium Carbonate.—General conditions: Sample No. 2 agglomerated with 18 per cent solution. Stabilizing agents, lime and sodium carbonate. Agglomerated material allowed to stand one hour before flooding. Results presented in Table 3.

TABLE 3.—*Stability Tests with Lime and Sodium Carbonate on Sample No. 2*

Test	Lime, Per Cent	Sodium Carbonate, Per Cent	Efflux	Rate of Flow, In. per Hr.
32	1	1	Clear	2.4
33	2	2	Clear	2.8
34	3	3	Clear	3.4

Discussion: The degree of stability of the glomerules increased with the amount of stabilizing agents used. These tests indicate that relatively large amounts of stabilizing agents will be required with extremely fine particles. The fact remains, however, that very fine material, which without stabilizing soon becomes impermeable, can be stabilized so that solution may be percolated by flood leaching.

CYANIDE LEACHING TESTS WITH ASHDOWN TAILINGS

Actual cyanide leaching tests were made in order to determine whether or not the precipitation of calcium carbonate in the glomerules would coat the mineral particles so that the values could not be dissolved. For this purpose a sample of old tailings from the Ashdown district, containing 0.036 oz. of gold per ton, was used. The sample contained lumps of clay slime, which were not broken before leaching. Table 4 shows the particle-size distribution obtained by a wet screen analysis, and the size of the material in the sample obtained by a dry screen analysis.

TABLE 4.—*Screen Analysis of Ashdown Tailings*

Mesh	Dry Screening, Per Cent	Wet Screening, Per Cent
+3	8.8	0.0
- 3 + 20	8.3	0.0
- 20 + 42	14.3	9.7
- 42 + 80	32.2	27.0
- 80 + 150	16.3	16.0
- 150 + 200	8.9	7.3
- 200	11.2	40.0

The presence of clay in the tailings was shown by the plasticity of the moist slimes and by an analysis which gave 13.9 per cent alumina.

Section I. Cyanide Agitation-leaching Tests.—Cyanide agitation-leaching tests were made to find the extraction that could be expected from the Ashdown tailings. These tests were made with 200 grams of tailings and 500 c.c. of solution in 2.5-liter bottles. Cyanide equivalent to 2 lb. NaCN per ton of solution and lime equivalent to 4 lb. CaO per ton of solution were used. The bottles were placed in a bottle agitator and left for varying periods of time. After the agitation period, the residue was removed and washed by filtration.

The gold content of the portions of Ashdown tailings used in the different tests varied considerably. Consequently, the recovery in each test was computed from the gold in solution and the gold in the residue.

The cyanide agitation-leaching tests gave a maximum recovery of 75 per cent in 15 hr., with a cyanide consumption of 0.59 lb. NaCN per ton of ore.

Section II. Cyanide Percolation-leaching Tests.—For percolation-leaching tests, the bottoms were removed from 2.5-liter reagent bottles. A rack was constructed to hold the bottles in an inverted position. Screens were placed over the necks of the bottles, and a layer of minus 3-mesh plus 4-mesh quartz placed on the screens to make a bottom for the bed of material to be leached. Samples containing 1800 grams of the Ashdown tailings that had been given the preliminary treatment described in each test were then charged into the bottles and packed by $\frac{1}{4}$ -min. vibration. The bottles were then placed in the rack and the addition of cyanide solution was started.

Test 1. Percolation leaching without agglomeration and stabilization. To find whether or not the Ashdown tailings could be leached by percolation without agglomeration and stabilization, one bottle was charged with the Ashdown tailings without any preliminary treatment. Cyanide solution containing 2 lb. NaCN per ton of solution was then added at the top.

Results: Considerable subsidence took place when the charge became wet with solution. A tight bed formed, preventing any appreciable percolation of solution.

Discussion: The test shows that this sample of Ashdown tailings cannot be leached by the percolation method without a pretreatment, to form and maintain a permeable bed.

Test 2. Percolation leaching with agglomeration and stabilization, using lime and carbon dioxide. Conditions: 1800 grams of Ashdown tailings; 20 grams hydrated lime mixed in dry; 200 c.c. cyanide solution used in agglomeration; CO₂ passes into charge for 4 minutes.

Four liters of a cyanide solution containing 2 lb. NaCN per ton of solution was passed through the charge eight times in 15 hr. The charge was allowed to drain between passages of solution, in order to draw air into the bed. Following the cyanide solution, two washes containing 1.5 liters of water were applied.

Results: A recovery of 76 per cent of the gold was made in the test. The glomerules did not make washing difficult because the first wash removed practically all of the dissolved gold. A cyanide consumption of 0.82 lb. NaCN per ton of ore occurred in the test. The permeability remained undiminished during the test.

Discussion: This test shows that the agglomeration-stabilization treatment with lime and carbon dioxide developed for quartz is effective for producing and maintaining permeability in ores and tailings even when considerable clay is present. The extraction is not adversely affected by the stabilization treatment. Continued treatment with cyanide solutions does not affect the permeability of an agglomerated and stabilized bed. The consumption of cyanide was greater than in the agitation-leaching test. Probably an excess of carbon dioxide

reduced the protective alkalinity sufficiently so that a part of the alkaline cyanide was used to form hydrocyanic acid.

Test 3. Percolation leaching with agglomeration and stabilization using lime and sodium carbonate. Conditions: 1800 grams Ashdown tailings; 20 grams hydrated lime mixed in dry; 200 c.c. cyanide solution containing 20 grams Na_2CO_3 used in agglomeration. Charge allowed to stand 1 hr. before solution inflow started.

Four liters of cyanide solution containing 2 lb. NaCN was passed through the charge 10 times in 15 hr. The charge was allowed to drain between passages of solution. Following the cyanide solution, two washes containing one liter of water were applied.

Results: A recovery of 74 per cent of the gold was made in the test. The first wash removed practically all of the dissolved gold. A cyanide consumption of 0.60 lb. NaCN per ton of ore occurred in the test. The permeability remained undiminished during the test.

Discussion: This test, using lime and sodium carbonate to stabilize, gave substantially the same extraction and permeability as the test with lime and carbon dioxide. The cyanide consumption was reduced to about the amount lost in the agitation-leaching test.

SUMMARY

1. Moisture agglomeration increases the permeability of slimes and finely ground ores sufficiently to allow satisfactory percolation of leach solutions.

2. Beds of moisture-agglomerated slimes subside and become practically impervious when flooded with solution.

3. A method of stabilizing moisture-agglomerated slimes by the precipitation of calcium carbonate in the glomerules has been developed. Laboratory tests with agglomerated and stabilized slimes gave satisfactory percolation of solution by flood leaching.

4. Laboratory cyanide-leaching tests on a gold-bearing tailing gave substantially the same extraction by percolation leaching after agglomeration and stabilization as that obtained by agitation leaching.

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