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J. Bruce Clemmer, Chief

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RESEARCH AND PROCESS INVESTIGATIONS ON
LAKEVIEW ORE, LAKE COUNTY, OREG.

by

I. L. Nichols, K. E. Tame, J. R. Ross,
and J. B. Rosenbaum

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Intermountain Experiment Station
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RESEARCH AND PROCESS INVESTIGATIONS ON
LAKEVIEW ORE, LAKE COUNTY, OREG.

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SUMMARY

Ore from the Lakeview Mining Co. was tested in the pilot plant and laboratory as requested by the Grand Junction Operations Office. The pilot plant test showed the unusually slimy ore could be handled by countercurrent decantation and that solvent extraction was feasible for recovering uranium from a pregnant solution contaminated with molybdenum, arsenic, and phosphorus. Subsequent laboratory research disclosed that use of a finer grind than employed in the pilot plant improved the leachability of the ore without adverse affect on handling of the leach pulp.

Approximately 15 tons of ore, assaying 0.34 percent U_3O_8 , was processed in the pilot plant at the rate of 1 ton per day. The 1/2-inch ore was ground at 45 percent solids to minus 28 mesh in an open-circuit rod mill. Agitation leaching for 28 hours at an ambient temperature of 55° C. using 200 pounds H_2SO_4 and 10 pounds MnO_2 per ton dissolved 93 percent of the uranium and 50 percent of the molybdenum. The leach residue was washed countercurrently in five thickeners having an area of 7 square feet each, using the equivalent of 4 tons of wash water per ton of ore. Flocculation with a total of 0.9 pound of Separan gave a pregnant liquor containing 0.3 gram solids per liter and thickener underflows ranging from 43 percent solids in the first thickener to 39 percent solids in the last thickener. Washing efficiency was 99.5 percent. An operating technique that proved essential for effective thickening of the slimy leach tails was recycling of solution within each thickener to dilute the thickener feed to about 14 percent solids.

The turbid pregnant solution from countercurrent decantation was partly clarified by settling to give a solution having a solids content of 0.03 gram per liter for amine solvent extraction. About 40 percent of the molybdenum in the leach liquor precipitated and reported in the slime fraction from the clarification operation. The clarified solution assayed in grams per liter, U_3O_8 0.74, Mo 0.07, P 0.07, and As 0.08; the pH was 1.0 and the redox potential was minus 360 mv. A four-stage partitioned mixer-settler with dual-roll impellers was used in extracting the uranium and molybdenum. The pregnant solution was processed at a rate of 0.7 gallon per minute, which corresponds to operation of the leach plant at 1 ton of ore per day. A 5:1

ratio of aqueous to organic extractant consisting of 5 percent amine, Amberlite LA-1 (formerly Rohm & Haas 9D-178), dissolved in kerosine gave raffinates assaying consistently less than 0.001 gram per liter U_3O_8 , which corresponds to 99.9 percent extraction of U_3O_8 . Molybdenum extraction averaged 85 percent.

The loaded organic assayed 3.5 grams per liter U_3O_8 and 0.3 gram per liter Mo. A five-stage partitioned tank mixer-settler was used for stripping. Uranium was stripped in three stages at a 6.5:1 ratio of organic to 1 molar NaCl solution acidified to pH 1.0. The strip solution assayed 24 grams per liter U_3O_8 and contained 99.8 percent of the uranium in the loaded organic. Molybdenum subsequently was stripped from the uranium depleted organic in two stages with 1 molar Na_2CO_3 at a 19:1 ratio to give a solution assaying 5 grams per liter Mo. Loss of the 5 percent amine averaged 1 gallon per 1,000 gallons of leach liquor. About half of the loss is attributed to entrainment by the solids in the incompletely clarified pregnant solution.

Uranium was precipitated from the loaded salt strip solution by addition of NH_4OH and MgO , singly or in combination. The highest grade product was made by precipitating at $70^\circ C$. with NH_4OH . This yellow cake after drying at $110^\circ C$. assayed 84 percent U_3O_8 and met specification grade in all respects. The principal contaminants were 0.35 percent Mo, 2.05 percent $SO_4^{=}$ and 0.5 percent $PO_4^{=}$. Ammonia requirement was 0.22 per pound of U_3O_8 .

Scanning tests were made for recovery of molybdenum from the soda ash scrub solution. Precipitation of a lead or calcium molybdate from the alkaline solution recovered 98 percent of the molybdenum in products assaying 20 percent Mo. Precipitation as the sulfide at pH 2 recovered 99 percent of the molybdenum in a product assaying 35 percent Mo.

An alternative procedure for stripping uranium and molybdenum from the loaded amine while simultaneously precipitating the uranium was examined in the laboratory. Use of a caustic soda solution for stripping in a single stage gave a sodium diuranate product assaying 80 percent U_3O_8 . Molybdenum remained in the strip liquor and was recovered by partly neutralizing with hydrochloric acid and adding calcium chloride to precipitate calcium molybdate.

Laboratory flotation tests made on the ore for removing arsenic and molybdenum, showed that arsenic was floatable whereas molybdenum was not. No processing advantages were evident from removing the arsenic. Fine grinding and flotation proved advantageous, however, in reducing the quantity of acid and oxidant required for leaching the ore. Use of 200 pounds of acid and 10 pounds of MnO_2 in the pilot

plant dissolved 93 percent of uranium from the 28 mesh wet ground feed, whereas leaching the flotation concentrate and tailing yielded 95 percent of the uranium, with a total consumption of only 90 pounds of acid and 2 pounds of MnO_2 . Subsequent tests showed that wet grinding the ore to minus 65 mesh, the same size as the flotation feed, gave a 95 percent extraction on leaching with 100 pounds of acid and no oxidant. About 5 percent of the uranium was dissolved during wet grinding. Because of acidic soluble salts present in the ore, the pH of the ground pulp was 3.8. Steps would have to be taken in a commercial plant to mitigate corrosion from the acid pulp in the grinding circuit. Thickening tests on the leach residues from fine-ground ore showed flocculant and area requirements were about the same as for the minus-28-mesh grind used in the pilot plant.

DESCRIPTION OF THE ORE

A 17-ton sample of unoxidized Lakeview Mining Co. ore, crushed to 1/2-inch, was furnished by the Grand Junction Operations Office, AEC, for the pilot-plant test. The ore as received contained 12 percent moisture. It analyzed, in percent, U_3O_8 0.340, V_2O_5 0.02, Mo 0.10, RCO_3 0.4, RSO_4 2.6, S⁼ 1.6, Fe 3.0, As 0.59, P 0.05, and Organic Carbon 0.3. A radiometric assay of 0.26 percent U_3O_8 showed the ore was at disequilibrium. A 50-pound sample for laboratory tests, cut from the feed belt in the Grand Junction pilot plant, was practically identical with the 17-ton sample submitted for the pilot plant tests except that the chemical assay was 0.400 percent U_3O_8 and the radiometric assay was 0.33 percent U_3O_8 .

Microscopic examination showed the Lakeview ore was composed mainly of volcanic glass, kaolinite, and sluffing-type bentonite clay. Quartz, muscovite, albite, and a trace of calcite also were present. Small amounts of magnetite, pyrite, realgar, orpiment, and barite were found in a heavy mineral concentrate that had about the same radioactivity as the ore. No discrete molybdenum mineral was observed. The uranium mineral tentatively was identified as extremely fine-grained uraninite.

PILOT-PLANT TESTING

The initial objectives in the investigation of the Lakeview ore were to determine whether the acid leach pulp could be handled by countercurrent decantation, and to assess the amenability of the pregnant solution to solvent extraction for uranium recovery. These objectives required pilot plant scale research. Enough laboratory work was done to establish general conditions for leaching, thickening, and solvent extraction before starting the pilot plant, but laboratory research to define optimum leaching conditions had to await completion of the pilot plant test.

Grinding

The ore was ground at 45 percent solids in open circuit at a rate of 1 ton per day in a 16- by 32-inch rod mill turning at 32 r.p.m. and loaded with 160 pounds of 1/2-inch to 1-1/2-inch rods. Owing to the thixotropic nature of the ore, a slurry containing about 45 percent solids was as thick as could be handled in the mill. Rod consumption was 0.5 pound per ton of ore. The grind was 2 percent plus 35 mesh and 41 percent minus 325 mesh. A sizing analysis showed that the highest uranium concentration occurred in the 100- to 200-mesh fraction that represented 20 percent of the ore and assayed 0.38 percent U_3O_8 . The minus-325-mesh fraction assayed 0.30 percent U_3O_8 . Screen analyses and distribution of uranium in the rod mill feed and discharge are given in table A of the Appendix.

Leaching

The rod mill discharge was diluted from 45 to 40 percent solids with wash solution from the No. 2 thickener for leaching at an ambient temperature of 55° C. in three 3- by 4-foot leach tanks in cascade. The agitators were 12-inch diameter, three-bladed, marine-type impellers turning at 520 r.p.m. A speed of 425 r.p.m., tried initially, was too slow to move the viscous pulp. Leaching time was 28 hours when processing 1 ton of ore per day. The plant was started at a feed rate of 1.5 tons per day, which gave a leach retention time of 19 hours, but this rate overloaded the thickeners. Uranium extraction was the same at either feed rate. Leaching with 200 pounds of H_2SO_4 and 10 pounds of MnO_2 per ton of ore dissolved 93.2 percent of the uranium. An increase in acid and MnO_2 to 250 and 15 pounds had no discernible effect on extraction. Leaching at 70° C. with 150 pounds of acid when using 5 pounds MnO_2 and 10 pounds $Fe_2(SO_4)_3$ as the oxidants extracted 91.2 percent of the uranium. Although all of the acid and oxidant were charged to the first leach tank, no pH gradient was observed in the leach circuit. The pH was 0.7 in each of the tanks. The residues from these leach cells assayed 0.043, 0.034 and 0.024 percent U_3O_8 , showing that only 2 to 3 percent of the uranium was extracted in the second and third leaching steps.

Feed and residue assays in the pilot plant indicated that 50 percent of the molybdenum was dissolved, whereas the pregnant solution which assayed 0.06 to 0.09 gram Mo per liter, accounted for only 25 to 35 percent of the molybdenum in the feed. Subsequently, it was found that the molybdenum tends to precipitate upon aging of the pregnant solution. Samples of slime settled from the pregnant liquor assayed over 1 percent molybdenum.

Daily averages of leach circuit data are given in table B of the Appendix. The assay of the residue from the final washing thickener is included in the table to show the absence of leaching in the thickening circuit. The 2-day retention time in the leach and thickener circuits must be considered in comparing the residue assays. The data are averaged separately for the two principal operating periods; the first when 200 pounds of acid and 10 pounds of MnO_2 were used, and the second when leaching with 150 pounds of acid and using 5 pounds of MnO_2 and 10 pounds of $\text{Fe}_2(\text{SO}_4)_3$ as the oxidants.

Countercurrent Decantation

The leached pulp was transferred by a 1-inch ODS pump to a 15-gallon surge tank which overflowed to the first CCD thickener. Before entering the thickener, the pulp was mixed with advancing wash solution from the No. 2 thickener in a 7-inch diameter by 5-inch bowl equipped with a laboratory stirrer. Separan^{1/} was metered as an 0.1 percent solution. The effectiveness of the flocculant, the thickening rate, and terminal densities were improved by recycling solution within each thickener to dilute the Separan to about an 0.01 percent solution and the thickener feed to 12 to 14 percent solids. The diluted Separan was added to the overflow of the pulp mixing bowl. Adequate mixing of the flocculant and pulp was obtained in traveling 18 inches down a 1-inch hose to the thickener feed well. A similar arrangement was used for blending and reflocculating the feed to each thickener.

Thickener underflows were handled by 3/4-inch Denver^{2/} adjustable stroke diaphragm pumps equipped with Graham^{2/} variable speed drives for controlling the pumping speed to maintain 8 to 12 inches of clear liquor in the thickeners. Air lifts were used for advancing the thickener overflows in the washing circuit. At the start, 4 tons of wash water and four 36- by 30-inch thickeners were used in the CCD circuit. Because of the slimy nature of the pulp and the resultant low terminal densities, a fifth thickener was added after 5 days' operation. The area per thickener was 7 square feet per ton day. Satisfactory flocculation was obtained with a total of 0.8 to 1.0 pound Separan per ton of ore. Half the Separan was added to the first thickener and the remainder was evenly divided among the remaining thickeners. Average underflow densities graded from 43 percent in the first thickener to 39 percent in the final thickener. Washing efficiency was 98.9 percent with four thickeners and 99.5 percent with five thickeners. Overall U_3O_8 recovery for 5

^{1/} Use of proprietary reagents and equipment does not constitute endorsement of these items over competitive items.

^{2/} Same as Footnote 1.

days' operation when leaching with 200 pounds of acid and 10 pounds of MnO_2 and washing with 4 tons of water in five thickeners was 92.8 percent.

Diagrams of the leach-CCD circuit and the blending and diluting system are shown in figures 1 and 2. Typical leach-CCD results are given in block diagram form in figure 3. Screen analyses and uranium distribution in the screen fractions of the final residue are shown in table A of the Appendix. Daily averages of operating data for each thickener are given in tables C through G in the Appendix. Efficient washing in each thickener is evidenced by the similarity in underflow and overflow solution assays.

Solvent Extraction

The primary thickener overflow, which contained 0.3 gram per liter solids, was partly clarified to a solids content of 0.03 gram per liter by settling on a continuous basis in the solvent extraction head tank. A plywood divider, 3 feet deep, served to partition the 7-foot diameter tank, thus permitting turbid feed to enter on one side of the partition while continuously removing clarified pregnant solution from the other side. The clarified pregnant solution assayed in grams per liter, U_3O_8 0.74, Mo 0.07, Fe 1.2, Al_2O_3 1.1, MgO 0.2, Mn 1.0, P 0.07, As 0.08, and SO_4 25. Partitioned mixer-settlers with mixing impellers, also serving as low head pumps, were used in processing the pregnant solution at a rate of 0.7 gallon per minute, which corresponds to operation of the leach plant at 1 ton of ore a day. Maximum throughput capacity of the extraction unit, as limited by the phase-disengagement rate, appeared to be 1 gallon per minute of pregnant solution when operating the unit with a 3-inch layer of the organic. This corresponds to about 2 square feet of settler area per gallon of aqueous per minute. The throughput capacity could be doubled by increasing the depth of the organic phase in the settlers to about 12 inches. Figure 4 shows the 4-stage extraction and 5-stage stripping units used for processing the Lakeview solution. Overall dimensions of the extraction unit is 40- by 20-inches deep. Each mixing compartment is 9 by 12 inches; settling compartments are 9 by 30 inches. The marine-type impellers for mixing and pumping are 5.5 inches in diameter. A 3-inch disc mounted on the shaft immediately above the impeller serves to improve the pumping efficiency. The stripping unit is 36- by 44- by 20-inches deep. Mixing compartments are 8 by 10 inches and settlers are 8 by 24 inches.

Uranium and molybdenum were extracted in four stages using 0.1 molar amine, Amberlite LA-1, in kerosine at an aqueous to organic ratio of 5:1. An organic depth of 2 to 3 inches was maintained in each settler and sufficient organic was recycled by means of weir adjustment to maintain a 3:1 organic to aqueous ratio in the mixers. Aqueous contact time in each mixer was 1.6 minutes. Owing to the presence of 0.03 gram of solids per liter in the pregnant feed, a layer of gruel

Figure 1 - Acid leach-CCD pilot plant flowsheet for Lakeview ore.

Figure 2 - Schematic diagram of blending and diluting system in CCD circuit using No. 2 thickener as example.

Figure 3 - Acid leach and countercurrent decantation of Lakeview ore at a feed rate of 1 ton per day.

Figure 4 - Compartment tank mixer-settlers extracting uranium and molybdenum from Lakeview Mining Co. acid leach pregnant solution. Four amine extraction stages in unit at left; three salt-strip stages for uranium, and two carbonate strip stages for molybdenum in unit at right.

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about 1 inch deep accumulated at the interface in the first two stages. The gruel was removed at the end of the 14-day campaign and filtered to reclaim the organic content.

The uranium was stripped from the loaded organic in three stages at an organic to aqueous ratio of 6.5:1 with a 1 molar salt solution acidified to pH 1.0 with sulfuric acid. A 2-inch depth of organic was maintained in the settlers. Recycling organic to the mixers was not required.

The salt strip for uranium was followed by 2-stage scrubbing with a 1-molar soda ash solution for removal of molybdenum at a 19:1 organic to aqueous ratio. Two inches of organic was maintained in the settlers, and enough soda ash solution was recycled to maintain a 19:3 ratio of organic to aqueous in the mixers. The settlers in the stripping and scrubbing units were severalfold larger than needed for phase separation.

The salt-strip solution generally assayed 24 grams per liter U_3O_8 and 0.07 gram per liter Mo. Other stripping tests were attempted with a neutral salt solution, but gruel formed in the strippers. An acidic 1.5-molar salt solution was found to strip more of the molybdenum which resulted in a contaminated uranium product. The carbonate scrub solution assayed 0.15 gram per liter U_3O_8 and 5.0 grams per liter Mo. Adequate retention time of the aqueous phase in the mixers is essential for efficient stripping or scrubbing. At an organic to salt solution ratio of 6.5:1, the contact time of the salt solution in each mixer was 20 minutes, which was just adequate for the conditions employed. When scrubbing at an organic to aqueous ratio of 19:1, contact time of the carbonate solution was insufficient for removing molybdenum from the organic. Stripping of the molybdenum was improved when the aqueous contact time in the mixers was tripled by recycling soda ash solution from the settlers to give a 19:3 organic to aqueous mixing ratio.

Extraction and stripping results are summarized in figure 5. A barren raffinate, assaying 0.001 gram per liter U_3O_8 and 0.01 gram per liter Mo, was produced in two extraction stages indicating that the remaining two stages were superfluous under the operating conditions employed. The 5:1 ratio of aqueous to organic was selected in anticipation of a higher molybdenum content in the pregnant solution. Neither arsenic nor phosphorus was extracted by the amine. The stripping gradient shows all three salt strip stages were needed for the uranium. Since complete removal of molybdenum from the organic is not required, a single carbonate scrub would have sufficed.

Figure 5.- Extraction of Lakeview Liquor Using 0.1 Molar Amberlite LA-1 in kerosine.

Salt and soda ash requirements for stripping and scrubbing were 2.4 and 1.6 pounds per pound of U_3O_8 recovered. A lower flow rate of soda ash to increase the molybdenum loading was not investigated in the pilot plant because of the excessive time needed to achieve equilibrium with small flows in the oversized strippers.

An organic materials balance for the entire campaign revealed the loss of 1 gallon organic per 1,000 gallons of pregnant solution. The loss was manyfold higher than indicated by solubility data and by routine entrainment determinations on the raffinate. Much of the loss can be attributed to adsorption of organic on the solids in the only partly clarified pregnant solution. Previous pilot plant experience in amine extraction has shown less than half as much organic was lost when processing solutions containing about 0.02 percent solids.

Precipitation

Precipitation of yellow cake from the 1-molar salt strip solution, which assayed 24 grams per liter U_3O_8 , required 0.22 pound of NH_3 or about 0.32 pound MgO per pound of U_3O_8 . Some 340 gallons of loaded salt stripping solution was precipitated in 50-gallon batches using NH_4OH or combinations of precipitants. The products were filtered and washed in a plate and frame press. The best grade product was made by precipitating with NH_4OH at $70^\circ C$. A sample dried at $110^\circ C$. assayed 84.0 percent U_3O_8 and met all specifications. Impurities, in percent, were V_2O_5 0.01, Mo 0.35, $PO_4^{=}$ 0.5, $CO_3^{=}$ 0.12, Cl^- 0.02, F^- 0.015, As 0.02, $SO_4^{=}$ 2.05, Cu 0.02, Fe 0.10, Ca 0.3, and B 0.0003. The grade was raised to 88.9 percent U_3O_8 by calcining at $700^\circ C$. Use of ammonia to raise the pH to 4.5 or 5.5 followed by addition of MgO to pH 7 gave products that were difficult to wash free of sulfates. Precipitates made in this manner contained from 8 to 12 percent $SO_4^{=}$. Lime was used to adjust the pH to 3.5 for partial removal of sulfate in one test before precipitating the uranium with MgO . The gypsum product contained 3 percent of the uranium. Excess MgO was used for precipitation in this test. The product assayed in percent, U_3O_8 78.3, MgO 9.7, $SO_4^{=}$ 0.16, $PO_4^{=}$ 0.4, $CO_3^{=}$ 0.4, Mo 0.16, Fe 0.02, Ca 1.4, and B 0.0004. Quantitative filtration data were not obtained. When using ammonia as the precipitant, hot filtration appeared necessary for a practicable rate.

Metallurgical Balance

A uranium balance for the pilot-plant run is given in block diagram form in figure 6. Accountable losses were 6.8 percent of the uranium as insoluble and, 0.8 percent as soluble loss in the washed residues, 0.1 percent in the solvent extraction raffinate, and 0.2 percent in the carbonate scrub solution. Unaccountable losses of 0.5 and 0.3 percent were incurred in the leach-CCD circuit and the solvent extraction circuit.

Fig. 6 - Uranium Balance

LABORATORY RESEARCH

Flotation

Flotation tests were made on the ore to determine the effect of arsenic removal on uranium extraction. The ore for flotation was ground to pass 65 mesh in a laboratory ball mill using Salt Lake City tap water. Typical results obtained by flotation with Minerec B, Aero-float 25, Copper Sulfate, and Dowfroth 250 are shown as follows:

	Weight, percent	Assay, percent		Distribution, percent	
		U ₃ O ₈	As	U ₃ O ₈	As
Concentrate	10.8	0.477	4.08	15.9	84.5
Tailings	<u>89.2</u>	<u>.304</u>	<u>.09</u>	<u>84.1</u>	<u>15.5</u>
Calculated head	100.0	.323	.52	100.0	100.0

The arsenic concentrate assayed 0.14 percent Mo, which was only slightly higher than the heads, indicating negligible flotation of molybdenum.

Leaching tests showed that 94 percent of the uranium in the flotation tailing could be dissolved with only 75 pounds of acid per ton of tailing. More surprising, in view of the acid requirements in the pilot plant, only 150 pounds of acid and 10 pounds of MnO₂ extracted 97 percent of the uranium from the concentrate. The calculated overall extraction was 94.6 percent with a total consumption of 90 pounds acid and 2 pounds MnO₂ per ton ore. These results demonstrated that arsenic had no particular effect on uranium extraction. Of more importance, however, was the indication that fine grinding or oxidation during grinding and flotation materially reduced acid and oxidant requirements.

Leaching

Leaching tests to establish general conditions for the pilot plant were made on 35-mesh dry ground ore in accordance with the routine amenability testing procedure. The leaching results subsequently obtained on the flotation products indicated that additional work was justified to determine the effect of fine or dry grinding, wet grinding, and air oxidation on uranium extraction and reagent requirements. The dry and wet ground material was leached at 55° C. for 18 hours at 40 percent solids. Screen analyses for the different grinds are shown as follows:

Size	Weight, percent						
	Dry grind, mesh			Wet grind, time minutes			
	10	35	65	5	10	15	20
+ 35 mesh	47.9	-	-	1.5	-	-	-
- 35 + 65 mesh	13.4	31.9	-	9.6	1.8	0.4	0.1
- 65 +100 mesh	6.2	12.2	17.3	12.2	4.1	1.4	0.5
-100 +200 mesh	8.7	16.8	28.3	21.7	19.7	12.9	7.6
-200 mesh + 20 microns	8.8	18.5	27.1	26.2	31.5	33.6	31.9
- 20 microns	<u>15.0</u>	<u>20.6</u>	<u>27.3</u>	<u>28.8</u>	<u>42.9</u>	<u>51.7</u>	<u>59.9</u>
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Laboratory Sample

The leachability of the 50-pound sample received from the Grand Junction Operations Office for laboratory testing appeared to be sufficiently different from the ore delivered for pilot plant processing to justify separate testing. A series of batch leaching tests were made on both wet and dry ground portions of the sample to investigate the effects of size and oxidation on uranium extraction as related to acid requirements. The results of the more pertinent tests are summarized in table H of the Appendix.

Dry grinding the sample to 65 mesh resulted in 90.5 percent extraction as compared with 87.2 percent from the 35-mesh material when leaching with 150 pounds of acid. Similar tests made on the two materials using 10 pounds of MnO_2 as the oxidant increased the extractions to 92.3 and 93.9 percent. Improved extraction resulted from the finer grind and also from oxidation during dry grinding.

The influence of aerating a neutral pulp of dry-ground ore was checked in a leach with 100 pounds of acid. The slurry of 300 grams of 35-mesh ore at 40 percent solids was aerated, using a total of 3 cubic feet of air over a 2-hour period before the acid was added. Uranium extractions were 83.5 percent without air blowing, 85.1 percent after air blowing, and 90.5 percent when 10 pounds MnO_2 was used in lieu of aeration.

A 10-minute wet grind in the laboratory ball mill gave a product containing substantially more fines than the 65 mesh dry grind. Leaching with 150 pounds of acid extracted 94.5 percent of the uranium. Extraction increased to 95.3 percent when the sample was wet ground for 20 minutes to substantially 100 mesh. A slight gain in extraction to 94.8 and 95.8 percent was experienced when 10 pounds of MnO_2 was added.

as the oxidant in comparable leaching tests. The higher oxidation state of the leach solution as shown by the redox potential and the improved uranium extraction attests to the efficiency of air oxidation during wet grinding of the Lakeview ore.

Pilot Plant Sample

The pilot plant sample appeared easier to leach than the initial laboratory sample. Use of 150 pounds of acid extracted 89.4 and 93.8 percent of the uranium from dry ground 35-mesh and 65-mesh ore as compared with 87.2 and 90.5 percent from the laboratory sample. The difference was less noticeable when leaching wet ground ore because extraction from both samples leveled off at 94 to 95 percent. Addition of MnO_2 as the oxidant when leaching wet-ground ore gave a small but consistent increase in extraction. When leaching ore ground for 20 minutes, equivalent to a 100-mesh grind, 100 pounds of acid dissolved 95.3 percent of the uranium. Doubling the acid gave only a slight gain in extraction.

Aerating a slurry of the 35-mesh dry ground ore before adding 150 pounds of acid resulted in 2.3 percent increase in uranium extraction. In this instance, air oxidation appeared equivalent to about 5 pounds of MnO_2 . The test data for the pilot plant sample are summarized in table I of the Appendix.

Molybdenum Recovery

Scanning tests were made on precipitation and recovery of molybdenum from the soda ash scrub solution produced in the solvent extraction pilot plant. The solution assayed in grams per liter, 3.95 Mo, 0.44 U_3O_8 , 53 Cl^- , 2.2 $SO_4^{=}$, 0.06 As, and 0.06 P, and had a pH of 7.9. Precipitants for molybdenum included lime, $CaCl_2$, Pb acetate, H_2S and Na_2S at various concentrations and pH. An excess of lime precipitated only 34 percent of the molybdenum. Sulfidizing was effective only in acidified solutions, and Na_2S appeared better than H_2S . Acidification of the solution to pH 2 and precipitation with 3.8 pounds of Na_2S per pound of Mo recovered 99 percent of the molybdenum in a product assaying 35 percent Mo and 51 percent sulfur. Owing to the formation of $CaCO_3$, precipitation with $CaCl_2$ required 10 pounds per pound of Mo for 98 percent molybdenum removal. The product assayed 20 percent Mo, 26 percent Ca, and 24 percent carbonate. Addition of lead acetate caused the pH to rise from 7.9 to 8.3. Use of 4.2 pounds precipitated 96 percent of the molybdenum in a product assaying 20 percent Mo and 53 percent Pb.

Although the lead acetate precipitation gave a good recovery and product, this reagent appears too costly for use as a precipitant. Calcium chloride and sodium sulfide appear to have good possibilities

as precipitants and would merit more investigation should additional research on molybdenum recovery be warranted.

A few exploratory tests were made using caustic stripping for recovering uranium and molybdenum from the loaded amine. The loaded amine was contacted in a laboratory mixer-settler with 10 percent NaOH solution to give a sodium diuranate precipitate and a strip liquor containing sodium molybdate. The uranium products, dried at 110° C., assayed 75 to 80 percent U_3O_8 and only 0.02 percent molybdenum. Organic entrained in the uranium product resulted in a carbon content of 2.6 percent. Conceivably most of the organic could be washed from the diuranate precipitate. Excess alkalinity of the caustic solution had to be neutralized before the molybdenum could be precipitated as calcium molybdate by addition of calcium chloride.

The caustic stripping procedure appears to have good possibilities for handling amine loaded with uranium and molybdenum. Principal difficulties were in discharging the sodium diuranate from the settler and washing the product free of organic. Additional research is in progress to develop and evaluate caustic stripping for separation and recovery of uranium and molybdenum. Another alternative for separating uranium and molybdenum is to strip both metals from the loaded amine with soda ash solution, and then selectively precipitate the uranium by addition of caustic. This procedure, however, would result in a lower grade uranium product and appears more complicated and costly than the caustic stripping procedure.

CONCLUSIONS

The Lakeview ore can be handled by acid agitation leaching, countercurrent decantation washing, and amine solvent extraction. Unlike most uranium ores tested, the uranium mineral does not tend to slime. Relatively fine grinding is obligatory for uranium extraction of 93 to 95 percent with moderate expenditure of acid and oxidant. Owing to the slimy nature of the leach residue, 0.8 to 1.0 pound of flocculant per ton ore, considered high by ordinary standards, is needed for CCD washing. Recycling of solution within each thickener for dilution of the flocculant and thickener feed is essential for efficient operation. Underflow densities higher than the pilot plant range of 43 to 39 percent should be readily obtained in commercial thickeners because of the greater pulp depth. Oversize pumps are suggested for the unusually viscous thickener underflows. Use of 4 tons of wash water and 5 thickeners results in a washing efficiency of 99.5 percent. Some sacrifice of washing efficiency by use of less water or only 4 thickeners may be economically justified.

The pregnant solution from the primary thickener should be clarified to a solids content of less than 0.02 percent to inhibit loss of organic in amine solvent extraction. Other pretreatment of the solution is not necessary, but provision must be made for separating molybdenum from uranium in the loaded organic to produce a specification-grade uranium product. The salt strip-carbonate scrub procedure checked in the pilot plant extracted 99.9 percent of the uranium from the pregnant leach solution and recovered 99.7 percent in a specification-grade product assaying 84 percent U_3O_8 .

APPENDIX

TABLE A. - Screen Analysis and Uranium Distribution

Mesh	Rod Mill Feed			Rod Mill Discharge			No. 5 Thickener Underflow		
	Weight, percent	Assay U ₃ O ₈ , percent	Distribution U ₃ O ₈ , percent	Weight, percent	Assay U ₃ O ₈ , percent	Distribution U ₃ O ₈ , percent	Weight, percent	Assay U ₃ O ₈ , percent	Distribution U ₃ O ₈ , percent
+ 4	32.5	0.239	26.1						
- 4 + 10	17.8	.402	24.0						
- 10 + 28	11.4	.488	18.6						
- 28 + 35	4.3	.340	4.9	2.2	0.282	1.8	1.4	0.046	2.6
- 35 + 48	3.6	.295	3.6	4.9	.344	5.0	4.0	.045	7.7
- 48 + 65	5.2	.241	4.2	10.2	.337	10.2	8.0	.039	13.2
- 65 + 100	4.8	.223	3.6	12.9	.370	14.1	11.4	.034	16.7
-100 + 200	6.1	.233	4.8	19.6	.382	22.2	18.0	.031	23.9
-200 + 325	2.1	.241	1.7	9.5	.361	10.2	7.2	.026	8.1
-325	<u>12.2</u>	<u>.208</u>	<u>8.5</u>	<u>40.7</u>	<u>.302</u>	<u>36.5</u>	<u>50.0</u>	<u>.013</u>	<u>27.8</u>
Calculated head	100.0	.298	100.0	100.0	.337	100.0	100.0	.023	100.0

TABLE B. - Leaching Data - Daily Averages

1957 Date	Leach feed, percent U ₃ O ₈	pH			Temperature, ° C.			Leach solution, U ₃ O ₈ gm./l.	Residue assay, percent U ₃ O ₈	
		Agitator No. 1	Agitator No. 2	Agitator No. 3	Agitator No. 1	Agitator No. 2	Agitator No. 3		Leach pulp	Final thickener underflow
5/16	0.33	-	-	-	-	-	-	-	-	-
5/17	.32	0.6	0.9	1.0	45	36	35	2.13	-	-
5/20	.31	-	-	-	-	-	-	-	-	0.024
5/21	.34	.4	.6	.7	48	44	41	1.47	0.023	.025
5/22	.35	-	-	-	57	51	45	-	-	.024
5/23	.32	.7	.7	.7	61	60	52	1.78	.023	.023
5/24	.36	-	-	-	60	60	53	-	-	.024
5/25	.33	-	-	-	49	53	49	-	-	.025
5/26	.34	.7	.7	.7	49	54	50	-	.022	.024
5/27	.35	-	-	-	50	54	50	-	-	.023
5/28	.33	-	-	-	50	55	50	-	-	.025
5/29	.33	-	-	-	50	55	51	2.11	.028	.025
Average ^{1/} 5/25-29	.34	.7	.7	.7	50	54	50	2.11	.025	.024
6/4	.33	-	-	.4	64	53	46	2.20	.028	.021
6/5	.34	-	-	.7	70	67	62	1.91	.031	.023
6/6	.35	-	-	.8	70	66	61	1.83	.033	.029
6/7	.33	-	-	.8	70	65	61	1.79	.032	.032
Average ^{2/} 6/4-7	.34	-	-	.7	69	63	58	1.93	.031	-
Overall average	.34	-	-	-	-	-	-	1.90	.028	-

^{1/} Leached with 200 pounds H₂SO₄ and 10 pounds MnO₂ per ton. Averages for 5 days' operation under stable conditions.

^{2/} Leached with 150 pounds H₂SO₄, 5 pounds MnO₂, and 10 pounds Fe₂(SO₄)₃ per ton.

TABLE C. - Thickener No. 1 - Daily Average Data

1957 Date	Feed solids, percent	Separan lb./ton	Clear liquor depth, inches	Underflow				Overflow		
				Solids, percent	Solution			Solution		
					U ₃ O ₈ gm./l.	Specific gravity	pH	U ₃ O ₈ gm./l.	Specific gravity	pH
5/20	17	0.4	13	34	-	-	-	0.65	1.022	1.1
5/21	15	.4	12	37	-	-	-	.71	1.023	1.0
5/22	-	.41	13	37	-	-	-	.76	1.022	1.0
5/23	-	.40	14	42	-	-	-	.75	1.023	1.1
5/24	12	.40	13	40	0.72	-	1.0	.79	1.024	1.0
5/25	-	.41	14	41	-	-	-	.74	1.026	.8
5/26	-	.41	12	42	-	-	-	.78	1.025	.8
5/27	-	.40	13	43	-	-	-	.75	1.024	.8
5/28	-	.40	12	45	-	-	-	.77	1.023	.8
5/29	12	.40	10	45	.75	1.023	.8	.75	1.025	.8
Average 5/25-29		.40	12	43					1.025	
6/4	-	.40	9	39	-	-	-	.72	1.020	.9
6/5	-	.42	12	44	-	-	-	.73	1.021	1.1
6/6	-	.40	15	43	-	-	-	.75	1.020	1.2
6/7	12	.40	10	44	.63	1.020	1.2	.69	1.020	1.2
Average 6/4-7		.40	12	43					1.020	1.1
Overall average		.40	12	41				.74	1.023	1.0

TABLE D. - Thickener No. 2 - Daily Average Data

1957 Date	Feed solids, percent	Separan lb./ton	Clear liquor depth, inches	Underflow				Overflow		
				Solids, percent	Solution			Solution		
					U ₃ O ₈ gm./l.	Specific gravity	pH	U ₃ O ₈ gm./l.	Specific gravity	pH
5/20	18	0.10	12	30	-	-	-	-	-	-
5/21	14	.12	19	38	-	-	-	-	-	-
5/22	-	.10	15	36	-	-	-	-	-	-
5/23	-	.10	10	39	-	-	-	-	-	-
5/24	14	.10	14	40	0.26	1.014	1.3	0.26	1.014	1.3
5/25	-	.11	12	38	-	-	-	-	-	-
5/26	-	.15	9	40	-	-	-	-	-	-
5/27	-	.14	8	41	-	-	-	-	-	-
5/28	-	.14	6	41	-	-	-	-	-	-
5/29	14	.14	7	38	.21	1.012	1.2	.21	1.010	1.2
Average 5/25-29		.14	8	40						
6/4	-	.12	13	40	-	-	-	-	-	-
6/5	-	.13	12	40	-	-	-	-	-	-
6/6	-	.13	10	38	-	-	-	-	-	-
6/7	14	.12	10	40	.20	1.012	1.5	.18	1.015	1.5
Average 6/4-7		.12	11	40						
Overall average		.12	11	39						

TABLE E. - Thickener No. 3 - Daily Average Data

1957 Date	Feed solids, percent	Separan lb./ton	Clear liquor depth, inches	Solids, percent	Underflow			Overflow		
					U ₃ O ₈ gm./l.	Solution		U ₃ O ₈ gm./l.	Solution	
						Specific gravity	pH		Specific gravity	pH
5/20	18	0.1	7	35	-	-	-	-	-	-
5/21	14	.1	13	39	-	-	-	-	-	-
5/22	-	.09	15	37	-	-	-	-	-	-
5/23	-	.1	8	40	-	-	-	-	-	-
5/24	14	.1	10	39	0.086	1.005	1.7	0.087	1.008	1.7
5/25	-	.10	12	37	-	-	-	-	-	-
5/26	-	.12	9	39	-	-	-	-	-	-
5/27	-	.14	10	42	-	-	-	-	-	-
5/28	-	.12	8	41	-	-	-	-	-	-
5/29	14	.12	6	39	.077	1.004	1.4	.084	1.003	1.4
Average 5/25-29		.12	9	40						
6/4	-	.14	13	39	-	-	-	-	-	-
6/5	-	.16	10	40	-	-	-	-	-	-
6/6	-	.13	11	42	-	-	-	-	-	-
6/7	14	.13	11	42	.057	1.008	1.7	.067	1.01	1.6
Average 6/4-7		.14	11	40						
Overall average		.12	10	39						

TABLE F. - Thickener No. 4 - Daily Average Data

1957 Date	Feed solids, percent	Separan lb./ton	Clear liquor depth, inches	Underflow				Overflow		
				Solids, percent	Solution			Solution		
					U ₃ O ₈ gm./l.	Specific gravity	pH	U ₃ O ₈ gm./l.	Specific gravity	pH
5/20	18	0.10	9	31	0.052	1.001	1.7	-	-	-
5/21	14	.11	11	38	.048	1.005	2.0	-	-	-
5/22	-	.10	8	36	.031	1.004	2.1	-	-	-
5/23	-	.10	8	38	.022	1.005	2.2	-	-	-
5/24	14	.14	6	39	.020	1.004	2.1	0.021	1.005	2.1
5/25	-	.10	8	40	-	-	-	-	-	-
5/26	-	.10	11	39	-	-	-	-	-	-
5/27	-	.10	8	40	-	-	-	-	-	-
5/28	-	.15	5	42	-	-	-	-	-	-
5/29	14	.17	8	41	.027	1.003	1.6	.026	1.003	1.6
Average 5/25-29		.12	8	40						
6/4	-	.15	12	40	-	-	-	-	-	-
6/5	-	.14	9	41	-	-	-	-	-	-
6/6	-	.15	12	42	-	-	-	-	-	-
6/7	14	.13	11	43	.024	1.003	1.9	.022	1.004	1.8
Average 6/4-7		.14	11	41						
Overall average		.12	9	39						

TABLE G. - Thickener No. 5 - Daily Average Data

1957 Date	Feed solids, percent	Separan lb./ton	Clear liquor depth, inches	Underflow				Overflow		
				Solids, percent	Solution			Solution		
					U ₃ O ₈ gm./l.	Specific gravity	pH	U ₃ O ₈ gm./l.	Specific gravity	pH
5/25	-	0.14	9	39	0.016	1.004	2.1	-	-	-
5/26	-	.16	9	38	.012	1.004	2.2	-	-	-
5/27	-	.12	7	39	.009	1.004	2.2	-	-	-
5/28	-	.14	5	39	.013	1.003	1.9	-	-	-
5/29	14	.16	5	38	.010	1.003	1.8	0.008	1.002	1.8
Average 5/25-29		.14	7	39		1.004	2.1			
6/4	-	.13	11	38	.011	1.002	1.9	-	-	-
6/5	-	.14	8	38	.009	1.002	2.0	-	-	-
6/6	-	.15	7	40	.009	1.002	2.0	-	-	-
6/7	14	.14	7	42	.008	1.002	2.0	.008	1.002	1.9
Average 6/4-7		.14	8	40	.011	1.002	2.0			
Overall average		.14	8	39		1.003	2.1			

TABLE H. - Leaching Tests, Laboratory Sample,
0.40 Percent U₃O₈

Standard Conditions: 55° C., 18 hours, 40 percent solids

Description	Leach solution		Residue analysis percent,	Extraction, percent
	pH	E.m.f.	U ₃ O ₈	U ₃ O ₈
DRY GRIND				
35 mesh, 150 lbs. H ₂ SO ₄ , no oxidant	0.6	335	0.054	87.2
35 mesh, 150 lbs. H ₂ SO ₄ , 10 lbs. MnO ₂	.7	355	.033	92.3
65 mesh, 150 lbs. H ₂ SO ₄ , no oxidant	.7	335	.040	90.5
65 mesh, 150 lbs. H ₂ SO ₄ , 10 lbs. MnO ₂	.7	355	.026	93.9
35 mesh, 100 lbs. H ₂ SO ₄ , no oxidant	.9	320	.070	83.5
35 mesh, 100 lbs. H ₂ SO ₄ , air blown	.9	340	.063	85.1
35 mesh, 100 lbs. H ₂ SO ₄ , 10 lbs. MnO ₂	1.0	330	.040	90.5
WET GRIND				
10 min. grind, 150 lbs. H ₂ SO ₄ , no oxidant	.7	370	.023	94.5
10 min. grind, 150 lbs. H ₂ SO ₄ , 10 lbs. MnO ₂	.8	370	.022	94.8
20 min. grind, 150 lbs. H ₂ SO ₄ , no oxidant	.8	355	.020	95.3
20 min. grind, 150 lbs. H ₂ SO ₄ , 10 lbs. MnO ₂	.9	360	.018	95.8

TABLE I. - Leaching Tests, Pilot Plant Sample,
0.34 percent U_3O_8

General Conditions: 55° C., 18 hours, 40 percent solids

Description	Leach solution		Residue analysis, percent U_3O_8	Extraction, percent U_3O_8
	pH	E.m.f.		
DRY GRIND				
10 mesh, 150 lbs. H_2SO_4 , no oxidant	0.6	355	0.059	82.1
10 mesh, 150 lbs. H_2SO_4 , 10 lbs. MnO_2	.6	355	.045	86.5
35 mesh, 150 lbs. H_2SO_4 , no oxidant	.6	345	.035	89.4
35 mesh, 150 lbs. H_2SO_4 , 10 lbs. MnO_2	.7	360	.026	91.6
65 mesh, 150 lbs. H_2SO_4 , no oxidant	.7	350	.021	93.8
65 mesh, 150 lbs. H_2SO_4 , 10 lbs. MnO_2	.7	360	.019	94.1
35 mesh, 200 lbs. H_2SO_4 , no oxidant	.5	355	.029	91.3
35 mesh, 200 lbs. H_2SO_4 , 10 lbs. MnO_2	.5	370	.023	93.2
WET GRIND				
5 min. grind, 150 lbs. H_2SO_4 , no oxidant	.6	360	.023	92.8
5 min. grind, 150 lbs. H_2SO_4 , 10 lbs. MnO_2	.7	365	.019	94.3
10 min. grind, 150 lbs. H_2SO_4 , no oxidant	.6	365	.018	94.6
10 min. grind, 150 lbs. H_2SO_4 , 10 lbs. MnO_2	.7	370	.016	95.2
15 min. grind, 150 lbs. H_2SO_4 , no oxidant	.7	360	.020	93.1
15 min. grind, 150 lbs. H_2SO_4 , 10 lbs. MnO_2	.7	365	.015	95.5
20 min. grind, 150 lbs. H_2SO_4 , no oxidant	.7	360	.015	95.5
20 min. grind, 150 lbs. H_2SO_4 , 10 lbs. MnO_2	.7	365	.014	95.8
20 min. grind, 100 lbs. H_2SO_4 , no oxidant	1.1	355	.016	95.3
20 min. grind, 150 lbs. H_2SO_4 , no oxidant	.7	350	.015	95.5
20 min. grind, 200 lbs. H_2SO_4 , no oxidant	.5	360	.014	96.0
DRY GRIND AND AIR OXIDATION, LEACH AT 70° C.				
35 mesh, 150 lbs. H_2SO_4 , no oxidant	.9	325	.033	90.2
35 mesh, 150 lbs. H_2SO_4 , air blown	.8	335	.026	92.3
35 mesh, 150 lbs. H_2SO_4 , 5 lbs. MnO_2	.9	330	.028	91.7