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AECU-3367

CHEMISTRY

UNITED STATES ATOMIC ENERGY COMMISSION

INNOVATIONS IN PROCESSING URANIUM ORES

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September 1956

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U. S. DEPARTMENT OF THE INTERIOR
BUREAU OF MINES
DIVISION OF MINERALS TECHNOLOGY, REGION III
J. Bruce Clemmer, Chief

RESEARCH REPORT NO. 46.1

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by

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A. I. M. E.

Salt Lake City, Utah, September 27, 1956

Security restrictions on processing uranium raw materials to yellow cake were lifted in August 1955, coincidental with the Geneva Conference on Peaceful Uses of Atomic Energy. Numerous reports and technical papers on uranium extraction and recovery methods have appeared since that time, and it is doubtful that another review of past and current practices would add much to the technical literature. The development of some procedures for treating uranium ores that looked good on paper were arrested, perhaps temporarily, by mechanical handling problems or peculiarities in the chemistry of uranium and associated elements. Others have been incompletely evaluated and still others that tested well in the laboratory and pilot plant are dormant because of competition from other processes, or because of their restricted applicability to particular ore types that have not reached quantity production. Owing to the vast number of new ideas and procedures that have been advanced, considered, and tested, we recognize the futility of attempting a comprehensive summation in this paper. A cross section of the field, based on random sampling, is as good a coverage as we can hope to provide here. We have chosen, therefore, to discuss some innovations that may provide a clue to the uranium ore processing techniques of the future. An effort has been made to select those innovations that have not had much publicity and which illustrate new metallurgical techniques or embody interesting modifications and applications of established principles and procedures. With the two exceptions noted later, all of the research described was sponsored by the United States Atomic Energy Commission.

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- ^{1/} This paper has been approved for presentation by the Atomic Energy Commission and the Director of the Bureau of Mines.
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PHYSICAL BENEFICIATION

The individuals and firms engaged in mining and processing uranium ores, and in adjunctive research activities, frequently have had considerable experience in the field of ore dressing. Therefore, as might be expected, a prodigious amount of work has been done on physical beneficiation testing of uranium ores, particularly in efforts to concentrate subgrade materials. Although some success has been attained on specific ores by gravity concentration and flotation, the results in general have been characterized by low ratios of concentration and difficulty in making a throwaway tailing.

The frequent occurrence of uranium mineralization as incrustations in fracture planes and as soft, pulverulent coatings on sand grains long ago suggested the possibility of concentrating the uranium by scuffing the ore to abrade the uranium minerals without breaking the relatively harder gangue particles. This selective grinding, commonly referred to as attrition grinding, can be done wet or dry. The fine fraction obtained by size classification of the ground material is enriched in uranium. The basic considerations and mechanisms for attrition grinding of carnotite ores were given in a Bureau of Mines Bulletin published in 1913.^{5/} Examples are recorded in which Colorado Plateau carnotite ores that assayed 2 percent U_3O_8 were upgraded to between 4 and 8 percent U_3O_8 with a 50 percent recovery. The unusual emphasis on grade at that time is explained by the fact that the concentrates were shipped to Europe for processing.

More recent work at the Intermountain Experiment Station has been focused on ores assaying about 0.1 percent U_3O_8 . Methods employed included wet scuffing followed by fractionation in classifiers and cyclones, and dry attritioning in a laboratory disintegrator followed by air classification. Recovery of 70 to 80 percent of the uranium in concentrates assaying 0.2 to 0.4 percent U_3O_8 were obtained from the amenable ores. Wet and dry procedures gave about the same results. Power requirements were lower in wet attritioning and sizing, but wet methods entail a solids-liquid separation step employing a flocculant to accelerate thickening and filtration of the slime concentrate.

^{5/} Moore, R. B., and Kithil, K. L., A Preliminary Report on Uranium, Radium, and Vanadium: Bureau of Mines Bull. No. 70, Dec. 1913.

Physical upgrading of uranium ores by mechanical means is being done by Vanadium Corporation of America in Monument Valley employing a simple attritioning procedure.^{6/} Ore assaying 0.05 to 0.10 percent U_3O_8 is lightly ground in a rod mill, and fractionated into sand and slime using a spiral classifier. Slime entrained in the classifier sands is scalped out in a cyclone and returned to the classifier. The sand discharge from the cyclone is discarded. The enriched slimes from the classifier are thickened, using a flocculating agent to accelerate the settling rate, then filtered and dried. About 60 to 70 percent of the uranium is recovered in a product assaying 0.25 to 0.30 percent U_3O_8 , which becomes part of the feed to VCA's Durango plant.

A serious drawback to concentration of uranium ores by attrition grinding, is that the slime concentrate presents special handling problems in conventional acid or carbonate leaching processes. The handling problems are minimized by salt roasting which improves settling and filtering of the slimes.

Quite good results were attained by the Bureau station in Tucson in floating a carbonaceous uranium concentrate from asphaltic ores of the Temple Mountain district in Utah. The tailing assay was comparable to that obtained by conventional acid leaching, but the ratio of concentration was only 4 to 1. Since the flotation concentrate must still be burned and the calcine leached with acid, no clear-cut economic advantage was established for the preflotation step.

Similar results were obtained on low-lime sandstone ores containing carnotite, autunite, torbernite, or uranophane as the principal uranium mineral. Flotation of the uranium from the finely ground ores with a fatty-acid type collector gave a severalfold enriched concentrate but a throwaway tailing was difficult to make because of uranium losses in the slime. Further improvement in the flotation of such ores is contingent upon development of a more effective and specific collector for slimed uranium minerals.

^{6/} Brinker, Fred A., Concentration of Vanadium and Uranium Ore at Monument No. 2 Mine of Vanadium Corporation of America. Paper presented at Western Mining Conference of the Colorado Mining Association, Denver, Colo., Feb. 3, 1956.

Considerable research has been done to develop a method for selective flotation of calcareous ores of the Lukachukai district assaying about 0.20 percent U_3O_8 and 10 to 12 percent $CaCO_3$. Batch flotation tests were supplemented by continuous runs in a 100-pound-per-hour pilot plant at the Intermountain Experiment Station. The continuous test unit was operated around-the-clock for several weeks. The ore was ground in a rod mill to 100 mesh and floated to recover the small amount of uranium-bearing carbonaceous material which was subsequently combined with the uranium concentrate. The calcite was then floated and double cleaned to give a calcite concentrate. The calcite rougher and cleaner tailings comprised the feed for uranium flotation. A typical single-shift run showed that 78 percent of the uranium could be recovered in a concentrate that represented 27 percent of the weight and assayed 0.6 percent U_3O_8 and 11 percent $CaCO_3$. The calcite product represented 13 percent of the weight and assayed 0.3 percent U_3O_8 and 57 percent $CaCO_3$. About 18 percent of the uranium and 72 percent of the lime reported in the calcite product. The throwaway tailing assayed 0.015 percent U_3O_8 and less than 1 percent $CaCO_3$. The continuous tests made with raw or reclaimed water showed that a partial separation of carnotite from calcite could be made in an upgraded product while making a throwaway tailing. The results, however, were not attractive enough to justify the complicated flowsheet.

The alternative purchasing schedule established several months ago for the Moab and Monticello AEC depots, whereby the seller can elect waiver of the lime penalty in lieu of payment for vanadium, may be expected to increase deliveries of high-lime ores that will heighten interest in lime flotation studies. Calcium carbonate, usually called lime in uranium industry parlance, is a deleterious constituent in uranium ores treated by salt roasting or acid leaching. In the salt roast process used for vanadium-uranium ores, the calcite results in high vanadium and uranium losses. The objection to calcite as an acid consumer in acid processing needs no elaboration. Selective removal of lime by flotation would appear to offer a ready solution to the lime problem in certain ores.

Flotation of a throwaway lime concentrate has not been too successful. Batch and small-scale continuous flotation tests have shown that the best that can be expected from a feed assaying 15 percent $CaCO_3$ and 0.28 percent U_3O_8 would be a lime concentrate assaying 80 percent $CaCO_3$ and 0.10 percent U_3O_8 . About 65 percent of the lime could be rejected with a loss of 6 percent of the uranium. A lower uranium-lime concentrate was made in some tests, but only at the expense of lime rejection.

The success enjoyed by carbonate leach plants working on high-lime uranium ores and the development of sufficient ore supplies to warrant both acid and alkaline units in an integrated plant, suggests the alternative possibility of splitting the ore into a low-lime fraction for acid leaching and a high-lime fraction for treatment in an alkaline circuit. A generalized flowsheet, illustrating such a combination of unit operations, is shown in figure 1.

Splitting the lime ores into high- and low-lime fractions without regard for uranium distribution presents no particular problem. Tests on Big Indian Wash ores, assaying 13 to 28 percent CaCO_3 , demonstrated that nearly 95 percent of the lime could be floated into a concentrate containing about 30 percent of the uranium in the ore. The tailing contained only 1 to 2 percent CaCO_3 . Laboratory tests showed that these flotation products responded well to respective carbonate and acid leaching. Incidental oxidation during flotation processing appears to have a beneficial effect on the subsequent carbonate leach of the calcite concentrate. A prefloat for removal of carbonaceous matter and sulfides, which frequently contain a disproportionately high percentage of the uranium, is a logical accessory to lime flotation and offers the possibility of separate treatment for refractory components of the ore. Testing is being done to establish whether the flotation reagents would be harmful in subsequent processing of the flotation products, with regard particularly to fouling of ion exchange resins.

When vanadium recovery is not a consideration, the premises for justifying selective flotation of lime are that better uranium extraction or lower costs can be achieved by processing a part or all of the ore in an acid circuit than by treating all of the ore in an alkaline circuit. As alkaline leaching works well on the majority of ores containing 10 to 25 percent lime, close figuring would be needed in most instances to establish an advantage for lime flotation. Exceptions are recognized in plants already committed to an acid circuit or if flotation of a throw-away calcite concentrate should prove to be practicable.

HYDROGEN PRECIPITATION OF URANIUM FROM SODA ASH SOLUTION

Uranium is normally recovered from soda ash solution by addition of an excess of caustic soda to precipitate sodium diuranate. After filtering the precipitate, the excess caustic is converted to sodium carbonate and bicarbonate for recycling to the leach by carbonating the filtrate with flue gas. Since vanadium is not precipitated, it builds up with progressive recycling of the leach solution. Uranium precipitation is incomplete if the solution contains more than 2 or 3 grams per liter of vanadium, and therefore solutions high in vanadium require another processing step to remove the vanadium as ferrous or lead vanadate prior to caustic precipitation of the uranium.

Considerable research has been done on the recovery of uranium from pregnant carbonate solutions using hydrogen gas as the reductant. Reduction is carried out at 100° to 150° C. under a hydrogen partial pressure of 50 to 200 pounds per square inch. The reaction is catalyzed by nickel powder or shot and the rate of reaction can be controlled by the amount and form of the catalyst. The initial work employing nickel powder catalyst was done and reported by Professors Forward and Halpern of the University of British Columbia. ^{7/} Subsequent work by the National Lead Co. at the Winchester Laboratory of the AEC developed the nickel shot catalyst which is more easily separated from the uranium precipitate than the powder catalyst. The hydrogen reduction procedure gives a low-uranium barren solution regardless of the vanadium content of the leach solution. Unfortunately, however, the vanadium coprecipitates with the uranium.

The pressure hydrogen reduction procedure has an advantage over caustic precipitation in that vanadium does not interfere with precipitation of uranium, and the solution for recycle is free of uranium and vanadium and needs only makeup addition of the soda ash

^{7/} Forward, F. A. and Halpern, J., Treatment of Uranium Ores by Basic Pressure Leaching Methods. Paper presented at A.I.M.E. Meeting, New York City, February 15, 1954.

consumed in the leach. It has the disadvantage of requiring pressure equipment, a nickel catalyst which must be separated from the precipitate, and hydrogen which is less readily available than the flue gas used in caustic precipitation. The method is unattractive for high-vanadium ores as the precipitate must be reprocessed to separate uranium and vanadium.

PRESSURE OXIDATION LEACHING

To take advantage of the presence of sulfide minerals in some uranium ores the Canadians and Australians, in particular, have examined pressure oxidation leaching. The aqueous pulp of the ore is treated with air or oxygen at elevated temperature and pressure, thereby oxidizing the sulfide minerals to produce the acid needed for dissolving the uranium minerals. A paper by Forward and Halpern ^{8/} described the conditions and controls employed. General conditions for extracting 90 to 95 percent of the uranium from ores assaying 0.1 to 0.6 percent U_3O_8 and 1 to 13 percent sulfur were given as 4 to 6 hours leaching at 130° C., and an oxygen partial pressure of 10 pounds per square inch. The feed was ground to about 60 percent minus 200 mesh and leached at 65 percent pulp solids.

The principal advantage of autogenous leaching appears to be acid generation. Presumably, if insufficient sulfides are present, pyrite or sulfur could be added to supply the acid required for leaching. Also, ores that are refractory to atmospheric leaching may respond better to the more rigorous conditions of the pressure leach. Disadvantages are the higher capital and maintenance costs of pressure equipment. So far as known only laboratory work has been done on the process. Experience with pressure oxidation leaching of cobalt concentrates at Calera's Garfield Refinery, under more severe conditions than contemplated for uranium ores, may provide guidance for assessing the practicability of pressure oxidation leaching of uranium ores on an industrial scale.

^{8/} Forward, F. A., and Halpern, J., Acid Pressure Leaching of Uranium Ores: Journal of Metals, March 1955, pp. 463-466.

AMMONIUM CARBONATE LEACHING

Alkaline processing of uranium ores has several advantages over acid treatment because of the inherent selectivity of the alkaline solution for uranium, the bland nature of the lixiviant which makes it unnecessary to use expensive corrosion resistant construction materials, and the relative ease of recovering the uranium from solution and regenerating the leach liquor.

The alkaline solution used currently by industry for carbonate leaching contains 5 to 10 percent sodium carbonate and 1 to 2 percent sodium bicarbonate. Recovery of uranium from solution with regeneration of the leach liquor by caustic precipitation and the possibility of using pressure hydrogen reduction have been previously mentioned. Work also has been done on uranium precipitation and solution regeneration using such processes as diaphragm electrolysis, mercury cathode reduction, and ion exchange. If ammonium carbonate is used in place of sodium carbonate for leaching, the uranium can be recovered by sweeping the solution with steam to reduce the ammonium carbonate concentration and precipitate uranium oxide with the ammonia being recovered for reuse.

Research work has been done by Battelle Memorial Institute to develop an ammonium carbonate leaching process incorporating this attractive recovery and regeneration procedure. A generalized flowsheet is given in figure 2. Ore ground to minus 100 mesh is leached in towers or autoclaves at 50 percent solids in a solution containing 100 grams of ammonium carbonate and 20 grams ammonium bicarbonate per liter. Whether leaching is done at elevated or atmospheric pressure, a closed system must be used to prevent loss of ammonia. The pregnant solution is separated from the leach residue by countercurrent decantation. Uranium hydrates, precipitated by steam stripping the pregnant solution, are recovered by filtration. The ammonia and part of the CO_2 in the gas from steam stripping are absorbed in the filtrate, and the solution is further carbonated with flue gas for recycling to the leaching step.

The principal advantage of the ammonium carbonate process over conventional soda ash leaching appears to be in the ease of precipitation and potential reagent savings. The main disadvantage is the necessity for maintaining a gas-tight system to prevent ammonia loss. Additionally, further studies of the process at the Winchester laboratory by National Lead Co. revealed uranium extraction problems when the ores contain vanadium.

CHAR-IN-PULP

A process embodying the essential features of both the resin-in-pulp and solvent extraction of slurries is the char-in-pulp system tested by the Arthur D. Little Co. The procedure is similar to the char adsorption process used by the Golden Cycle Corp. at Victor, Colo. for recovering gold from cyanide leach pulps. The essential difference is that, whereas the char selectivity adsorbs the gold complex, in the uranium process the char serves as a carrier for dioctyl pyrophosphoric acid (OPPA), or a similar organic phosphate, that extracts the uranyl cation. The loaded char is screened from the barren pulp and stripped of uranium by countercurrent contacting with a soda ash solution. The char after stripping is recycled to the adsorption step. Uranium is recovered from the soda ash strip solution by neutralization or caustic precipitation with the barren strip liquor being regenerated and recycled. Figure 3 is a flowsheet of the process. Because of silica poisoning, periodic reactivation of the char may be necessary.

Potential advantages of the char-in-pulp over the conventional resin-in-pulp procedure are that it should be possible to combine the leaching and adsorption steps in a single operation and thus avoid a sand slime separation. An advantage over solvent extraction of leach slurry is that organic entrainment losses are minimized. More research may overcome the disadvantages of organic phosphate loss owing to solubility in the aqueous phase, incomplete uranium extraction, and the large quantity of char that must be handled because of low uranium loadings.

NONAQUEOUS EXTRACTION

The development of vegetable gums and synthetic flocculants for accelerating the thickening and filtering of leach pulps has taken much of the pain out of liquid-solid separations in the uranium ore processes.^{9/} Even so, countercurrent decantation or filtration involve capital and operating expenditures that the operator would like to avoid. Considerable thought and research have gone into nonaqueous extraction methods as a means of circumventing an aqueous system in hopes of shortcutting the recovery routine.

Selective leaching of uranium directly from ores by nonaqueous reagents has not shown much promise. However, by treating the ores with mineral acids in the presence of very little water, it is possible to solubilize or convert the uranium to forms which are extractable by nonaqueous solvents. We will describe two so-called nonaqueous systems, both of which involve the use of about 10 percent of water in the acid-curing step. Work on the acetone-chloride extraction process, illustrated in figure 4, was done at Battelle. In this procedure, ore crushed to 10-mesh is pugged with hydrochloric and sulfuric acids for curing to solubilize the uranium. The cured material is fed continuously into a pool of acetone in an enclosed drag-type extractor. The uranium, iron, vanadium, calcium, and other metallic ions, are absorbed as chloride complexes by the acetone which overflows the extractor. The leach residue, which settles with extreme rapidity, is dragged over a heated drying section at the upper end of the extractor where entrained acetone is volatilized. The acetone vapor is condensed for return to the extractor pool. The loaded acetone is gassed with anhydrous ammonia, which precipitates the uranium, iron, vanadium, and calcium hydroxides. These are removed by filtration and regenerated acetone is returned to the drag extractor. The final concentrate after washing is low grade and contains about 10 to 15 percent U_3O_8 , depending upon the concentration of impurities. Since acetone is not selective for uranium chloride over other metallic chlorides, the process is primarily suited for upgrading. Advantages

9/ Rosenbaum, J. B., and Clemmer, J. B., Accelerated Thickening and Filtering of Uranium Leach Pulps, Proceedings of the International Conference on Peaceful Uses of Atomic Energy, August 1955, Vol. 8, pp. 38-44.

of the system are that little water is required, overall reagent requirements are not excessive, and a relatively small installation might be practicable for upgrading ores in an isolated location. Disadvantages are that both hydrochloric and sulfuric acids are needed, corrosion problems are likely to be severe, a gas-tight system is needed to minimize acetone losses, and the precipitated hydroxides are bulky and low grade.

Another nonaqueous extraction process was developed by National Lead Co. at the Winchester, Mass. laboratory of the AEC. This procedure uses tributyl phosphate (TBP) to extract uranyl nitrate and gives a high-grade uranium concentrate. The ore is crushed to minus 10 mesh and pugged with sulfuric acid and a little water. The pugged material, after curing for an hour or so to dissolve the uranium, is wetted with an ammonium nitrate solution and the damp mass is leached on a moving belt by percolation with 5 percent TBP dissolved in kerosene. The leach residue is washed with a volatile organic solvent to recover the entrained TBP for return to the system. The loaded organic phosphate from the percolation leach is stripped with water to give an uranyl nitrate solution for precipitation with ammonia. After filtration, the ammonium diuranate cake is calcined to drive off ammonia and give a product assaying 99 percent U_3O_8 . The ammonium nitrate solution from the precipitation step is recycled for wetting the acid-cured material. The process flowsheet is given in figure 5.

Advantages of the process are that little water is needed, a minimum number of steps are employed in obtaining a high-grade precipitate, and reagent requirements are low. Disadvantages are that the acid-cure procedure is not particularly effective on ores requiring an oxidant for good uranium solubilization, and an economical method for recovering entrained TPB and kerosene from the percolation leach tails has not been evolved.

SOLVENT EXTRACTION OF SULFURIC ACID ELUATE

Ion exchange processes for recovering uranium from acid-leach solutions or pulps are based upon selective adsorption of an uranyl anion complex with subsequent elution using a mineral acid or an acidified salt solution. The elution step removes the uranium and regenerates the resin for the adsorption cycle. Uranium is

recovered from the eluate by neutralizing with ammonia, caustic soda, or magnesium oxide. The precipitation of uranium from an ion exchange eluate, assaying from 7 to perhaps as high as 15 grams per liter U_3O_8 , involved handling relatively large quantities of solution. The cost of chemicals for elution and precipitation are important items. Even though a portion of the barren eluate may be recycled in some systems, considerable expense may be entailed in disposal of the remainder of the eluate to conform with stream pollution regulations. Reextraction of the ion exchange eluates with an organic solvent was examined in small-scale continuous test units at the Intermountain Experiment Station, and appeared to offer an overall advantage in reagent cost over the direct precipitation of the eluate. Another advantage was that a lower grade eluate would be acceptable and hence less ion exchange equipment would be needed. This exploratory work was subsequently expanded into a more comprehensive investigation at the Winchester laboratories of the Commission. A combination process involving sulfuric acid elution, followed by solvent extraction of the eluate, is illustrated in figure 6.

The loaded resin is eluted with 1 molar sulfuric acid and the eluate, assaying about 7 grams per liter U_3O_8 , is reextracted with an organic phosphate in kerosene. The sulfuric acid raffinate, which contains about 0.2 gram per liter U_3O_8 , is returned to the ore leaching step to fully utilize the acid and recover the residual uranium. Complete removal of uranium from the eluate is unnecessary in the combination process. The loaded organic is stripped with acid or carbonate solution to give a barren organic for recycle. A final uranium product is obtained by precipitation of the loaded strip liquor assaying 65 grams per liter or more in uranium.

Advantages of the process are that savings in the ion exchange plant are possible owing to acceptability of lower eluate loading, eluate disposal problems are minimized, and a final uranium product of higher purity can be attained at a lower reagent cost. Disadvantages are the corrosive action of the acid solution and complexity of the flowsheet.

THICK SLURRY SOLVENT EXTRACTION

Provided organic entrainment losses can be suitably controlled, extraction of uranium by organic solvents from a thick leach slurry offers an ultimate in process simplification. The process as given in figure 7 was developed at the Salt Lake City station as an offshoot of the research on clear liquor and slime slurry solvent extraction.^{10/} Ore ground to about 35-mesh is leached at 40 to 60 percent solids with sulfuric acid in a conventional manner with or without an oxidant as needed. A reducing agent such as iron powder or sodium thiosulfate may be added to the final leach tank to reduce ferric iron to the ferrous form and thereby minimize iron contamination in the final product. The thick leach slurry at a pH of 1.0 or less is then contacted countercurrently with a dilute solution of dodecyl phosphoric acid in kerosene. Internal mixer contactors, which combine mixing and phase separation in the same apparatus, are used for the extraction and stripping steps. Barren leach slurry is treated for solvent recovery, if necessary, before being discarded. The loaded organic is stripped by countercurrent contacting with hydrochloric acid, and the regenerated organic is recycled to extraction. Loaded acid strip liquor assaying about 60 grams per liter U_3O_8 is concentrated to 350 grams per liter U_3O_8 by evaporation and 70 to 80 percent of the hydrochloric acid is recovered for reuse in the stripping step. An ammonium diuranate product is obtained by treating the concentrated uranyl chloride solution with ammonia.

Advantages over clear liquor or slime slurry recovery procedures are that fewer steps and less equipment are needed. The method has the advantage over the nonaqueous procedures in that conventional agitation leaching can be used rather than acid curing which does not always give good uranium extraction and has proved expensive in terms of labor and reagents. The salient disadvantage of thick slurry extraction is that a practicable method for keeping organic entrainment losses within acceptable limits has not yet been devised. Research on thick slurry extraction has been attractive enough to warrant a continuation of the work.

^{10/} Ross, J. R., Rosenbaum, J. B., and Clemmer, J. B., Solvent Extraction of Uranium from Leach Liquors and Pulps. Paper presented at Uranium Subsection meeting of A. I. M. E., Moab, Utah, April 6, 1956.

Fig. 1 - Generalized flowsheet for flotation of lime from uranium ores,

Fig. 2 - Generalized flowsheet of Ammonium Carbonate Leaching Process.

Fig. 3 - Generalized flowsheet of Char-in-pulp Process.

Fig. 4 - Generalized flowsheet, Acetone-Chloride Nonaqueous Process.

Fig. 5 - Generalized flowsheet - TBP-Nitrate Nonaqueous Process.

Fig. 6 - Generalized flowsheet Solvent Extraction of Sulfuric Acid Eluate.

Fig. 7 - Generalized flowsheet - Thick Slurry Solvent Extraction Process.

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