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G. M. Ritcey[a]
[a] Canmet Energy, Mines and Resources Canada, Ottawa, Canada

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Solvent Extraction — Projection to the Future

G. M. RITCEY

CANMET
ENERGY, MINES AND RESOURCES CANADA
OTTAWA, CANADA K1A 0G1

ABSTRACT

The process of purification of metal-bearing solutions using solvent extraction has proven to be one of the major improvements to the optimization of the metallurgical flowsheet. Numerous improvements have been made throughout the solvent extraction circuit, as well as considerable research on the subject throughout the world which could, if implemented, add considerably to the overall understanding and viability of the process. The paper will discuss the various interactions within the process that could ultimately prove to be the flowsheets of the future.

INTRODUCTION

The solvent extractive process has been industrially used for about 35 years. The process, initially used for uranium, then copper and subsequently base metals, precious metals and less common metals, now enjoys a wide use. Solvent extraction has been used to:

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- concentrate initially-dilute values
- separate metals of similar types in solution
- purify solution for subsequent recovery of pure products
- decrease evaporation costs.

Thus the process, when integrated into the overall hydrometallurgical flowsheet, is composed of essentially the flowsheet shown in Fig. 1. The purification stage, for example using solvent extraction, is a very small but very important part of the overall integrated flowsheet and occupies only a minor part of the mill operation. Therefore any economic impact that the solvent extraction plant might have on the total plant will result only after a major improvement in process technology. Figure 2 shows the typical solvent extraction purification part of the flowsheet. Following contact of the clarified aqueous solution with the solvent for extraction of the metal or metals, the solvent may be scrubbed with an aqueous phase to improve purification, or go directly to the stripping stage. Stripping is generally accomplished with an aqueous solution, selected to be amenable for the subsequent product recovery by electrowinning, crystallization or precipitation.

This paper will examine each of the areas in the solvent extraction process and discuss possibilities whereby process modifications could result in significant improvements to the flowsheet. Such improvements of interest and concern to the operating plant include economics, energy and environment.

FEED SOLUTION AND PREPARATION

Considerable expense is incurred in present plants to produce a clarified feed so that minimal emulsions and crud are formed in

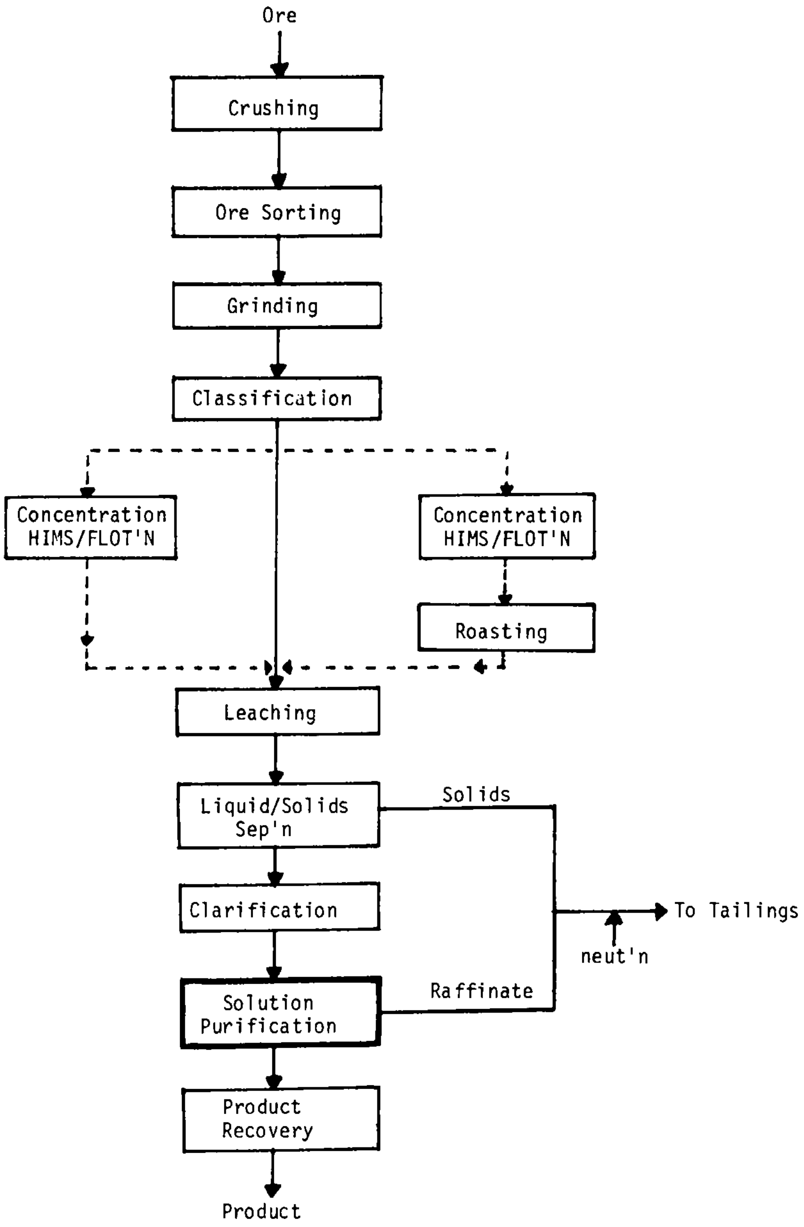

Figure 1 - Integrated Process Flowsheet

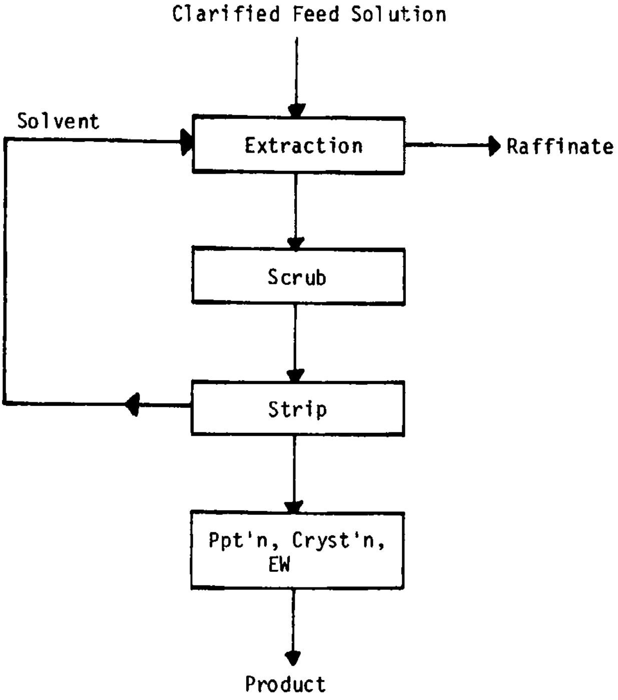

Figure 2 - Typical Solvent Extraction Purification Schematic

the subsequent extraction stage. These cruds and stable emulsions are due to the constituents of the feed solution, solubilized or in suspension, in combination (1,2) with the shear or energy input during extraction(1-3). Thus to minimize emulsion and crud, very expensive liquid-solids separation and clarification stages are necessary in the preparation of the feed. These separation stages account for approximately 50% of the capital and operating costs in the circuit(4,5). Thus a significant reduction in the capital and operating costs would result if the costly liquid-solids operation could be eliminated.

SOLVENT-IN-PULP

Solvent-in-pulp extraction (SIP) has the potential to be a successful alternative,(5) to eliminating the expensive liquid-solids operation. Further research is necessary to minimize sorption losses on the gangue particles. The research is necessary on the development of specific extractants; on equipment design and operation; the use of surfactants(6); and an understanding of the surface chemistry and the relationship between mineralogy and the wetting properties of the solvent. With success the process could be viable within a few years. In addition to the economic savings accrued through the elimination of liquid-solids separation, soluble metal losses to the gangue tailings are eliminated, thus increasing the per cent metal recovery. Increased revenue and decreased energy requirements thus result.

A flowsheet incorporating SIP that has been applied successfully on a pilot scale for the treatment of uranium solutions(5) is shown schematically in Fig. 3.

SOLVENT SYSTEM

Extraction

The extractants presently in use are generally anionic, cationic or solvating reagents dissolved in diluents with the occasional aid of a modifier to improve phase separation and solubility of the metal-organic species. However specific extractants have not been developed with much success except for the chelating ex-

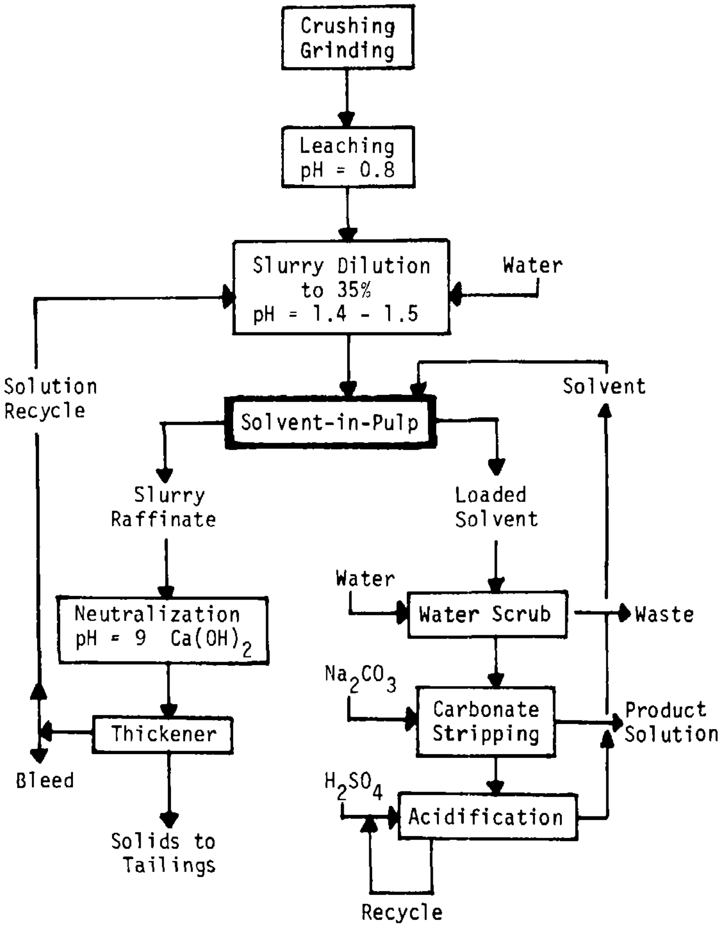

Figure 3 - Schematic of Proposed Solvent-in-Pulp Extraction Circuit

tractants for copper. Unfortunately, most of the chelates suffer from slow kinetics in acidic solutions, but they do possess good kinetics in alkaline systems. In the absence of other interfering ions, tertiary amines are selective for uranium; and similarily DEHPA can be selective for various metals in the absence of competing metals at certain pH values. The neutral extractants such as TBP or TOPO are not really very specific. Variations of the phosphorus-type extractants have shown an increasing selectivity for Co/Ni separation with change from phosphoric - phosphonic - phosphinic(7). However, with the increase in separation factor (by rejecting nickel), the kinetics also decrease(8). There have been numerous investigations on the possible use of mixed extractants to improve the extraction by synergism(9-12). Not only may the extractive equilibrium be affected, but also the kinetics might be significantly increased. By improving on the kinetics, the possibility of separation of similar metals can increase, thus decreasing the number of stages required for purification of the loaded solvent. Accelerators have also been used to improve the kinetics in LIX extractants(13).

There have been numerous extractants developed to date, only a small number of which have been industrially utilized. This fact well illustrates the existing gap between the chemist involved in extractant design and development and the hydrometallurgist, a gap which could be bridged to the benefit of both the chemical industry and the mining industry with interdiciplinary cooperation. Among the classes of compounds used as extractants to date are oximes, amides, amines and organophosphorus extractants. Each extractant possesses certain attributes which makes it a valuable reagent to a particular system or systems. We already have some excellent molecules available for most extraction situations, with only selectivity, equilibrium and kinetics somewhat lacking. It would therefore be interesting to speculate, based on synergism of mixed extractants, what could be produced by constructing a molecule combining the most desirable features of two or more single extractants. Minimum organic solubility in the aqueous phase must be maintained, and increased metal loading would be desirable. Such characteristics of mixed molecules seem feasible, and if attained would effectively reduce plant operation costs. Such systems might also be tailored to provide for co-extraction and permit selective stripping. Figure 4 could thus incorporate decreased stages for separation of two metals as well as a total reduction in plant area. Thus again a reduction in energy requirements and costs would be achieved.

Scrubbing

Purification of the loaded solvent can usually be accomplished easily by judicious selection of the type of scrub solution and the method used in scrubbing. If the extractant has been properly selected for its extraction and scrubbing properties, any inadequacies in metals discrimination during extraction can be overcome during scrubbing. It may therefore be wrong to select an extractant which is presumably more selective than another for metals discrimination if the trade-off is poor kinetics, poor equilibrium and therefore poor economics. It could be more profitable to select an extractant with fast kinetics for extraction and one which is amenable to scrubbing to produce the high purity desired of the product. Figure 5 shows what could result from the choice of the extractant system relative to the circuit size. With a system with high rates of mass transfer, a centrifugal contractor could be used

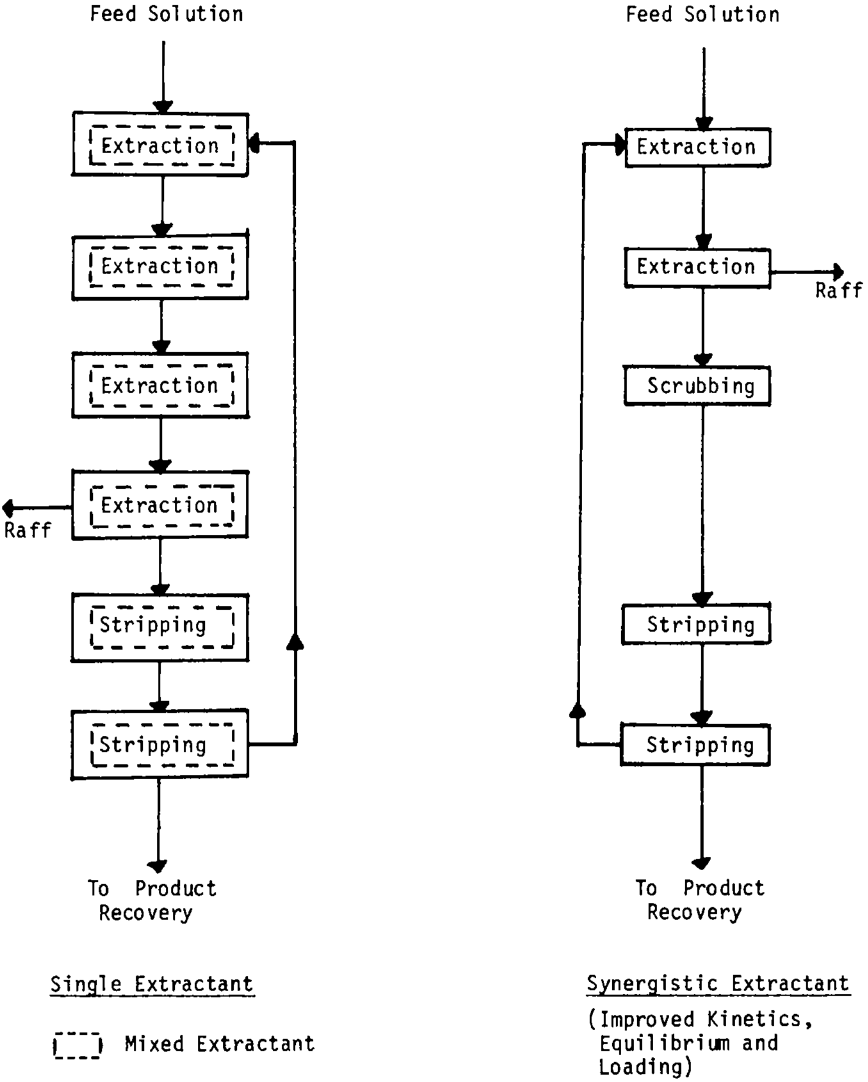

Figure 4 - Comparison of Process Steps Using Synergistic Extractants

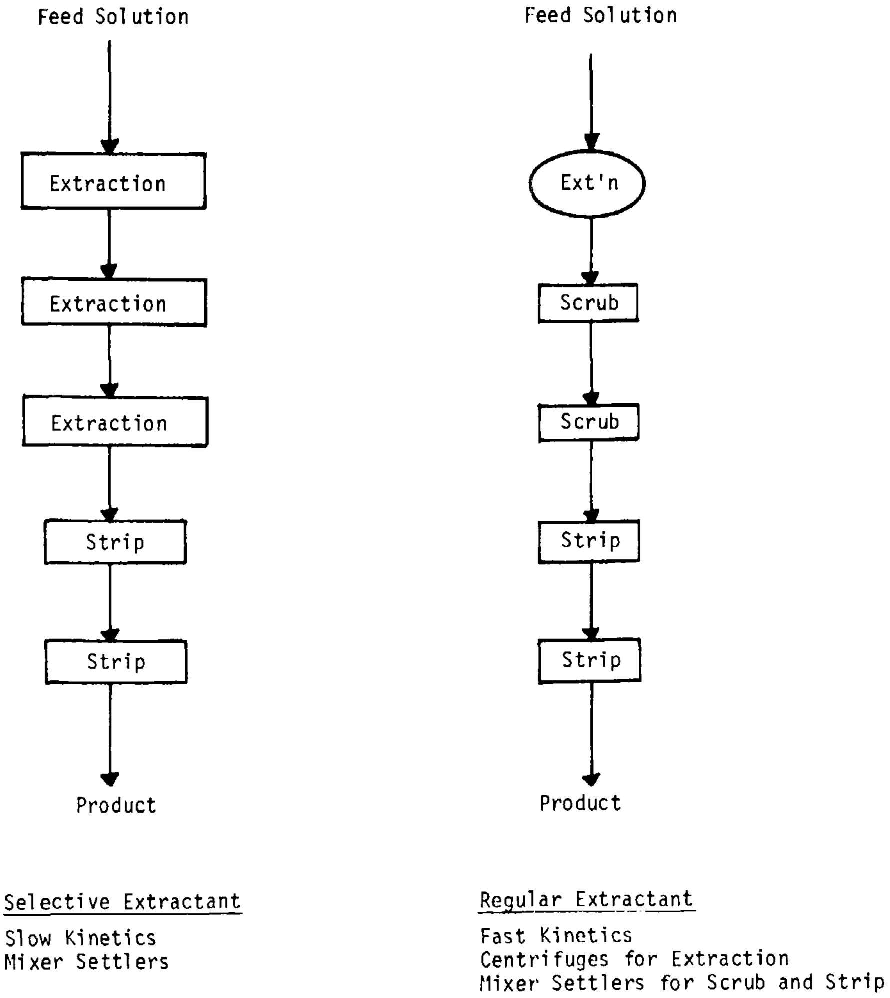

Figure 5 - Extractant Choice and Circuit Size.

to advantage, not only to extract the metal in question, but also to reject undesirable impurities which may have slower kinetics. Subsequent scrubbing is carried out in centrifuges or mixer settlers. The result could be a substantial reduction in plant size and solvent inventory.

Stripping

Conventionally, stripping is performed by an acidic or alkaline solution dictated by the final product demanded of the process. Thus an H_2SO_4 strip solution is used as the medium for subsequent electrowinning, crystallization or precipitation; nitric or hydrochloric are used to produce metal oxides by evaporation and denitration or dechlorination. Carbonates, or oxalates are used to produce sodium or ammonium salts of the metals or oxides by decomposition. These routes, all technically sound and producing the desired final products, nevertheless constitute several process stages in the plant. Also, if pH control is difficult, as it is with ammonium sulphate for uranium stripping, then uranium precipitation often occurs, causing cruds and solvent losses. This all means expensive equipment, energy, labour and possible loss of reagents. If the process could be improved and shortened to satisfy these economics, then considerable cost reductions could be possible. In recent years there has been interesting research carried out first by Burkin (14) and Shell (15) and then by Davey Power Gas (16), on the use of pressure reduction of loaded solvents. More recently Demopoulos, Distin and co-workers(17-22) have continued the investigation. Excellent results have been achieved for Cu, Ni, Co, Fe from carboxylic acid extractants (Versatic) or phosphoric acids (DEHPA) and for Cu, Ni, Co from derivatives of 8-hydroxyquinoline (Kelex 100). It is implied that this new process could be used only for metals or compounds under conditions where they could be reduced by hydrogen, to produce, e.g., Cu, Ni, Co, Au, Ag, UO_2, MoO_2, V_2O_3, etc. This technique could be particularly useful in those cases where problems are encountered in removing the metal from the organic phase by conventional stripping means, for example cobalt. A typical flowsheet utilizing pressure reduction is shown in Fig. 6. While considerable work remains to be done in this area to complete the fundamental understanding of the organic structures amenable to such a process (without affecting the stability of the reagent), the process shows decided advantages and would appear to be worthwhile pursuing. This should be especially true for the higher value products, i.e., perhaps above the price of copper. Also, an engineering design to incorporate continuous reduction would further improve the economics over batch precipitation.

With conventional stripping, the reagents are usually consumed in the subsequent process, if precipitation is used. For example, in the purification of uranium leach liquors, the uranium is stripped from the tertiary amine with a sulphuric acid or ammonium sulphate solution. This strip solution is neutralized and a uranyl compound is finally precipitated by the addition of MgO, H_2O_2 or ammonia. There is no recycle of the barren solution from precipitation. Furthermore the resultant products are not of nuclear grade purity, and so require further processing in a TBP refinery circuit. If a process could be devised to strip the uranium (or other metal) from the loaded solvent, followed by hydrolysis of the product and drying, with recycle of the precipitant, then such a process could be economically attractive particularly if further refining was unnecessary. Work at our laboratories during the past

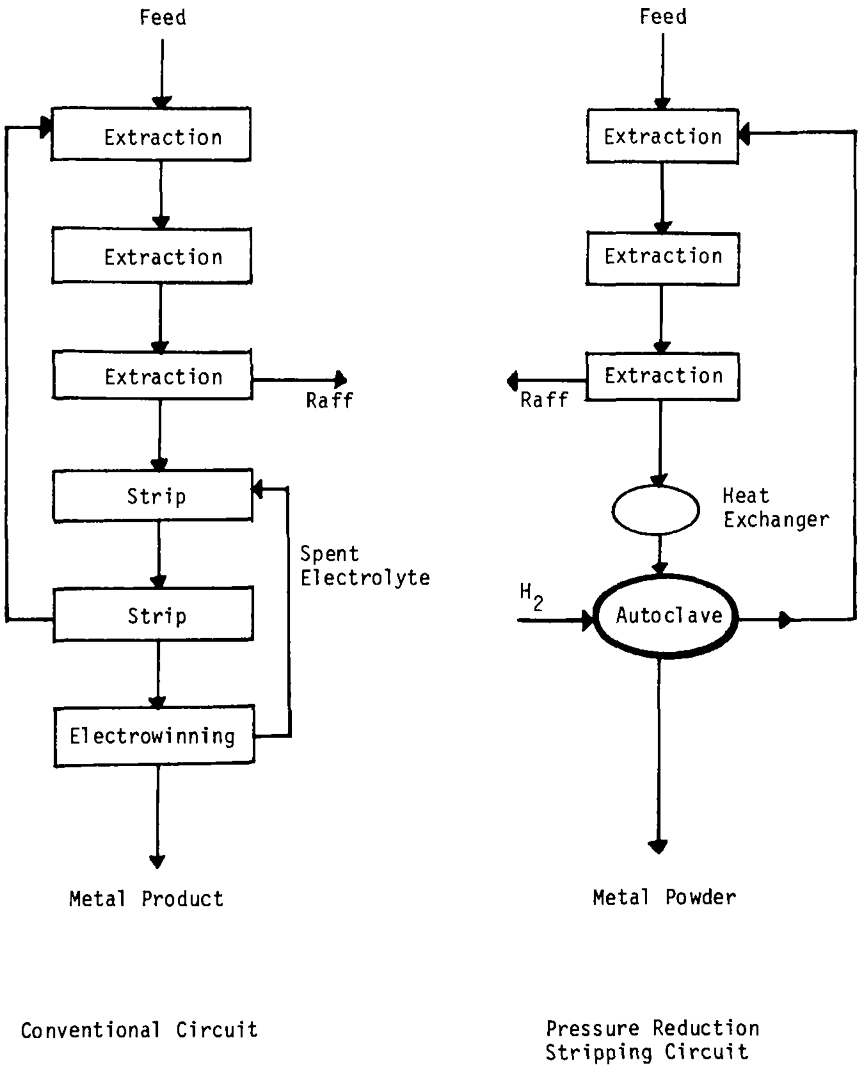

Figure 6 - Schematic Flowsheet Incorporating Pressure Stipping-Precipitation.

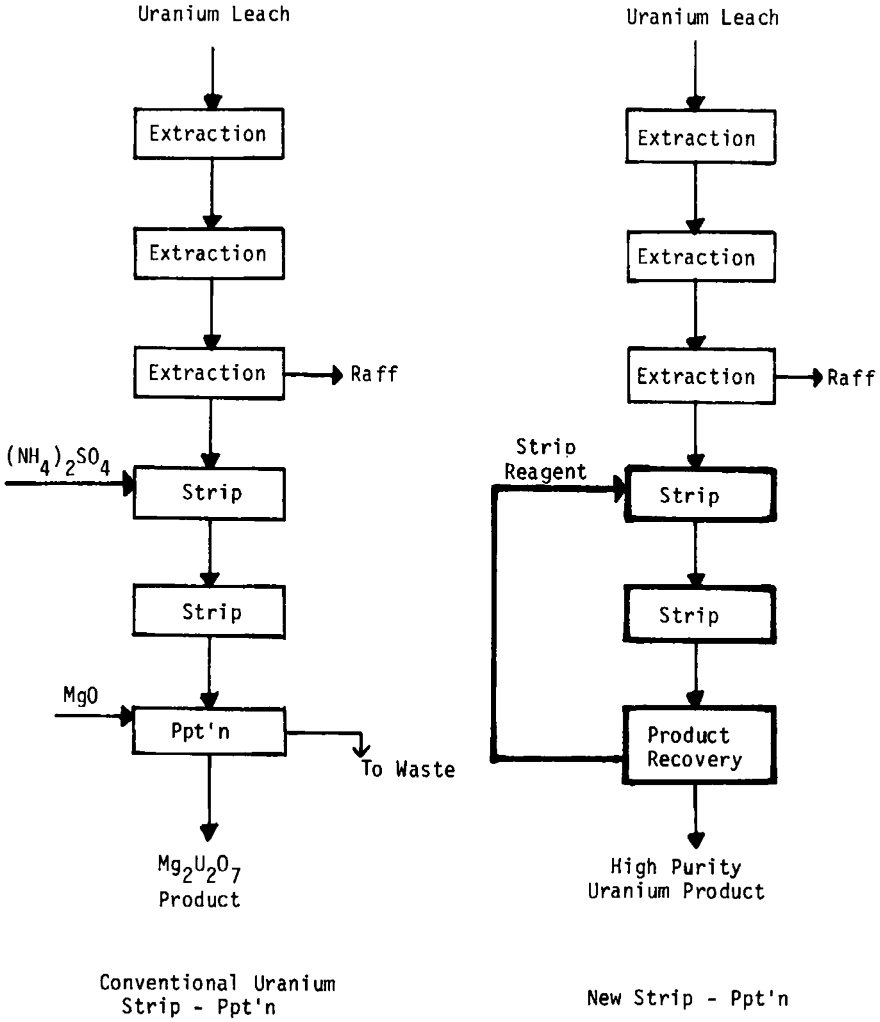

Figure 7 - Stripping-Precipitation Circuits Modification

year has evolved such a process flowsheet for the uranium circuit(3). This is shown schematically in Fig. 7, indicating a reagent cost reduction as well as the possible production of a high purity product at the mill site.

A further application of novel stripping-precipitation techniques is demonstrated by the hydrolytic stripping procedure reported by Thorsen and Monhemius(23,24). Their work indicated the possibility of precipitation of metal oxides of Fe, Ni or Cu directly from a carboxylic acid by reacting the loaded solvent with water at 150-200°C. Thus the stripping and metal recovery portion of the conventional flowsheet can be eliminated. The obvious use for such a process is in the application to iron removal as hematite in a zinc circuit, for example. Such an iron compound is thus more environmentally acceptable than either jarosite or goethite, both of which coprecipitate significant amounts of zinc and other values such as silver in the conventional route. The iron oxide could possibly be suitable for use in iron-making(24). This flowsheet is shown diagramatically in Fig. 8.

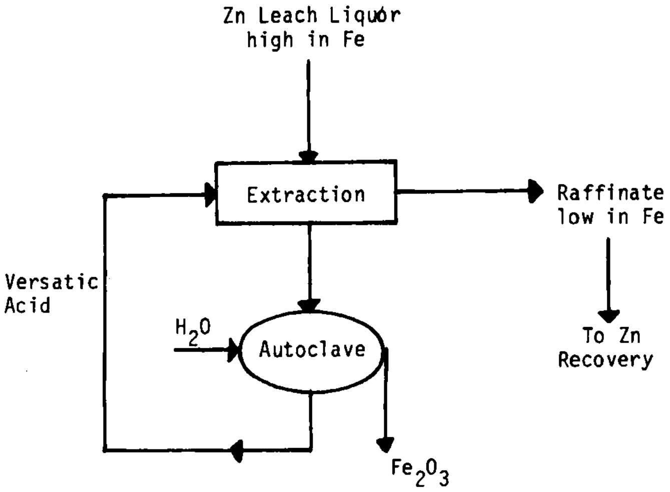

Figure 8 - Conceptual Flowsheet for Hydrolytic Stripping - Precipitation

The stripping part of the circuit (and the scrubbing) is easily seen to be a very important part of the solvent extraction circuit, with more effects on the economics and downstream part of the overall plant than in the extraction circuit. However most research efforts in the past have been directed towards the extraction circuit, to

- optimize and select the best extractant
- provide kinetic and equilibrium data on extraction
- provide solubility of extractant
- determine the dispersion and coalesence characteristics
- determine the need of a modifier and the selection of a diluent

Thus considerable research has been published on extraction only, with no real concern for the remainder of the flowsheet. If the researcher had, in many of the examples cited, gone just a little further in considering the total purification system, perhaps much of the meaningless data in the journals would not have been printed.

EQUIPMENT, OPERATION AND SOLVENT LOSSES

One cannot discuss the solvent extraction system without considering the equipment; its design and operation can have profound effects on the process. Although we now have many types of stagewise (mixer settlers) and differential (columns) contactors, plants still are being built in which insufficient consideration has been given to the selection of the contactor. The important decision always was to select the extractant system! Then the process was given over to the contractor for design. Unfortunately many plants have been built with several hundred per cent overdesign, and using the wrong equipment. More than one plant has had to shut down and replace their contactors with a design completely different from the original installation. For example, just recently a plant replaced their Kenics in-line mixers with pump-mix mixer-settlers. If a sufficiently good understanding of the physico-chemical and engineering aspects had been achieved in pilot studies, such expense would not have occurred. Because a plant may be using a certain contactor for the same process as its neighbour company, it does not necessarily follow that the plant should select the same equipment for there may be sufficient differences in the physico-chemical properties of the feed solution to warrant a different contactor. Furthermore, consideration must be given to the scrubbing and stripping stages when equipment selection is made. Unfortunately there are again sad results in plants where the contactors were selected for the entire circuit on the basis of extraction tests. It would not be unreasonable to expect that 2-3 different types of contactors could be required for a given solvent extraction process plant.

What this is leading up to is to ensure that the proper selection of the equipment has been made, and that optimum operation of the equipment is maintained in order to provide maximum mass transfer efficiency with minimum solvent losses. These solvent losses can be caused by: solubility, entrainment, volatility, and crud. These losses are, to a large part, governed by the solvent system selected, but once selected, the losses are affected by the type of contactors and the type of operation(25). Thus high temperature operation can increase soluble losses, increase volatile losses; but will improve coalescence and reduce entrainment losses. Volatility can be minimized by phase continuity choice, diluent selection and covering of the mixer settlers, use of columns, in-line mixers or other closed contactors such as the RTL (Graesser Contactor). Assuming all the above losses are minimized, the operation of the equipment is critical to provide sufficient energy input to the system in order to create the desired mass transfer. That is, if extremely small droplets are created by high energy input and resultant high shear, then stable emulsions can be produced(1). If ore particles are present in the feed stream, then the result may be a crud and consequent expensive solvent losses(1). Such a situation is often obtained in poorly engineered processes. In the past few years there has been an increased awareness and concern for the type of dispersion created by the contactor, and thus further improved contactor designs have been developed, notably the Krebs mixer settler(26) and the Davy CMS of the stagewise types(27). There is no universal contactor, and the chemical and physical characteristics of the systems relative to the dispersion and coalescence, kinetics, and mass transfer efficiency must be considered in the choice of the contactor(25).

BY-PRODUCT RECOVERY

Most hydrometallurgical process plants ignore the fact that often there are potentially valuable by-products that might be recovered in the process. These values unfortunately are often lost and impounded with the tailings, usually because the market demand is not sufficient at that time. Even if a bulk separation is made, with a separate storage area provided for future markets, considerable additional revenue could ultimately result. For example, in Canada the uranium ores in the Elliot Lake area contain considerable amounts of thorium and rare earths(28), and in the high grade Saskatchewan ores there are substantial amounts of arsenic and nickel(29). The present processes provide for the recovery of uranium only, while the tailings receive all other potential values. Small process changes could result in considerable conservation of a country's natural resources and increased revenue to the mill through by-product recovery.

ENVIRONMENT

Although the development and optimization of the metallurgical processes, including solvent extraction, has progressed through phases of concern for economics and energy, the more recent concern is for industrial plant hygiene and the environment. Therefore, as noted under equipment, the proper selection of the contactor design and proper operation is necessary to minimize solvent losses in the plant and in the effluents discharged. Poorly vented work areas and the use of low volatile solvents cause hygiene concerns in the plant and can easily be eliminated. Waste raffinates, often containing large amounts of entrained solvents, may be discharged to the environment if the equipment and its operation are not optimal. Future environmental constraints may well force the operator to provide for in-plant treatment of such raffinates to minimize the environmental concerns. Coalescers, centrifuges or perhaps activated carbon treatment may be necessary to control the amount of organic released to the environment(30). A flowsheet is shown in Fig. 9 incorporating activated carbon treatment. Naturally if centrifugal contactors can be employed in the stripping circuit, it is possible that a sufficiently-low raffinate discharge will be produced without the necessity for carbon treatment.

FLOWSHEETS OF THE FUTURE?

Up to this point we have considered possible modifications to the solvent extraction process which, in most cases, have some indications of success with perseverence. That is, most of the suggestions for improvements, although reducing the processing, energy and environmental problems, are not terribly earth-shaking.

Since this paper has the objective of "crystal-ball gazing" into the future, then we should consider the following possible scenario. We have recognized that the liquid/solids separation is expensive, but could be overcome by solvent-in-pulp extraction. Also, pressure reduction of loaded solvents has been defined for certain metals. Also let us consider the leaching stage. Some interesting work has been discussed by Van der Zeeuw(31) and by

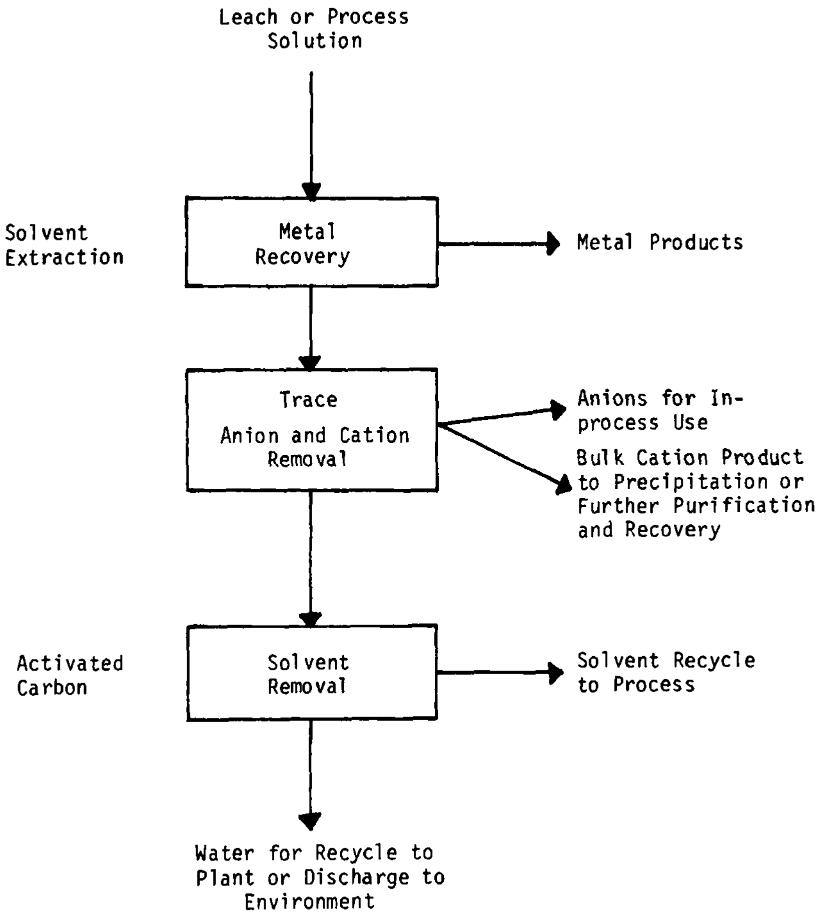

Figure 9 - Schematic Outline of Purification Route

Thorsen(32,33) on the direct leaching of zinc calcine with Versatic acid to produce an organic solution containing zinc carboxylate.

If solvent leaching for Zn or any other metal can be achieved, either directly or by the addition of solvent to the last stage of a multi-stage leach process, followed by autoclave reduction, the only major liquid-solid separations would be primary thickening and the reclaiming of the metal powder. Thus a very considerable improvement in the overall solvent extraction flowsheet, and in the entire mill operation, would be accomplished. However, much research and engineering design is necessary if the future flowsheets in Fig. 10 and Fig. 11 are to be realized.

Finally, let us consider a specific example. In the case of the conventional uranium process, where sulphuric acid is used for

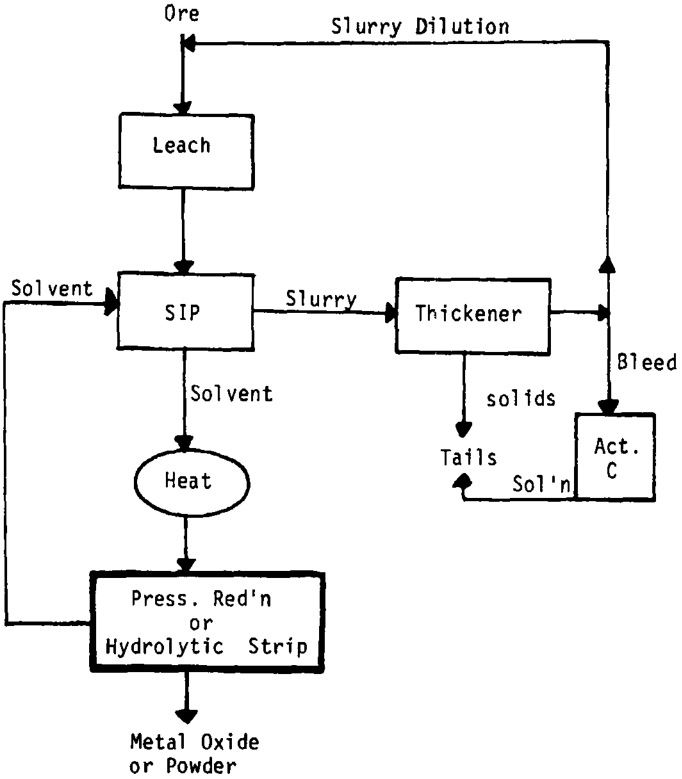

Figure 10 - Future Flowsheet Incorporating SIP and Pressure Reduction or Hydrolytic Stripping and Solution Recycle

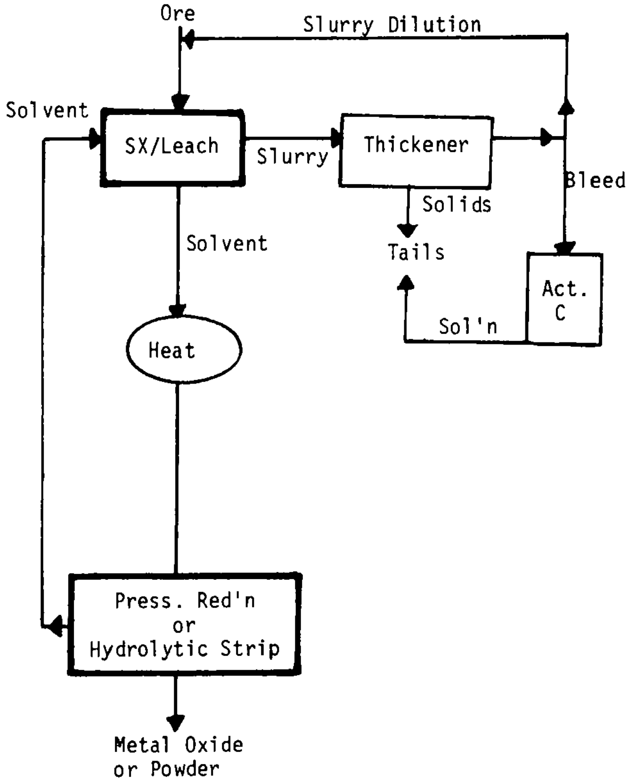

Figure 11 - Future Flowsheet Incorporating Solvent-Leaching, Pressure Reduction and Solution Recycle

leaching, essentially all the radium^{226} and other radionuclides and pyrite report in the tailings. The following is an attempt to put some of the various options together in a modified uranium flowsheet to improve the overall process and economics and reduce the high environmental costs.

If we can assume that with several modifications to the present technology the overall economics and environmental aspects will be improved, then the following is an attempt to put the various options together in a modified flowsheet using the H_2SO_4 route.

In the leaching, the ore is generally being subjected to long leaching periods, the ore requiring relatively fine grinding and the use of expensive reagents. However the only immediate improvement to increase the kinetics and therefore decrease the leach plant size is to optimize the grind vs oxidation conditions, by proper choice of the oxidant and control of EMF. Leaching under pressure, to utilize the pyrite in the ore could be an improvement to some plants, especially where the ore contains sufficient sulphide, and where freight costs for transporting H_2SO_4 to the plant are excessive. The oxidant may be oxygen, peroxide or even ozone might be beneficial in some cases. Therefore let us assume an autoclave leach plant, very short retention time of perhaps 4-6 hours, compared to many present circuits operating at greater than 30 hours and consequently a very large leach plant.

Next let us consider the very expensive liquid-solids separation stage, occupying also a large portion of the plant. A flowsheet incorporating solvent-in-pulp, for the purification stage, would eliminate most of the costs attributed to that expensive stage.

The uranium in the loaded solvent would ordinarily be stripped with ammonium sulphate (or another reagent) and the uranium subsequently precipitated from the strip liquor. Such a precipitation system is a once-through process, with no reuse of the reagent being possible. The future flowsheet, to conserve expensive reagents and produce a high purity uranium product should incorporate a stripping-precipitation cycle that would enable recycle of the strip reagent after hydrolysis of a uranium product.

In such a circuit, the tailings problem presently encountered would be minimized. Following the SIP extraction stage the slurry raffinate would be discharged to a cyclone or thickener, and the solids then given a water wash to remove sulphate followed by a wash with chloride solution. This latter wash would solubilize the radium, thus providing tailings containing perhaps 50 pCi/g Ra^{226} or less. The chloride solution containing the radium would thus be isolated in the mill by sorption on an ion exchange resin, or precipitated, and this concentrated radium subsequently shipped for burial with high level wastes. In such a modified flowsheet, a maximum of solution recycle would be accomplished, by recycling the acidic raffinate back to the leaching stage. A periodic bleed of this recycle stream would be required to control impurity build-up. In addition, another decided advantage to such a process would be the elimination of the expensive lime neutralization stage normally carried out. The schematic for such a flowsheet, for which CANMET is investigating, is shown in Fig. 12. Such a flowsheet could provide the mill operation with:

- decreased reagent costs in leaching, liquid-solids separation, solvent extraction, precipitation, and tailings neutralization;
- increased uranium recovery due to decreased soluble uranium losses normally encountered in the liquid-solids separation;

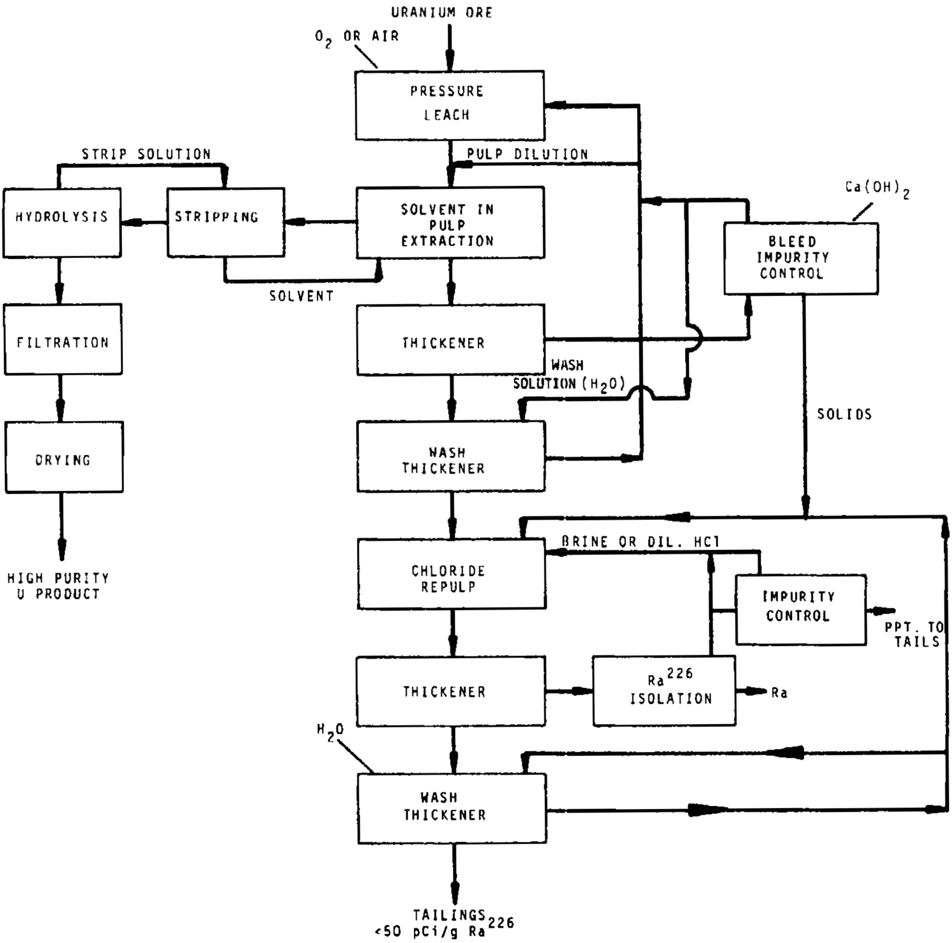

Figure 12 - Future Uranium Flowsheet Incorporating SIP, Strip-Hydrolysis and Solution Recycle.

- decreased tailings impoundment areas because no lime requirements and subsequent gypsum precipitates;
- isolation and recovery of radium in the mill;
- disposal of tailings more environmentally acceptable than at present;
- increased water recycle;

- decreased energy requirements in the total circuit;
- decreased capital and operating costs; and
- possibility of producing a higher purity uranium product at the mill site.

Such a flowsheet, with minor modifications, might be considered in many hydrometallurgical applications.

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