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# METHODS FOR REMOVAL OF ACTINIDES FROM HIGH-LEVEL WASTES

RADIOACTIVE WASTE

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*The extent to which actinides must be removed from spent reactor fuel in order to reduce the hazard index of high-level waste after a 1000-yr storage period to 5% of the hazard index of pitchblende has been determined, and a partial evaluation of the feasibility of accomplishing these removals has been made. The feasibility study was directed primarily at high-level waste from commercial reprocessing of light-water-reactor fuel. Conceptual processes were derived from published information. Several specific problems must be solved before satisfactory process flow sheets can be developed for obtaining the necessary degree of removal of uranium, neptunium, plutonium, americium, and curium.*

## INTRODUCTION

Fractionation of high-level wastes into long- and short-lived fractions is a necessary part of some of the waste management alternatives presently under study by the U.S. Atomic Energy Commission (USAEC) for rendering the wastes more manageable in terms of long-term hazards and storage requirements.<sup>1</sup> Removal of actinides from the high-level waste is an important step in fractionation since these elements are the greatest contributors to the long-term hazard. The actinides removed from the wastes could be recycled to nuclear reactors for burnup to shorter lived fission products<sup>2</sup> or disposed of in a special manner that takes into consideration their relatively low heat generation rate but very long half-lives.

On the basis of the long-term hazard, there is potential merit in the use of separations processes that will greatly reduce the losses of fissile and fertile values to the high-level waste during conventional reprocessing of reactor fuels and that will also separate the high-level waste into fission-product and actinide fractions. Such separations could potentially alleviate the long-term storage of the remaining fission products since, following solidification and only 500 to 1000 years of storage, they would have a specific radiological toxicity comparable to that of deposits of naturally occurring minerals and ores. A storage period of 500 to 1000 years is a time span for which the effects of geologic, climatic, and other natural phenomena can reliably be extrapolated.

In conventional fuel reprocessing, about 99.5% of the fissile and fertile values are recovered from either uranium-plutonium or uranium-thorium fuels. All other actinides are usually discharged to the high-level fission-product wastes along with the 0.5% of unrecovered fissile and fertile values. Waste from each type of fuel will require removal of uranium, plutonium, neptunium, americium, and curium; waste from thorium-uranium fuels will also require removal of thorium and protactinium.

In the present study, the required degree of separation of actinides was determined so that the resultant fission-product fraction had a toxicity index comparable to uranium ores. A conceptual processing sequence was developed for removing specific actinides from high-level wastes. Candidate process flow sheets for recovering the individual actinides were studied in detail, and specific problem areas that will require research and development to adapt the flow sheets to the particular problem of waste fractionation were identified. In the selection of candidate processes, preference was given to those which had been

utilized on the largest scale of operations and in which recycle of process liquids and reagents could be most readily accomplished. It was considered of paramount importance that waste streams generated by further treatment for recovery of actinides be compatible with recycle to previous stages in the processing or with concentration and combination with the high-level waste in circumstances where the volume of high-level waste is not greatly increased. A limited number of laboratory experiments were used as an aid in selecting candidate processes for the removal of actinides. The present study provides a part of the necessary technological basis for evaluating waste management alternatives currently under study by the USAEC.

Studies thus far have been directed at the recovery of actinides from high-level wastes generated by the reprocessing of light-water-reactor (LWR) fuel because that is the only type of fuel for which chemical reprocessing experience exists on a large scale. It is expected that studies will be extended to include recovery from liquid-metal fast-breeder (LMFBR) fuel in the near future. Studies of fractionation of high-level wastes from reprocessing high-temperature gas-cooled reactor (HTGR) fuels are not expected to begin before the technical feasibility of the waste fractionation concept has been satisfactorily demonstrated for high-level wastes from the reprocessing of LWR and LMFBR fuels.

A previous study<sup>3</sup> of waste fractionation included an estimation of the necessary degree of actinide removal from high-level waste and a general survey of possible methods for removing the actinides from fission products. Some of the main conclusions reached in the study were as follows:

1. The separation factors for the required removal of actinides from high-level waste can range from as low as 10 to as much as  $10^6$  to  $10^8$ , depending on the assumptions and specific case conditions. The more likely case is somewhere in the middle of the range and is believed to be between 100 and 10 000.

2. Adequate basic technology exists to develop separations processes that give a separation factor of  $10^4$ , but these processes have not been applied to the particular problem of waste fractionation.

3. Solvent extraction and ion exchange processes have the best capability for achieving the required separation factors.

4. Solids present in all high-level radioactive waste will complicate separations process development.

It is emphasized here that the present study deals only with the high-level wastes generated by the reprocessing of commercial spent fuels. Although the major losses of actinides in reprocessing are to the high-level wastes, there are other losses primarily associated with cladding hulls and with miscellaneous low- and intermediate-level waste solids and solutions. Management concepts for these wastes are the subject of studies in other USAEC waste management programs. Nevertheless, it is expected that if the feasibility of removing actinides from high-level wastes can be established, most of the technology will be applicable to reducing the actinide losses to the other wastes.

## DEGREE OF REMOVAL REQUIRED

### Calculational Methods to Determine Actinide Removal Required from Spent Fuels

The details of the calculational method and the basis on which the calculations of the required actinide removals were made were reported previously.<sup>2</sup> Therefore, only the basis of the calculations needs be presented here to provide an understanding of the percentage removal requirements of the actinides from high-level wastes.

The effect of the degree of removal of actinides on the radiological toxicity was estimated by calculating a hazard index value<sup>a</sup> as a function of time for the mixture of fission products and actinides that remained in the high-level wastes after assumed percentages of initial actinide removal. The following are the bases on which these calculations were made:

1. The total quantities of actinides not recovered in the processing of 1 MT of spent LWR, LMFBR, and HTGR fuels were present in 2, 3, and 6 ft<sup>3</sup>, respectively, of solidified high-level fission-product waste.

2. The total quantities of the rare gases, total tritium, and 99.9% of iodine were separated as separate wastes in the fuel reprocessing and thus were not present in the high-level wastes. The

<sup>a</sup>The hazard index is defined as the volume of water required to dilute a unit volume of a radionuclide mixture to the levels defined in the Code of Federal Regulations (10 CFR, Part 20) as the maximum permissible concentration for unrestricted use of water. The hazard index provides only an approximate comparison of radiological risks, since it allows neither for accumulation or reconcentration of a nuclide in environmental media nor for the total impact of a number of nuclides. For limited comparisons, it is an acceptable alternative to dose calculation if used with caution.

total quantities of all other fission products were assumed to be present in the high-level waste. None of the activation products produced in the fuel cladding was assumed to report to the high-level waste.

3. Fuels from a 1000-MW(e) LWR of the pressurized-water-reactor (PWR) type, a 1100-MW(e) LMFBR of the General Electric Follow-On (GEFO) design, and a 1160-MW(e) reference design HTGR were used as the representative classes of fuels in a commercial industry. Two classes of fuel were considered for the LWR: PWR-U in which there is no recycle of the plutonium, and PWR-Pu in which the plutonium is recycled.

4. The average exposures of the LWR, LMFBR, and HTGR fuels were assumed to be 33 000, 41 200 and 94 300 MWd/MT, respectively. The fission-product and actinide contents of the spent fuels were calculated by using the ORIGEN code.

#### Degree of Actinide Removal Required for High-Level Wastes to Be Comparable in Radiological Toxicity to Uranium Ores

The possible merits of the removal of actinides from high-level wastes are illustrated in Fig. 1. The hazard index of high-level waste generated by the reprocessing of spent fuel from a typical LWR is compared with that of the same waste after further postulated removal of actinides by secondary treatment.<sup>4</sup> Conventional reprocessing removes 99.5% of the uranium and plutonium, whereas 99.9% of the uranium, 99.99% of the plutonium, 95% of the neptunium, and 99.9% of the americium and curium are removed in combined conventional and secondary reprocessing. For reference, the hazard indexes associated with the mineral pitchblende (~60% uranium) and with uranium ore containing 0.2% uranium (typical of large deposits that occur on the Colorado Plateau) are also shown. The hazard index of waste generated by conventional reprocessing decreases rapidly over the first thousand years (due mainly to the decay of <sup>90</sup>Sr and <sup>137</sup>Cs) but remains as great or greater than that of pitchblende for more than a million years. The hazard index for waste produced by the secondary treatment, however, falls within the range of naturally occurring radioactive materials after only several hundred years, a time span for which the effects of geologic, climatic, and other natural phenomena can reliably be extrapolated. One method for disposing of the separated actinides and <sup>129</sup>I would be to recycle them with fresh fuel to power reactors. Our calculations (Fig. 1) show that even after sustained recycle of these materials, the hazard index of the waste would still lie within the range of naturally

occurring radioactive minerals, although it is slightly higher than without recycle.

Table I gives the percentage removal of each actinide element for different reactor fuels that would be required to reduce the hazard index of wastes after a thousand years of storage to less than 5% of that of pitchblende. Further removal of the actinides would not be warranted unless one also removed the long-lived fission products: <sup>99</sup>Tc, <sup>93</sup>Zr, <sup>134</sup>Cs, and additional <sup>129</sup>I. Wastes from all of the fuel types require removal of uranium, plutonium, neptunium, americium, and curium; in addition, HTGR wastes require removal of thorium and protactinium. Separations processes

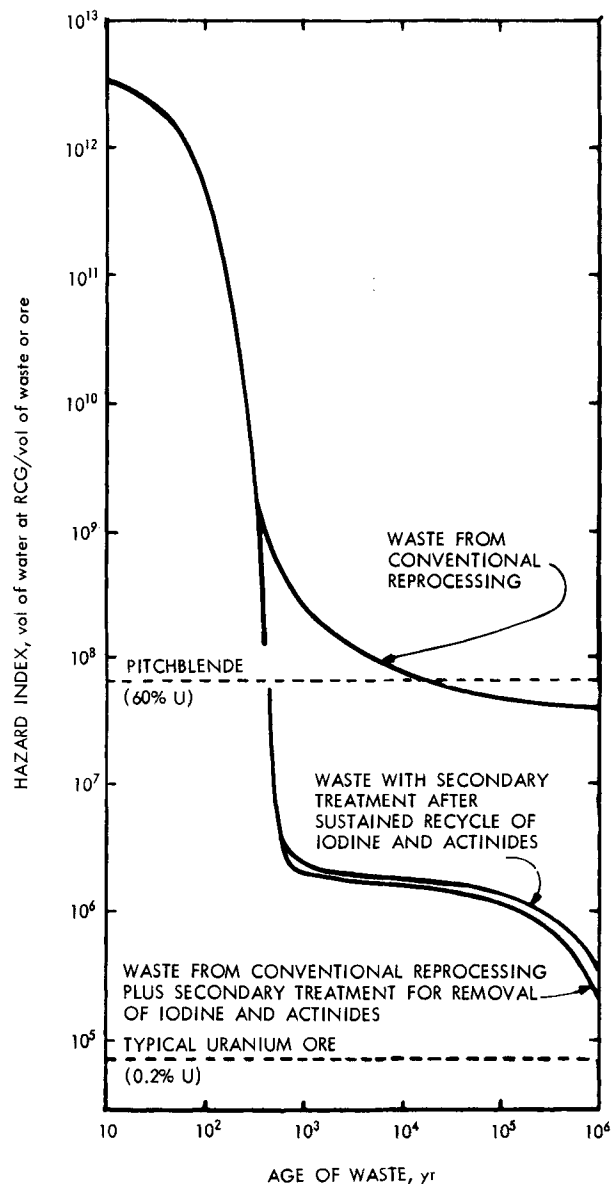


Fig. 1. Effect of age and method of treatment on the hazard index of high-level wastes from LWRs.

TABLE I  
Percentage Removals Required to Reduce the Hazard Index of Aged Wastes to <5% of That of Pitchblende\*

Actinide	PWR-U	PWR-Pu	LMFBR	HTGR
Th				95.0
Pa				95.0
U	99.9	99.9	99.9	99.99
Np	95.0	95.0	75.0	95.0
Pu	99.95	99.99	99.99	99.9
Am and Cm	99.9	99.9	99.9	95.0

\*Mineral containing 60% uranium.

currently used to recover actinides from gross fission products normally have given uranium, plutonium, and thorium recoveries of ~99.5%, and neptunium, americium, and curium recoveries in the 90 to 99% range.

It is therefore apparent that a comprehensive development program is needed to reduce the concept of waste fractionation to practice. The program must be designed to accomplish the following:

1. solve problems that are obvious from previous experience
2. increase the removal of actinides to the desired levels
3. determine the most desirable method for integrating the needed process cycles into an overall system
4. choose processes and reagents that minimize high-level waste treatment problems
5. determine the compositions of all waste streams that will be generated and ways to recycle these streams.

**PROCESSES FOR REMOVAL OF THE ACTINIDES**

Results of studies made thus far indicate that the desired removal of actinides from spent LWR fuels can best be obtained by improved recovery of actinides in conventional reprocessing plus secondary reprocessing of the high-level wastes. In conventional, commercial reprocessing (Fig. 2) by the Purex process, about 99.5% of fissile and fertile values are recovered from the spent fuel. The high-level aqueous fission-product waste contains the major fraction of actinides discharged to wastes in conventional processing. The schematic Purex flow sheet shown in Fig. 2 typifies the Nuclear Fuel Services plant, which has processed

several hundred tons of spent fuel. In the Midwest Fuel Recovery plant, not yet operating, neptunium will also be recovered by the Purex process. Concentrations of the key constituents in the high-level waste stream and the required recovery factors for the actinides are shown in Table II. To

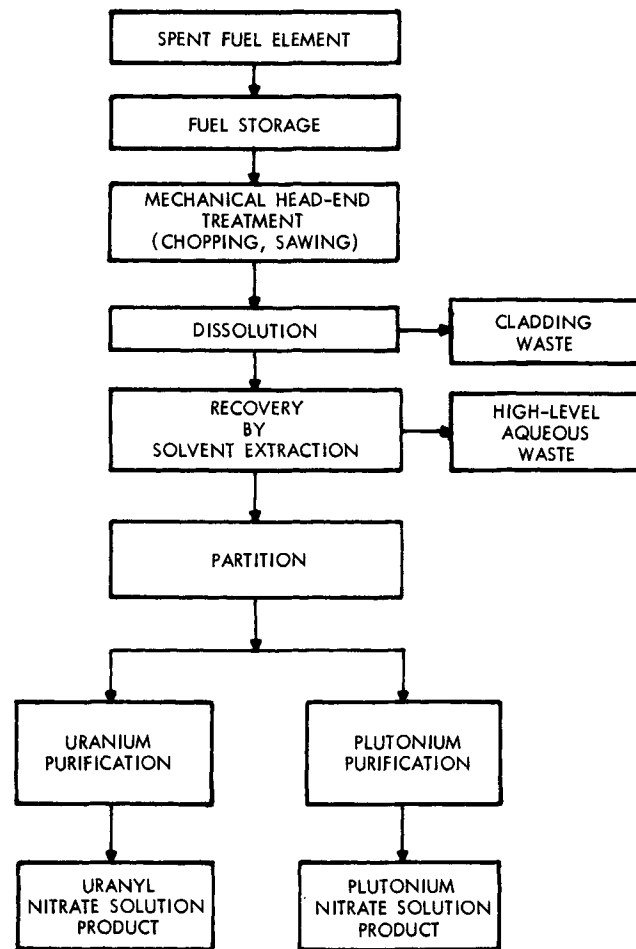


Fig. 2. Outline of conventional processing.

TABLE II

Composition of High-Level Waste from Reprocessing LWR Fuel and Actinide Recovery Factors Required\*

Constituent	Concentration (g/liter)	Recovery Factor Required
U	0.9	≥5
Np	0.09	≥20
Pu	0.009	≥50
Am	0.03	≥1000
Cm	0.007	≥1000
Total fission products	5.60	
Rare earths	2.0	

\*Basis: 33 000-MWd exposure; 5175 liters of waste per ton of fuel; 0.5% loss of uranium and plutonium to waste.

achieve the required removal of actinides from spent LWR fuels in which plutonium is recycled, minimum additional recovery factors from the high-level wastes of 5, 20, 50, 1000, and 1000 are needed for uranium, plutonium, neptunium, americium, and curium, respectively.

In the present work, only the removal of actinides from the high-level aqueous waste is considered. It has been assumed that aqueous waste streams from the partition and purification cycles can be recycled. This recycle appears to be feasible if suitable reductants are used in the partitioning and purification cycles. Eventually, waste from the purification of the solvents will have to be considered. The sodium carbonate scrubber wastes produced in conventional processing by the solvent purification process employed contain perhaps as much as a few hundredths of a percent of the plutonium. Methods for recovering this plutonium will have to be developed, or a more satisfactory solvent purification will have to be used.

Although it may be feasible to obtain the required recoveries by modifying existing plant processes and adding extra recovery processes, some difficult chemical problems will have to be solved before this can be accomplished (Table III). In addition to the undissolved residues in fuel dissolution, these problems are largely associated with chemical changes that occur in solutions of actinides and fission products during conventional processing and additional processing and with the recycle of all the waste streams generated. In the past, more complete recovery of plutonium has been inhibited by inextractable forms of plutonium. These inextractable forms consist of the residues from fuel dissolution and of forms generated during processing, not yet identified, which may include colloids, precipitates, sorbed species, and complex ions. Certain fission products, such as tin, zirconium, and molybdenum, are

easily hydrolyzed and precipitated and may present many difficulties in processing with regard to actinide losses and plant operations. Processes currently employed for separating americium and curium from fission products require process solutions of low acidity,<sup>5</sup> and hydrolysis and precipitation of actinides and fission products must be prevented in the course of processing if required recoveries are to be achieved. In addition to low acidity, the available processes for separating americium and curium require feed solutions that contain high concentrations of salting agents such as aluminum nitrate; hence, recycle of the salts is essential if the volume of the high-level waste is not to be greatly increased.

Methods for controlling neptunium valence will need to be developed so that the kinetic processes can be adjusted to achieve the desired degree of recovery of neptunium at the processing rate required. Alternative reductants for ferrous sulfamate, which is presently used for partitioning the plutonium from uranium and neptunium, will have to be developed so that the recycle of partition-cycle and purification-cycle wastes can be accomplished. This recycle will also minimize treatment problems in converting the high-level wastes to solids for storage. All waste streams generated in reprocessing must be effectively recycled so that volumes of all the wastes are not greatly increased. Recycle concepts will require evaluation with regard to the potential for buildup of impurities that are detrimental to certain processing steps.

The conceptual processing sequence for removing the actinides has not been finalized since studies are still in progress. The current concept is illustrated in Fig. 3. According to this flow sheet, insoluble residues from fuel dissolution would be separated and processed in a secondary dissolver in the primary Purex plant. If technically feasible, essentially complete removal of the reactor-bred plutonium, uranium, and neptunium will be accomplished by using extra stages of the Purex process in which exhaustive extraction is carried out. It is expected that 95 to 99% of the neptunium will be recovered in the primary extraction and an even more complete recovery in the exhaustive extraction. The small quantity of actinides removed by the exhaustive extraction will be recycled to the primary Purex plant to avoid the necessity for extra purification cycles for fission-product decontamination.

Next, the wastes are stored for a period of 3 to 4 years to reduce the radiation damage to process reagents, solvents, or ion exchange resins that are to be used in subsequent operations. The disadvantages of the storage requirement are that large volumes of wastes in liquid form are ac-

TABLE III  
Major Problems to Be Solved

- |  |
|--|
| <ol style="list-style-type: none"> <li>1. Undissolved residues of fuel</li> <li>2. Inextractable forms of plutonium</li> <li>3. Development of an americium and curium recovery process</li> <li>4. Actinide losses to solids generated in the processing</li> <li>5. Control of valence of neptunium during extraction</li> <li>6. Development of alternative reductants for uranium, plutonium, and neptunium partitioning</li> <li>7. Recycle of all waste streams generated by the processing</li> </ol> |
|--|

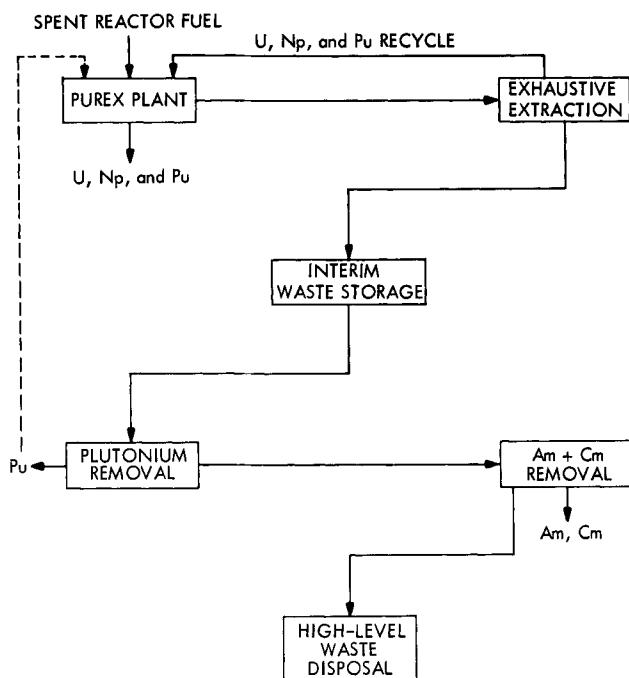


Fig. 3. Conceptual processing sequence for the removal of actinides.

accumulated and that plutonium grows back into the wastes via decay of curium and will again require removal.

In the following step, it is visualized that the daughter plutonium as well as any plutonium not extracted in the exhaustive extraction step would be removed. This recovered plutonium may or may not be recycled to the primary plant, depending on the effects of its isotopic composition. The nature of the processing required for this step has not yet been determined. It would probably consist of removing any actinide-bearing solids generated in the previous processing and treating them for recovery, plus chemically treating any soluble forms of inextractable plutonium such as colloids or complex ions. Provided that the solids can be solubilized in nitric acid or leached free of actinides by nitric acid, the actinide values would be recovered from that solution. However, if this is not feasible, the solids might be processed in a special manner by using molten salts or nitric-hydrofluoric acid mixtures. Any solubilized but inextractable forms of plutonium such as plutonium polymer or complex ions, which may exist, will present a difficult problem and will require development work so that the inextractable forms may be identified and the necessary methods for rendering them extractable can be developed.

The americium and curium are removed following final removal of the plutonium. Again, the specific recovery process has not been desig-

nated. Existing processes for removing americium and curium from fission products are deficient in one or more ways. Although several of the processes have the potential for the final removal step in which americium and curium are separated from the rare-earth fission products, methods for preparing a satisfactory feed solution to these processes will have to be developed. Conventional processes for separating americium, curium, and rare earths from gross fission products utilize high concentrations of metal salts, and methods for eliminating these salts from the final fission-product solid waste by appropriate recycle will have to be developed.

The two processes currently being evaluated for the final removal of americium and curium from the rare earths are cation exchange chromatography (CEC) (Refs. 6 and 7) and the Talspeak process<sup>8,9</sup> in which removal is effected by extraction from carboxylic acid solutions into di(2-ethyl) phosphoric acid (HDEHP). Processes being evaluated for the preparation of a suitable actinide-lanthanide feed to these processes include tributyl phosphate (TBP) extraction of lanthanides and actinides from solutions salted with aluminum nitrate<sup>5,10,11</sup> and extraction of interfering fission products by HDEHP from 6-M nitric acid solution. The extraction process utilizing TBP and aluminum nitrate salting also offers the potential for eliminating the light rare earths in the feed preparation step. Both the feed preparation step and the final removal step of americium and curium from the rare earths present some difficult problems in regard to recycle. In addition, the low acidities required to effect the final separation and to achieve separation of rare earths and actinides in the feed preparation by TBP extraction present difficult control problems relative to the prevention of product losses by hydrolysis and precipitation.

Conceptual flow sheets for the final removal step from the rare earths by CEC and the Talspeak process point out some of the problems to be solved regarding recycle and process control. In the CEC process (Fig. 4), the actinides and lanthanides are sorbed on a loading column. Chromatographic separation is then effected by band displacement through a series of ion exchange columns with an eluent of low pH value (~3) containing a complexing agent, diethylene triaminepentaacetate (DPTA). Zinc is used as the barrier ion so that the developing bands remain "sharp" for the individual elements over the many band displacement volumes required for separation and purification. End cutting of products is simplified by effecting band displacement through a series of columns of successively smaller diameter. This resin configuration min-

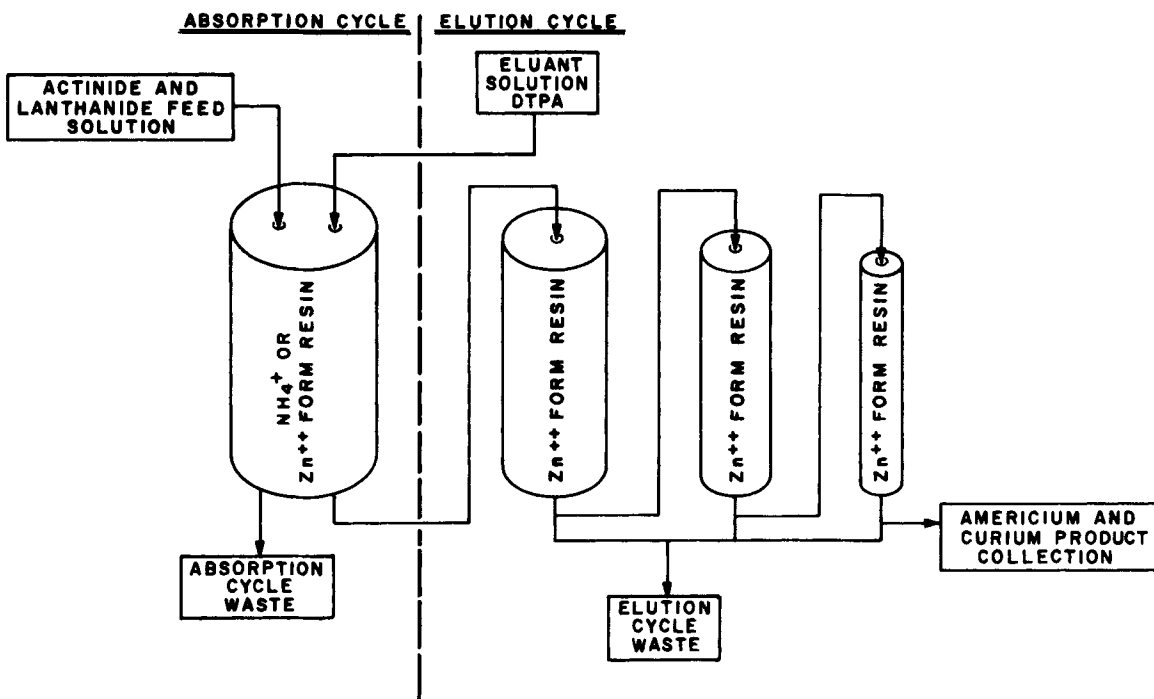


Fig. 4. Schematic flow sheet of the CEC process for recovery of americium and curium.

imizes the losses due to end cutting and, in turn, the quantity of material that must be reworked and recycled. Because of the large quantity of zinc required to provide the barrier ion, zinc will need to be recycled. In addition, significant amounts of contaminated waste resin will be produced because of mechanical, chemical, and radiation damage to the resin particles. Recycle of the organic complexing agent, DTPA, would be desirable; however, an alternative is to convert it to a noncontaminated gaseous chemical waste. Solutions to these recycle problems will require further study of both a conceptual and an experimental nature.

The CEC process has been developed<sup>7</sup> at the Savannah River Laboratory for a capacity of about 20 moles of rare earths and actinides, although not at full activity levels. This is about one-fourth the capacity necessary for the americium, curium, and rare earths in 1 MT of LWR fuel.

In the Talspeak process, a group separation of lanthanides and trivalent actinides is made; hence, it has the potential for the final removal step for americium and curium (Fig. 5). The group separation is effected by extraction from carboxylic acid solution into HDEHP. The aqueous-phase acidity is maintained at a pH of 2.5 to 3.0. The americium and curium and rare earths are co-extracted by HDEHP from 1-M lactic acid. The DTPA is used to partition the rare earths and trivalent actinides. The actinides are partitioned

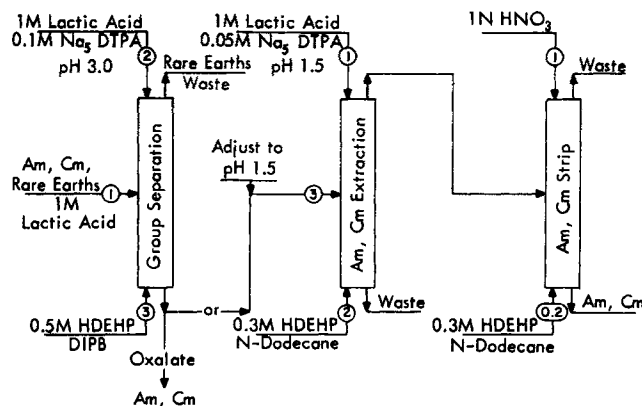


Fig. 5. Flow sheet for recovery of americium and curium by the original Talspeak process.

to the aqueous phase, where they may be further purified and concentrated by another cycle of solvent extraction or by oxalate precipitation. The Talspeak process utilizes a large quantity of process chemicals that ultimately appear in waste streams. These chemicals will need to be partially recycled, or the waste streams will have to be treated in a satisfactory manner for ultimate solidification with all of the high-level wastes. Other than sodium, the process chemicals are organic materials and thus have the potential for being converted to noncontaminated gaseous chemical wastes.



Since the candidate methods for the final removal of actinides from lanthanides will require low acidities at certain key points in the processing, both conceptual and experimental studies are being conducted to prepare low-acidity feed. Low acidity cannot be achieved without effecting precipitation of certain fission-product elements. For example, we observe that destruction of nitric acid by reduction with formic acid results in the precipitation of the noble metals, zirconium, and molybdenum. Since the zirconium and molybdenum solids are quite voluminous, separating and washing the solids free of trivalent actinides would be cumbersome and difficult even if technically feasible. Therefore, we are searching for processes that can remove all troublesome fission products. Methods currently under study include (a) HDEHP extraction of zirconium and molybdenum from 6-N HNO<sub>3</sub> solution, and (b) TBP extraction of actinide-lanthanide fraction from solutions salted with Al(NO<sub>3</sub>)<sub>3</sub>. The HDEHP process has been successively tested for zirconium and molybdenum removal in the laboratory. However, we have not yet developed a method for recovering the HDEHP solvent. The zirconium is tightly bound with the extractant and cannot be stripped from it by conventional methods such as scrubbing with sodium carbonate solution.

The process for extracting trivalent actinides and lanthanides by TBP from fission-product solutions salted with aluminum nitrate has been utilized in transplutonium-element programs.<sup>5,10</sup> The difficulties with this process which must be overcome are increasing the recoveries to the desired level and recycling the salting agent. The process is normally operated as a batch extraction in which the acidity is adjusted between successive extractions. Since the process requires low-acidity solutions (0.10 to 0.35 M), precipitation is encountered when too much acid is extracted by the TBP, and process control is cumbersome. Americium and curium recoveries of 95 to 99% are usually achieved by two batch extraction cycles. Experimental studies of this process have not been initiated. Crystallization of the aluminum nitrate from concentrated nitric acid solution may be an effective way of recycling the salting agent.

## CONCLUSIONS

Studies thus far indicate that it will be feasible to reduce the long-term hazards of high-level wastes generated by the reprocessing of LWR fuels by means of a more complete removal of actinides in conventional fuel processing plus secondary processing of the wastes. However,

many challenging problems must be solved before satisfactory processes based on current technology can be developed and, in turn, demonstrated on a pilot-plant scale at the expected levels of radioactivity. It appears that the necessary removal of uranium and neptunium can probably be accomplished by modifications and additions to the current Purex process. Several specific chemical problems which have been identified must be solved before satisfactory chemical process flow sheets can be developed for the degree of removal that will be necessary for plutonium, americium, and curium.

Satisfactory recycles of process chemicals appear to be feasible in conceptual processes in which improved recovery of uranium, plutonium, and neptunium is achieved by modifications and additions to the Purex process. It is also believed that satisfactory solutions to the recycle problems associated mainly with the secondary processing for americium and curium can be developed, but a substantial research and development effort is needed to obtain the necessary methods.

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