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### Title:

Liquid Membrane System for the Removal and Concentraion of Transuranic Elements

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#### **Abstract**

The goal of this program is to develop an efficient, reliable, and radiation-resistant modified liquid membrane system (*MLMS*) for the selective removal and concentration of transuranic elements (TRUs) and strontium-90 (90Sr) from dissolved Hanford sludge wastes. The efforts are divided into three categories: (1) demonstration and optimization of the *MLMS* for the TRUEX and SREX processes using simulant waste solution; (2) development of a radiation-resistant microporous divider and membrane module for testing with actual waste solutions; and (3) demonstration of the *MLMS* for the TRUEX and SREX processes using actual Hanford waste. Successful completion of these development efforts will yield a compact, versatile, and reliable *MLMS* for implementation with the TRUEX and SREX processes. The *MLMS* is simple, stable, more efficient, and easier to control and operate than conventional solvent-extraction processes, such as those employing centrifugal contactors. In addition, the *MLMS* process offers operational cost savings over the conventional technology, by exhibiting at least a 10% reduction in the consumption of extractant chemicals.

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### **Executive Summary**

The goal of this program is to develop an efficient, reliable, and radiation-resistant modified liquid membrane system (*MLMS*) for the selective removal and concentration of transuranic elements (TRUs) and strontium-90 (90Sr) from dissolved Hanford sludge wastes. The efforts are divided into three categories: (1) demonstration and optimization of the *MLMS* for the TRUEX and SREX processes using simulant waste solution; (2) development of a radiation-resistant microporous divider and membrane module for testing with actual waste solutions; and (3) demonstration of the *MLMS* for the TRUEX and SREX processes using actual Hanford waste. Successful completion of these development efforts will yield a compact, versatile, and reliable *MLMS* for implementation with the TRUEX and SREX processes. The *MLMS* is simple, stable, more efficient, and easier to control/operate than conventional solvent-extraction processes, such as those employing centrifugal contactors. In addition, the *MLMS* process offers operational cost savings over the baseline technology, by exhibiting at least a 10% reduction in the consumption of extractant chemicals.

The third effort described above comprises the Phase II research, and is to be conducted primarily at Battelle Pacific Northwest Laboratories (PNL), a facility equipped for handling actual radioactive waste. Work toward accomplishing the first two efforts is currently in the early stages of development at LSR.

Pursuant to effort category (1), a novel *MLMS* configuration specifically designed for this project has been developed. This new design will allow for long residence times at minimal pressure drop. Testing with the simulant TRUs and nonradioactive ("cold") strontium have only just begun, so few results can yet be reported. The objective of this aspect of the research is to surpass the performance of the baseline TRUEX/SREX process, which relies upon the use of a centrifugal contactor device to perform the liquid-liquid extraction chemistry.

Toward the goal of developing/identifying suitably radiation-resistant membranes (effort (2) above), thin film samples of a number of candidate polymers are being subjected to a range of irradiation dosages, and evaluated for their elongation at break, a simple-to-measure mechanical property which readily reflects the degradative chemistry resulting from exposure to radiation in excess of the material's tolerance threshold. Dogbone-shaped film samples of a range of high-performance polymers are being tested. Multiple dogbone samples of each of these materials are being irradiated for cumulative total dosages (CTD) ranging from 0 to 100 MRad (0 to 10<sup>6</sup> Gy) of e-beam irradiation. These dosages are meant to approximate the CTD which would be experienced by the system over the course of up to 300 years of continuous use with the current TRUEX/SREX processes. Samples exposed to these dosages, along with control films not exposed to any irradiation, are being tested on a tensile-type testing device designed and assembled in-house, for their percent elongation at break as a function of their level of irradiation. Preliminary results of these tests show that each of these polymers exhibits some resistance to irradiation, as gauged by their small decreases in elongation at break.

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### Introduction

The treatment and disposal of radioactive waste generated in past plutonium operations represents an immense technical and economic challenge. Large amounts of hazardous, highly radioactive wastes are accumulated at many contaminated sites throughout the U.S., with the majority located at the following four sites: The Hanford site in Richland, Washington; the Idaho National Laboratory in Idaho Falls; the Savannah River site in South Carolina; and the Oak Ridge National Laboratory in Oak Ridge, Tennessee. These radioactive wastes include high-level wastes (HLW), low-level wastes (LLW), transuranic (TRU) wastes, and highly radioactive mixed wastes. Most of the waste volume is made up of nonradioactive and low level waste components, but without treatment, this waste must be disposed of in a geologic repository, which is very costly. Removal and concentration of the highly radioactive components from their dilute solutions can minimize the quantity of high level waste needing disposal and, thereby, drastically reduce the costs of vitrification and geologic disposal.

In the case of Hanford tank sludge wastes, a baseline approach has been defined in which the sludges are leached with a highly caustic solution to dissolve certain nonradioactive components of the sludge. The leached sludges are then washed with a dilute hydroxide solution to remove most of the added caustic and the dissolved sludge components. Following removal of cesium, the leach and wash solutions can be handled as low-level waste (LLW). The leached sludges will contain transuranic elements, strontium, and other radionuclides; thus, they must be handled as HLW and immobilized in a borosilicate glass waste form.

Using even the most favorable assumptions concerning this baseline scheme, it is estimated that the processing of the Hanford tank sludges will generate some 20,000,000 kg of HLW glass. The cost to process and dispose of this sludge has been conservatively estimated to be \$3 billion. Furthermore, since repository space is limited, there may not be enough volume available in a deep geologic repository. Clearly, there is an incentive to reduce the volume of HLW glass beyond that obtained by implementing this baseline process.

This project utilizes the Modified Liquid Membrane System (*MLMS*) developed by LSR to separate the radioactive and non-radioactive components of Hanford tank sludge to drastically reduce the volume of HLW. The key radionuclides requiring separation are the transuranic elements (TRUs) and strontium. Two proven chemical processes will be utilized to achieve these separations: the transuranic extraction (TRUEX) process and the strontium extraction (SREX) process. While both the TRUEX and SREX processes were originally intended for traditional solvent extraction equipment, this project will deploy a unique supported liquid membrane.

### **Objectives**

The overall goal of this program is to develop an efficient, reliable and radiation-resistant *MLMS* for the selective removal and concentration of transuranic elements (TRUs) and strontium-90 (90Sr) from dissolved Hanford sludge wastes. The efforts are divided into three categories: (1) demonstration and optimization of the *MLMS* for the TRUEX and SREX processes using simulant waste solution; (2) development of a radiation-resistant membrane and membrane module for testing with actual waste solutions; and (3) demonstration of the *MLMS* for the TRUEX and SREX processes using actual Hanford waste.

The program shall demonstrate that the *MLMS* is simpler, more efficient, and easier to control and operate than conventional solvent extraction processes, such as those based on centrifugal contactors. The specific objectives for the proposed research are as follows:

- to demonstrate that the *MLMS* can remove greater than 95% of the simulant TRUs using CMPO as extractant
- to demonstrate that the *MLMS* can remove greater than 95% of the simulant strontium using DtBuCH18C6 as extractant
- to develop and evaluate a radiation-resistant support membrane which can withstand a radiation does of up to 25 Wh/kg (10 MRad, equivalent to 30 years of continuous TRUEX processing<sup>[1]</sup>) and which is chemically compatible with feed, strip, and membrane liquid solutions
- to demonstrate that the *MLMS* can effectively remove TRUs (using CMPO) and <sup>90</sup>Sr (using DtBuCH18C6) from actual Hanford waste with > 95% removal efficiency and at least a 10% reduction in extractant consumption.

Experimental testing with simulated waste solutions will be conducted in the LSR laboratory. The recipes for the simulant feed solution have been supplied by Pacific Northwest Laboratory (PNL). In parallel with these efforts, a radiation-resistant support membrane and modified liquid membrane module will be developed and evaluated at LSR. Following a successful demonstration with the simulant feed solutions and the development of the support membrane and membrane module, a bench-scale *MLMS* will be constructed for testing at PNL using actual Hanford waste.

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### Approach

The two main tasks in Phase I will be performed in parallel. The first task involves the demonstration and optimization of the *MLMS*, for which an experimental test loop will be designed and constructed. As shown schematically in Figure 1, this test loop is based upon a plate-and-frame module and will be used for both TRUEX and SREX evaluations. Experimental testing will focus on the measurement of Eu and Sr permeabilities and their removal capacity through the *MLMS* under various operation conditions.

All experiments in this task will be conducted with a simulant feed solution formulated according to recipes supplied by Pacific Northwest Laboratory. In this simulant feed solution, "cold" (non-radioactive) europium (Eu) is used to simulate americium (Am). Because Am(III) is the limiting species in the efficiency of the TRUEX process,<sup>[2]</sup> development efforts will focus on the removal of its simulant, Eu(III). "Cold" Sr will be used to simulate <sup>90</sup>Sr.

The membrane liquid (extractant solution) which will be used for the TRUEX process is 0.2 M CMPO (see below) plus 1.4 M TBP in dodecane. A solution of 0.2 M DtBuCH18C6 in 1-octanol will be used as the membrane liquid for the SREX process. An aqueous mixture of sodium carbonate and the sodium salt of 1-hydroxyethane-1,1-diphosphonic acid (NaHEDPA) will be used as the TRU stripping solution. A solution of 2 mM NaHEDPA with 0.25 M Na<sub>2</sub>CO<sub>3</sub> was shown to strip TRUs from the TRUEX solvent phase as effectively as the 0.2 M

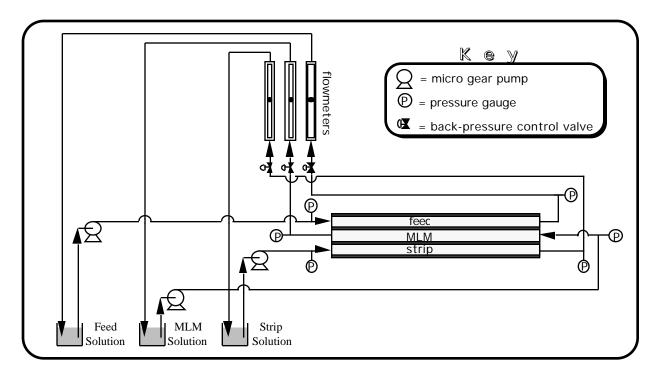


Figure 1. Schematic of experimental test loop.

HEDPA solution originally specified<sup>[19]</sup> in Hanford TRUEX process flowsheets, while simultaneously reducing by 100-fold the amount of phosphorus used. This latter feature shall significantly mitigate the concern that high phosphorus in the resulting TRU stream could preclude its effective vitrification. Dilute nitric acid solution will be used as the stripping solution for the SREX process. The possibility of combined TRU/Sr extraction testing will be evaluated.

Parametric testing will be performed to study the effect of particular operating conditions on *MLMS* performance and membrane life. Long term stability tests on the *MLMS* will also be conducted. Modifications to the membrane modules and test loop will be made as necessary to meet performance objectives. Operating parameters to be tested include:

- Operating temperature
- Feed, strip and membrane liquid velocities
- Flow direction arrangements between feed, strip and membrane liquids
- Type and wettability of the microporous support membrane

For each experiment, the effective extraction rate and removal efficiency of each permeant will be calculated. The results will be analyzed to determine the effect of each operating condition on the *MLMS* performance in the TRUEX and SREX processes. A kinetic model developed for earlier *MLMS* work in the LSR laboratory will be adapted to the current extractions, and will aid in the design of future systems.

### Development of Radiation-Resistant Support Membrane and Membrane Module

For the *MLMS* to be suitable for the TRUEX and SREX processes, both the support membrane and the module must be radiation-stable and compatible with the streams involved. Therefore, the selection and development of a support membrane for module fabrication is one of the most critical tasks in the proposed research.

Most commercially available polymer membranes are not suitable for the proposed application. In general, polymers undergo a complex series of free radical reactions when irradiated. These reactions typically effect either crosslinking or chain scission reactions, which can effect a net loss in the mechanical properties of the base polymer, leading to severe embrittlement and eventual catastrophic failure. One common means for ascertaining the extent of radiation-induced degradation is to compare a mechanical property--such as elongation at break-of an irradiated sample with that of an unirradiated control.

In the proposed research, certain commercially available membranes will initially be tested for their suitability for the TRUEX and SREX processes, since these materials have all been shown to exhibit some degree of radiation resistance. The evaluation will focus on the following aspects:

- (1) their chemical compatibility with feed, strip and membrane liquid solutions;
- (2) their mechanical integrity under the typical MLMS operation conditions,
- (3) their resistance to simulated radioactive environment.

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Meanwhile, concurrent attempts will focus on evaluating the possibility of preparing porous support membranes from more highly radiation-resistant polymers. Membranes prepared inhouse will then be tested for their applicability in *MLMS*-based TRUEX and SREX processes.

#### **Experimental Testing Using Actual Hanford Waste**

According to the experimental results obtained using the simulant solution, an experimental test loop will be designed and constructed for actual waste testing. This bench-scale test loop will consist of two radiation-resistant modules developed and constructed by LSR. The rest of the components in the loop will be selected or constructed by PNL according to the operation conditions specified by LSR. The bench-scale *MLMS* test loop will be designed to accommodate feed flowrates ranging from 20~100 mL/min. PNL will be responsible for the procurement of waste materials for testing, for performing the tests, and for reporting the results. PNL will also be responsible for ensuring that the testing is conducted in a safe manner and in compliance with all existing rules and regulations.

#### **Success Criteria**

The initial criterion of the project is the successful removal and concentration of TRUs and/or Sr from a simulant waste stream using a bench-scale *MLMS*. The key go/no-go decision criterion is the successful identification and development of a radiation-resistant porous support membrane for the membrane module.

# **Technology**

### The TRUEX and SREX Processes

The transuranic extraction (TRUEX) process<sup>[9,10]</sup> was developed for the treatment of selected radioactive wastes, such as those stored in underground tanks at the U.S. Department of Energy's Hanford Site. As shown schematically in Figure 2, this process involves the extraction of transuranic elements from nitric acid solutions with a solvent consisting of 0.2 M octyl(phenyl)-N,N-diisobutylcarbamoylmethylphosphineoxide (CMPO, see Fig. 3) and 1.4 M tributylphosphate (TBP) in a normal paraffin hydrocarbon (NPH) diluent. After passing through a suitable scrub step, the transuranic (TRU) elements are stripped from the extract. The solvent passes through a solvent wash step (0.25 M Na<sub>2</sub>CO<sub>3</sub>) and is then recycled to the extraction stage.

The TRUEX process using the mixed solvent extractant system has been demonstrated in an overall process based around centrifugal contactors developed by Argonne National Laboratory. Despite its many advantages, the centrifugal contactor has one major intrinsic disadvantage: incomplete phase separation which leads to solvent loss and reduced efficiency. Furthermore, the design and operation of centrifugal contactors are complex, which make system control and scale-up difficult.

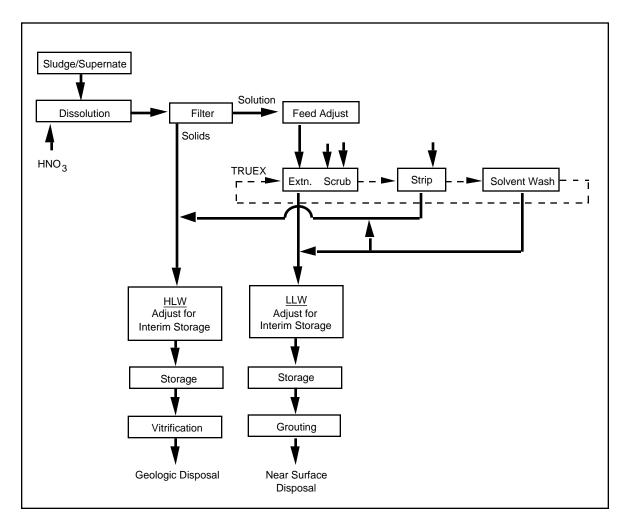


Figure 2. Generic flowsheet for the treatment of Hanford tank wastes by the TRUEX process.<sup>[9]</sup>

Figure 3. The Chemical Structures of (a) CMPO and (b) DtBCH18C6

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The strontium extraction (SREX) process [15,16] was developed at Argonne National Laboratory for the extraction and recovery of  $^{90}$ Sr (strontium-90) from Hanford high-level radioactive waste. In this process, strontium is extracted from acidic solution ( $\geq 1.0$  M HNO<sub>3</sub>) using a 0.2 M solution of di-t-butylcyclohexano-18-crown-6 (DtBuCH18C6, see Fig. 3) in 1-octanol. The extracted strontium is readily stripped from the organic phase using either water or dilute (< 0.05 M) HNO<sub>3</sub>. Using a synthetic dissolved sludge waste solution, a DtBuCH18C6-octanol system was shown to be sufficiently selective for strontium (relative to other common waste constituents) and sufficiently stable against hydrolytic and radiolytic degradation to permit its application to high-level waste processing. [15] When the SREX process was evaluated using actual Hanford sludge waste [16] (taken from tank number B-110), greater than 95% of the  $^{90}$ Sr was removed from the dissolved sludge solution. These experiments were conducted using batch contactors and a typical extraction procedure, as depicted schematically in Figure 4, using a few grams of sludge waste containing  $\sim 220~\mu$ Ci/g (8.1 MBq/g) of  $^{90}$ Sr, along with other constituents, such as Na, Bi, Cr, Fe, Si, phosphates, sulfates, etc.

### The Modified Liquid Membrane System

Development of both the TRUEX and SREX processes has been based on conventional extraction and stripping procedures. Regardless of the type of contacting devices used, however, conventional solvent extraction equipment has many disadvantages, including:

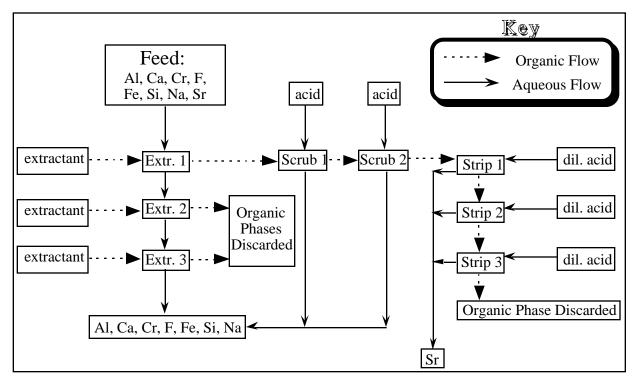


Figure 4. Schematic of the SREX process.[16]

- the need for dispersion and coalescence
- problems of emulsification and incomplete phase separation
- flooding and loading limits in continuous countercurrent devices
- the need for a density difference between the phases
- high initial, operating and maintenance costs

In addition, scale-up is always difficult. A number of newly developed non-dispersive extraction devices appear to eliminate the problems mentioned above in addition to providing very high mass transfer rates. Among these new devices are supported liquid membranes<sup>[17-22]</sup> and modified liquid membranes.<sup>[23,24]</sup>

The most basic configuration of a supported liquid membrane (SLM)<sup>[17-21]</sup> involves impregnating the pores of a microporous support membrane with the "liquid membrane" phase, which is held within the pores by capillary forces. The feed solution is exposed to one side of the SLM, where extraction takes place. The stripping solution is in simultaneous contact with the other side of the SLM, where the species of interest is stripped from the SLM. The primary advantage offered by the supported liquid membrane is that it is a nondispersive process yet offers a high mass transfer surface area for extraction. Moreover, the SLM process combines extraction and stripping into a single unit, thereby eliminating the equilibrium limitations experienced in a conventional two-step extraction and stripping process, and greatly simplifying the process and its operation. However, the SLM configuration is unstable due to the gradual dissolution of the limited membrane phase into both aqueous (*i.e.*, feed and stripping) streams.

An innovative variation on the supported liquid membrane configuration is the so-called hollow-fiber contained liquid membrane,<sup>[25]</sup> which comprises a module with two independent sets of fibers. One set carries the feed stream, while the other carries the stripping stream; the membrane liquid is held within the interstices between the two sets of fibers. The advantage of this configuration is that the membrane liquid is replenishable and therefore more stable than in a conventional supported liquid membrane. However, due to uneven distribution of the fibers, the effective liquid membrane thickness can be high, and can significantly increase the total mass transfer resistance in the membrane module.

The novel Modified Liquid Membrane System (*MLMS*) developed by LSR Technologies circumvents the stability limitation of the SLM approach but offers all the same advantages, namely: combined nondispersive extraction and stripping, no equilibrium limitations, low solvent losses, and high mass transfer area.<sup>[23,24]</sup> A schematic of the *MLMS* configuration is given in Figure 5. The aqueous feed and stripping solutions are separated by membrane liquid which is immobilized in and flowing between two microporous membranes. The liquid membrane mass transfer surfaces (for extraction and stripping) are localized in the pores of the microporous membranes by the relative pressure control between the membrane liquid and feed/strip solutions.

The feasibility of the MLMS has been demonstrated in two different applications: (1) the selective removal and concentration of copper from wastewater streams using conventional copper extractants in a diluent (e.g., LIX84 or Acorga 5640 in kerosene) as the membrane liquid; [23] and

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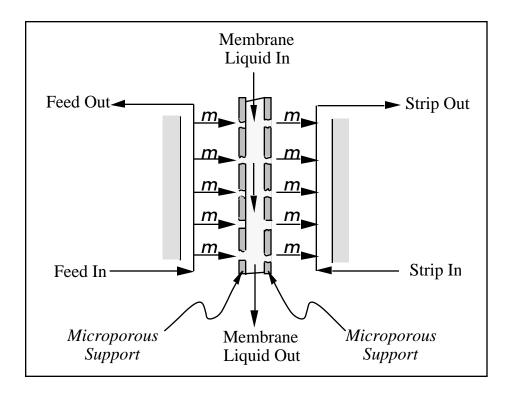


Figure 5. Schematic of MLMS Configuration (m is permeant)

(2) the selective removal and concentration of  $H_2S$  from gas mixtures containing both  $CO_2$  and  $H_2S$  using an aqueous tertiary amine solution as the membrane liquid.<sup>[24]</sup>

In the case of copper removal and concentration, the *MLMS* achieved up to 99.99% copper removal from the feed stream, lowering its concentration in one case from 439 mg/L to 0.059 mg/L. In another case, Cu was transferred against a 1000-fold concentration gradient from a 10 mg/L feed solution to a 10 g/L (10,000 mg/L) strip solution. Cu permeation rates greater than 6  $\mu$ g/cm<sup>2</sup>/min were measured in the majority of tests, and no performance instability was encountered in any of the tests. Under identical test conditions, such instabilities *were* experienced in a conventional SLM.<sup>[23]</sup>

When the *MLMS* was utilized for the selective removal and concentration of H<sub>2</sub>S from gas mixtures containing both CO<sub>2</sub> and H<sub>2</sub>S, selectivities as high as 30 and H<sub>2</sub>S permeabilities as high as 10<sup>7</sup> Barrers/cm were achieved, both of which are much higher than can be achieved in conventional membrane and non-membrane separation processes.<sup>[24]</sup> A number of prototype-scale *MLMS* module architectures have been evaluated at LSR Technologies, including a multichannel plate-and-frame module and spiral-wound module.

#### **Results and Discussion**

Because this project has only just commenced as of 12 July 1996, we are still in the preliminary stages of development and testing. Pursuant to effort category (1) -- the demonstration and optimization of the *MLMS* for the TRUEX and SREX processes -- a novel *MLMS* configuration specifically designed for this project has been developed. This new design will allow for long residence times at minimal pressure drop. The objective of this aspect of the research is to surpass the performance of the baseline TRUEX/SREX process, which relies upon the use of a centrifugal contactor device to perform the liquid-liquid extraction chemistry. Testing with the simulant TRUs and nonradioactive ("cold") strontium have only just begun, so few results can yet be reported.

Toward the goal of developing/identifying suitably radiation-resistant membranes (effort (2)), thin film samples of a number of candidate polymers are being subjected to a range of irradiation dosages, and evaluated for their elongation at break, a simple-to-measure mechanical property which readily reflects the degradative chemistry resulting from exposure to radiation in excess of the material's tolerance threshold. Dogbone-shaped film samples of a range of high-performance polymers are being tested. Multiple dogbone samples of each of these materials are being irradiated for cumulative total dosages (CTD) ranging from 0 to 100 MRad (0 to 10<sup>6</sup> Gy) of e-beam irradiation. These dosages are meant to approximate the CTD which would be experienced by the system over the course of up to 300 years of continuous use with the current TRUEX/SREX processes.<sup>[1]</sup> Samples exposed to these dosages, along with control films not exposed to any irradiation, are being tested on a tensile-type testing device designed and assembled in-house, for their percent elongation at break as a function of their level of irradiation. Preliminary results of these tests show that each of these polymers exhibits some resistance to irradiation, as gauged by their small decreases in elongation at break.

### **Benefits**

The features and advantages of the *MLMS* are summarized as follows:

- It combines extraction and stripping steps within the same processing unit, thereby simplifying the TRUEX and SREX processes; hence, process control and operation can be easily managed, resulting in reduced maintenance and operating costs.
- Solvent loss due to dissolution into the feed and strip streams is minimized, resulting in at least a 10% reduction in extractant consumption (relative to the conventional technology).
- It is a non-equilibrium mass transfer process, eliminating equilibrium limitations encountered in conventional extraction and stripping processes.
- Its configuration is completely stable and not prone to performance-limiting effects

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such as membrane phase degradation, swelling, and loss of liquid due to partitioning.

- The *MLMS* is flexible and can be adapted to a number of different separation requirements.
- High selectivity can be achieved by incorporating the appropriate extractant in the membrane phase for different processes, such as TRUEX, SREX, or combined TRU/Sr extraction processes.
- It can be designed with a high mass transfer surface area within a small volume, thereby reducing the footprint size.

These features and advantages of the *MLMS* will significantly contribute to the implementation of the TRUEX, SREX, or combined TRU/Sr extraction processes. An advanced separation processes will result which:

- reduces HLW treatment and disposal costs,
- minimizes secondary wastes,
- · achieves high removal efficiency, and
- meets the regulatory requirement.

### **Conclusions**

As this project has only just commenced as of 12 July 1996, we are still in the preliminary stages of development and testing. Consequently, no conclusions can yet be drawn.

### **Future Activities**

As this project has only just commenced as of 12 July 1996, we are only in the preliminary stages of development and testing. Consequently, most all of the work described above is still in the future.

# Acknowledgement

The valuable guidance and assistance of our METC Contracting Officer's Representative (COR), Dr. Venkat K. Venkataraman, is gratefully acknowledged. The period of performance for this contract is from 12 July 1996 through 11 December 1998, during which time part of the work will be subcontracted out to Battelle Pacific Northwest Laboratory.

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