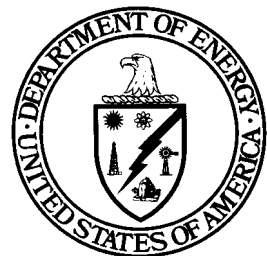




Summary Report DOE/EM-0501

Rapid Sampling Using 3M Membrane Technology

Characterization, Monitoring, and Sensor
Technology Crosscutting Program and
Subsurface Contaminants Focus Area



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Rapid Sampling Using 3M Membrane Technology

OST/TMS ID 1514

Characterization, Monitoring, and Sensor
Technology Crosscutting Program and
Subsurface Contaminants Focus Area

Demonstrated at
Savannah River Site
Aiken, South Carolina
Argonne National Laboratory
Argonne, Illinois
Paducah Gaseous Diffusion Plant
Paducah, Kentucky
West Valley Demonstration Project
West Valley, New York
General Electric Storage Facility
Morris, Illinois



Purpose of this document

Innovative Technology Summary Reports are designed to provide potential users with the information they need to quickly determine whether a technology would apply to a particular environmental management problem. They are also designed for readers who may recommend that a technology be considered by prospective users.

Each report describes a technology, system, or process that has been developed and tested with funding from DOE's Office of Science and Technology (OST). A report presents the full range of problems that a technology, system, or process will address and its advantages to the DOE cleanup in terms of system performance, cost, and cleanup effectiveness. Most reports include comparisons to baseline technologies as well as other competing technologies. Information about commercial availability and technology readiness for implementation is also included. Innovative Technology Summary Reports are intended to provide summary information. References for more detailed information are provided in an appendix.

Efforts have been made to provide key data describing the performance, cost, and regulatory acceptance of the technology. If this information was not available at the time of publication, the omission is noted.

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TABLE OF CONTENTS

1. SUMMARY	page 1
2. TECHNOLOGY DESCRIPTION	page 4
3. PERFORMANCE	page 7
4. TECHNOLOGY APPLICABILITY AND ALTERNATIVES	page 10
5. COST	page 12
6. REGULATORY AND POLICY ISSUES	page 14
7. LESSONS LEARNED	page 15

APPENDICES

A. REFERENCES	page A-1
B. RAPID FIELD SAMPLER EQUIPMENT LIST	page B-1
C. TABLES	page C-1

SECTION 1

SUMMARY

Technology Summary

The objective of the Empore™ membrane technology is to allow rapid and economical field sampling of surface and subsurface waters for heavy metal and radionuclide contaminants. At this time, membranes for the following elements have been tested: strontium (Sr), technetium (Tc), cesium (Cs) and lead (Pb). Analysis of the sampled contaminants is subsequently performed either in the field or at a remote analytical laboratory.

Applications of this technology include routine monitoring of sites having known contamination and the characterization of sites where the extent of contamination has not yet been determined. Samples for assay may originate from surface waters or from the subsurface via monitoring wells. DOE sites with significant surface or groundwater monitoring programs include Savannah River, Fernald, Oak Ridge, Rocky Flats and Idaho.

The baseline method for assay of site water for heavy metal and radionuclide contamination is to obtain samples and then to transport the samples to chemical laboratories for time-consuming chemical separation and analysis. In comparison, the Empore™ membrane technology can eliminate assay accuracy problems caused by sample transport, reduce transport costs and safety considerations, reduce secondary waste generation, reduce assay costs, and produce assay results with a shorter turn-around time. Routine monitoring tasks are thus performed more rapidly and at lower costs.

The Empore™ membrane is a commercial separation technology developed and patented by 3M. It consists of a method for enmeshing sorbent, surface-active particles in a web-like matrix, which is formed into a membrane. The particles are a product of molecular recognition technology and allow selective adsorption of heavy metals or radionuclides present as ions in aqueous solutions. The membrane is designed to have the integrity and handling strength for long-term performance under high levels of radiation across a broad range of pH and has a high particle surface availability. The combination of sorbent particles and membrane technology can be used to selectively remove specific contaminants down to predetermined detection levels at high flow rates.

This heavy metal and radionuclide separation technology has been adapted to two important DOE Environmental Management applications: the sampling and subsequent analysis of water samples for heavy metals and radionuclides and the treatment of radioactively contaminated water during the Decontamination and Decommissioning (D&D) process. The water treatment application is described in a separate Innovative Technology Summary Report ("Empore™ Membrane Separation Technology" demonstrated at Chicago Pile 5 (CP-5) Research Reactor Large-Scale Demonstration Project, Argonne National Laboratory-East, Argonne, Illinois). This Summary Report describes the use of the Empore™ membrane technology for sampling and subsequent analysis. More detailed descriptions of the radionuclide analyses are described in the publications cited in Appendix A.

Demonstration Summary

This report covers the period of 1995 to 1997 during which several site demonstrations of this technology were performed. To date, the Empore™ membrane technology has been demonstrated at four sites: the Savannah River Site (SRS) in 1995, 1996, and 1997; the Paducah Gaseous Diffusion Plant (PGDP), Paducah, KY in 1997; the West Valley Demonstration Project (WVDP), West Valley NY in 1997; and the General Electric (GE) storage facility, Morris, IL in 1997.

The most extensive demonstrations have been done at SRS for the sampling and analysis of Tc, Sr, and Cs isotopes as well as for Pb in 1997. Samples were volumes of groundwater taken from monitoring wells at various SRS locations and volumes of surface water taken from two different locations. Five individual groundwater monitoring wells were sampled for Tc at the PGDP.



Process water was sampled in the cases of the demonstrations at West Valley, NY and Morris, IL. At the WVDP location three sites were sampled for Tc and one site for Pb. At the GE storage facility, Morris, IL, water from the storage pool was sampled for both Tc and Sr. At all demonstration locations site personnel actively cooperated in the sampling and analysis activities. Points of contact for the site personnel are listed at the end of this section.

Assays performed for all elements subsequent to the sampling done with the Empore™ membranes for these demonstrations compared well with assays done using baseline technologies. The advantages offered by the Empore™ membrane technology included simpler sample transportation, more rapid return of assay results, and lower costs.

Empore™ membrane disks for Sr, Cs, Ra (radium), and Pb are commercially available from 3M Inc., St. Paul, MN. Disks for Tc sampling have already been sold to SRS in significant quantities. A water sampling system adapted for use with the Empore™ membrane disks is also commercially available from ISCO Inc, Lincoln, NE.

Future plans call for the enhanced development of the Empore™ sampling and analysis system. Selectively adsorbing particles are being developed for other ions containing U, Pu, and other actinides. Also under development are Empore™ membranes which are self indicating to allow rapid analysis to be done with simple instrumentation at the point of sampling.

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All published Innovative Technology Summary Reports are available on the OST Web site at <http://em-50.em.doe.gov> under "Publications." The Technology Management System, also available through the OST Web site, provides information about OST programs, technologies, and problems. The OST Reference # for **Rapid Sampling Using 3M Membrane Technology** is 1514.



SECTION 2

TECHNOLOGY DESCRIPTION

Overall Process Definition

The overall objective of the demonstrations was to establish the applicability of 3M Brand Membrane Technology in the sampling/analysis and remediation of liquids containing radionuclides and related metals. The primary focus of the Savannah River Site (SRS) demonstrations in 1995 and 1996 was on determinations of technetium (Tc), strontium (Sr) and cesium (Cs) using Empore™ technology. Determinations of Tc and lead (Pb) using rapid water sampling systems were demonstrated in 1997 at PGDP, SRS, WVDP, and GE Morris, IL. Use of the Empore™ Rad Disks is expected to:

- Reduce the number of method steps required to reach equivalent or better results provided by conventional methods by as much as 90% (laboratory preparation steps such as preconcentration, precipitation, filtration, purification, elution, and evaporation can be avoided).
- Dramatically reduce turnaround time.
- Increase laboratory throughput without increasing personnel or equipment levels.
- Decrease error frequency.
- Reduce reagent and waste volume.
- Reduce overall per-sample cost.

Empore™ Rad Disks are novel products designed to replace conventional radiochemical sample preparation methods using wet chemistry or packed columns. The Empore™ Rad Disks selectively target radioisotopes from aqueous samples, even in the presence of very large concentrations of competing ions. Because of the specificity of the Empore™ Rad Disks, radioisotopes of different elements simply pass through the disk and remain in the filtrate. Empore™ Rad Disks are a combination of Empore™ technology, which contains reactive particles in a membrane structure, and molecular recognition technology, which allows selective adsorption of a target isotope. The sorbent, surface-active particles are densely packed in the Empore™ disk, resulting in very efficient sorption of the target radioisotope in a shallow surface layer of the disk. Because no adhesives or binders are used to hold the particles, the full chemical activity of the particles is retained. Details of the resins used for these studies may be obtained from the 3M New Products Department cited in Section 1.

The disks are housed in individually molded polypropylene holders which serve to protect the disks during field transport, to provide leak-free service during pressurized sample loading, and to seal the loaded disks during shipment. Water is collected from the body of water using a standard ISCO 3700 series peristaltic pump and controller. The water is then passed through a 0.45- μm particle prefilter and into a sealed reservoir with a one-way valve. Next, the water is passed through a filter housing containing the Empore™ disks and collected in a weighed container. Multiple radionuclides can be collected during one sample-collection period by using sequential Empore™ disks. Based on tests conducted by SRS, the strontium collection disk should be first in the series, followed by the cesium collection disk, and finally the technetium collection disk. Experimental data have show that, even when the disks are placed in contact with each other in a single filter housing, no evidence exists of interference between the various radioisotopes. The Tc-Pb demonstrations used a similar system with the Tc disk placed before the Pb disk; a battery-powered balance was used to measure the amount of liquid processed through a disk. Figure 1 shows a picture of a sampling system, and Figure 2 shows a schematic of the design used in the 1997 demonstrations.





Figure 1. 3M Empore™ filter.

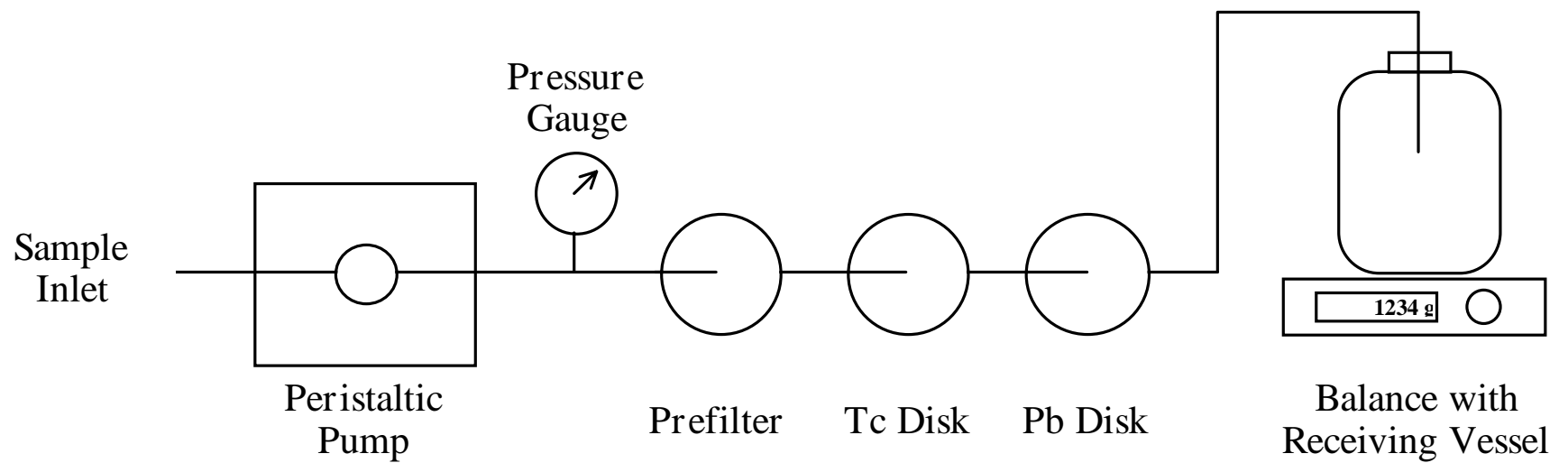


Figure 2. Schematic of sampling system.



System Operation

The ISCO/Empore™ sampler is designed to collect a composite sample over the course of a week or two; the results are the average concentration of the analyte of interest over the time period covered. It is designed for extended, unattended, routine monitoring. Alternatively, the Empore™ disks can be used to process individual grab samples in the field with a water sample pumped by a peristaltic pump through a filter holder containing the disks.

The sampler is portable and may be set in place by one technician. An example of a Rapid Field Sampler equipment list is shown in Appendix B. The sampling pump and top-loading balance require a power source, but they may be operated by a solar-panel-rechargeable battery if necessary. The 3M disk and disk holder must be replaced for each new sample taken. The field system does not introduce any secondary waste streams.

Only the disks need to be transported to a laboratory, and sample preparation for counting for three analyses can be performed by a single technician within a few hours. Some current work is directed toward the production of self-indicating disks so that determinations may be completed entirely in the field, but the demonstrations completed at the date of this report do not include this feature. The system is designed primarily for determinations of radionuclides and/or lead at low concentrations in water, so there are no potential safety or environmental concerns specific to this sampling system.

For the demonstrations described in Section 3, samples were collected using flow rates ranging from a few milliliters per min to over a liter per min. To accommodate a wide range of analyte concentrations, sample volumes ranged from 1.0 to 12.0 liters, but there is no general arbitrary limit. Only the disks in their holders were transported to the off-site laboratories. Analyses of the radionuclides collected on the disks were performed using liquid scintillation and solid scintillation counting. The disks were removed from the disk holders, dried in an oven, and placed in scintillation vials with scintillation counting solution. The Pb disks were digested with nitric acid (EPA Method 3050a) and then analyzed by inductively coupled plasma - mass spectrometry (ICP-MS) to get the lowest detection limits possible; other simpler standard methods for Pb analysis could be used for samples with higher concentrations.

The baseline methods used for comparisons are cited in Table 1, Appendix C, as well as in the tables accompanying Section 3. In each case, one-liter samples were transported to the off-site laboratory.



SECTION 3

PERFORMANCE

Demonstration Plan

The demonstrations during FY 1995 and FY1996 were intended to compare the sampling and analysis system with conventional methods for determinations of ^{99}Tc , ^{90}Sr , and ^{137}Cs concentrations in surface waters at the Savannah River Site (SRS) in Aiken, South Carolina.

During FY 1997, determinations of ^{99}Tc and of Pb were compared to the results obtained by conventional methods for the following sites:

- Five groundwater monitoring wells at the Paducah Gaseous Diffusion Plant (PGDP), Paducah, KY.
- Two groundwater and two surface water locations at SRS.
- Three process-water sites and one groundwater monitoring well at the West Valley Demonstration Project (WVDP), NY.

Also during FY 1997, determinations of ^{99}Tc and ^{90}Sr in water samples from the spent fuel rod storage pool at the General Electric (GE) storage facility, Morris, IL were compared to the results obtained by baseline methods.

Conventional determinations of radionuclides and other species in surface and ground waters require samples to be collected in the field, transferred to a laboratory and stored until the proper analytical sample preparation and analyses can be conducted. The demonstrations reported here used the field-portable sampling system described in Section 2, and the quantitative results were compared to those obtained by conventional sampling procedures. One-liter samples were collected for each determination by a baseline method, and these samples were transported to the off-site laboratory for the analyses described below.

The program objectives were to:

- Capitalize on 3M's capability of packaging surface reactive particles of a wide variety of reactive chemistries into porous membranes suitable for radionuclide and lead determinations at DOE sites.
- Choose or develop solid phase extraction particles with a high degree of specificity for the target analytes.
- Reduce the sample preparation process by simply passing the sample fluid through the membrane on site followed by analyses of the disks in a lab (or, in the future, directly at the field site).
- Transfer the technology for an initial product to end users through the commercialization process.

Results

Interference from Competing Ionic Species. Selective adsorption of the radioisotope onto the filter has been shown to be a function of the concentration of the radioisotope and the concentration of competing ions. For example, the manufacturer does not recommend the use of the disks for Sr determination if the total Sr content of the sample exceeds 3 mg. Quantitative recovery may be sacrificed beyond this threshold value because stable Sr (i.e., non-radioactive) competes for adsorption onto the disk. Additionally, extremely high levels (1%) of Na^+ , NH_4^+ , K^+ , and Ca^{+2} can interfere with the uptake of Sr by the disk. Minimal (if any) interference is seen at concentrations at or below 100 ppm. For most natural samples, concentrations of potential interfering ions will be well below 100 ppm, and are not anticipated to interfere.

Potential radiometric interferences can be caused by the presence of Ba, Ra, and Pb. Radioactive isotopes and/or daughter products of these three elements can pose radiometric interferences. ^{140}Ba will interfere when waters containing fresh fission products are being analyzed. Beta-emitting isotopes and



daughter products of Ra may pose interference problems. Pb may be retained on the disk, but because Pb compounds are very insoluble, the potential interference from ^{210}Pb should be insignificant.

The methods have not been evaluated for all possible matrices and interferences. Method suitability should be determined on specific waters of interest.

^{99}Tc , ^{90}Sr , and ^{137}Cs Determinations in Savannah River Site (SRS) Surface Waters. Results obtained at SRS during the study period show that significant improvements can be made over current quantification technologies. The method is applicable to field use, concentrating aqueous samples in the field such that transport of large volumes of water is avoided and no additional preservation of the samples is required if final analysis in the laboratory is delayed. By correctly choosing the disk, one can extract only the analyte of interest from the sample, thus reducing or eliminating the chemical purification required before final activity determination.

Radionuclide concentration data (obtained using the ISCO/Empore™ filter system) were available within 1 week of sample collection instead of the more typical 45-day turnaround time for routine monitoring programs now in use at SRS. Sample preparation for counting for three different analyses was performed within a few hours using only one technician. Current protocol requires multiple technicians performing separate lengthy chemical separations for each analyte. Also, the new method resulted in a lower detection limit because larger (several liters) sample volumes can be processed in the field, whereas only 1 liter is used for laboratory methods. The sample analysis cost is reduced by processing samples in the field, eliminating the need for multiple technicians performing lengthy laboratory methods.

Radionuclide concentrations (^{99}Tc , ^{90}Sr , and ^{137}Cs) in the streams for the samples collected and processed during specified study periods show strong agreement with traditional sampling and analyses results (see Appendix C, Table 2). Technetium samples were not determined using baseline methods during this study period.

Based upon studies conducted after this testing period, SRS has indicated that radium present in the samples may bias the results when determining Sr by the 3M Empore™ filter technology. This may explain why the measured concentrations are usually greater than for those obtained using the baseline technology.

^{99}Tc Determinations in Paducah Gaseous Diffusion Plant (PGDP) Ground Waters. Five groundwater monitoring wells were sampled, with a total of 40 samples collected on Empore™ Tc Rad Disks. Samples were collected at flow rates from 150 to 440 mL/min, and sample volumes ranged from 1.0 to 12.0 L. A total water volume of 116 liters was processed through the membranes. Off-site analysis of the membranes for ^{99}Tc was performed at Argonne National Labs (ANL) by liquid scintillation (LSC) and solid scintillation counting (SSC). Water samples were also analyzed for ^{99}Tc by two baseline methods: DOE Compendium Method 550 and ICP-MS.

The results from the Paducah testing are shown in Appendix C, Table 3 and are summarized in Figure 3. It was shown that 10 of the 11 data sets generated using liquid scintillation counting in three laboratories yielded results that were statistically equivalent to the standard method at the 95% confidence level. The three data sets generated for the beta spectrometer (SSC) were also equivalent to the standard method. All components of the sampling hardware were shipped, set up, and operated without damage. The samples collected on the Empore™ membranes were particularly convenient to transport, particularly when compared to the cumbersome transport of actual water samples. Pre-filtering was required for these ground waters with high solids content and was convenient to implement using quartz fiber pre-filters.

^{99}Tc and Pb Determinations in SRS waters (June 1997). Two groundwater and two surface water locations were used to obtain a total of 27 samples, with a total of 62 L of groundwater sampled. Collected samples were taken from 1.0 to 14.0 L, depending on the contaminant level, but only the disks were transported to the laboratory. The general procedures were similar to those cited for the Paducah samples cited above, and the results are shown in Appendix C, Table 4. Tc contamination ranged from 2 to 50 pCi/L, and the results from LSC membrane determinations for the 50 pCi/L samples were statistically equivalent to the standard methods. Pb was detected in the 10 microgram/L (parts per billion) range as determined by ICP-MS, and unfortunately this low level prevented meaningful comparisons of results.



⁹⁹Tc and Pb Determinations in the West Valley Demonstration Project (WVDP) Process Water Sites.

Field sampling was carried out at the West Valley Demonstration Project. Waters for the Tc studies were drawn from three-process water sites, and purge water from a groundwater monitoring well was used for the Pb test. Because of site regulations, technicians from West Valley did the actual field tests. The system withstood rough handling during shipment and field use, and the West Valley staff found the sampler easy to use. They quickly became proficient in operating the sampler, and it took them less than five minutes between samples to replace the Empore™ disks.

For the 18 samples collected for Tc analyses, typical sample volumes were 3 to 4 L. Average flow rates were of the order of 200 mL/min (when larger quantities of algae were present) up to 400 mL/min; prefiltering was performed with 0.45 µm capsule filters. The results are shown in Appendix C, Table 5. The membrane ⁹⁹Tc values determined by solid scintillation counting were in good agreement with the concentrations determined by ICP-MS and by extraction chromatography (DOE Method RP550), indicating that the membranes were efficient at removing the Tc from these waters. However, the values obtained from liquid scintillation counting were statistically significantly lower. The heavy organic coating of the disks by algae or some other matrix constituent poses a physical barrier to intimate contact of the Tc ions with the scintillator, thereby decreasing the efficiency of light production. In cases where similar problems are anticipated, a standard addition method in the field would be beneficial. Finally, no Pb was detected in the monitoring well by either the standard method or by the membrane determination, so no meaningful comparison was achieved.

⁹⁹Tc, ⁹⁰Sr, and ¹³⁷Cs Determinations in the General Electric (GE) Process Water Test Sites: Water samples from the spent fuel rod storage pool (basin) were collected on disks for technetium (⁹⁹Tc) and for strontium (⁹⁰Sr). An experimental membrane for cesium (¹³⁷Cs) was also tested. The results of these determinations are shown in Appendix C, Table 6. A gross alpha-beta measurement showed 1.8×10^6 dpm/L, and a gamma count revealed this activity to be mostly from ¹³⁷Cs (1.7×10^6 dpm/L) and ⁶⁰Co (1.7×10^5 dpm/L). Three ⁹⁰Sr separations were performed using a 500 mL basin water sample mixed 1:1 with 4 M nitric acid as required by the strontium membrane solid-phase extraction chemistry. To prevent secondary absorptive contamination of the Tc disk by the high concentrations of ¹³⁷Cs and ⁶⁰Co in the water, the basin samples were pre-treated by passing the sample through three consecutive Empore™ cation exchange disks. This reduced the measured concentrations of Cs and Co by a factor greater than 10^5 , but did not affect the pertechnetate anion. Samples were then made 0.1M in hydrochloric acid to further reduce secondary absorption of the Cs and Co nuclides.

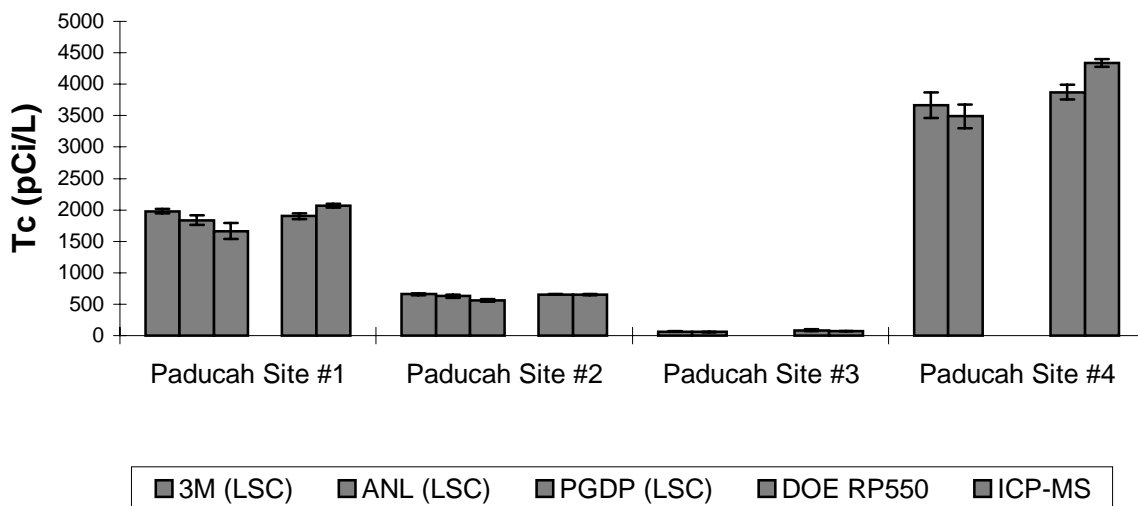


Figure 3. Data summary for rapid water sampling technology, Paducah demonstration.

SECTION 4

TECHNOLOGY APPLICABILITY AND ALTERNATIVES

Competing Technologies

The primary competing technology is the current baseline technology, which involves collection of large volumes of sample followed by traditional, wet chemistry laboratory analysis, as described in the following subsections and in Table 1, Appendix C. The baseline methods require highly trained personnel who are familiar with separation techniques such as extraction chromatography and coprecipitation as well as with the operation of sophisticated analytical instrumentation.

Technetium-99 (⁹⁹Tc) Analysis. Current analysis methods for ⁹⁹Tc in aqueous samples require the collection of at least 1 L of water. Samples are analyzed by isotope dilution/inductively coupled plasma-mass spectroscopy (ID/ICP-MS) to achieve the required detection limit of 1 pCi/L or 0.06 ng/L. One nanogram (1.4 pCi) of ⁹⁷Tc (a different isotope of technetium used as an internal standard) is added to the sample as a tracer, and the technetium is extracted from the aqueous sample using an extraction chromatography column. Technetium is eluted from the column, and the eluent is analyzed by ICP-MS for quantification. This process takes a few days with most of the time required for the liter of water to pass through the column.

Cesium-134 and -137 (¹³⁴Cs and ¹³⁷Cs) Analysis. Current analysis for the determination of radiocesium requires the collection of 1 L of water, which is counted in a Marinelli beaker on a high-purity germanium detector (HPGe) for 10,000 s to achieve a detection limit of 8 to 10 pCi/L. Alternatively, a 10-L sample can be collected and passed through an ion exchange column, and the resin is counted for 10,000 s on an HPGe detector to achieve a detection limit of a few pCi/L.

Strontium-90 (⁹⁰Sr) Analysis. The current U.S. Environmental Protection Agency (EPA) procedure requires that multiple selective precipitations be performed to separate strontium from matrix elements. Because of the many steps involved, this approach is labor-intensive and time-consuming. Alternatively, the strontium may be extracted onto an extraction chromatography column. This step is volume-limited; thus, the sample must be concentrated by evaporation or precipitation before column extraction.

Lead Analysis. Current technology consists of collecting samples in the field, transporting them to a laboratory and analyzing the solutions using atomic spectroscopy or inductively coupled plasma - mass spectroscopy (ICP-MS). The required instrumentation is expensive and must be used by a skilled analyst. Transportation of samples is cumbersome and may result in some losses of lead due to adsorption onto the walls of the container.

Technology Applicability

The principal application of the Empore™ disks is the preparation of aqueous samples for radiochemical analysis. The method is efficient, safe, reliable, and field-deployable. Multiple steps in the preparation of the sample and the source can be combined into a single step, thereby reducing labor costs and eliminating many potential sources of laboratory error, and reducing or eliminating the need for hazardous chemicals associated with traditional analytical procedures. Samples are easily batched, and, depending upon the analyte of interest, one-liter samples can be prepared in as little as 20 minutes. For example, 3M has developed a test method by which ⁹⁹Tc activity can be determined directly from the disk with no prior preparation of the sample or the disk. Cartridges containing 3M Empore™ membranes for the element-selective extraction of radioisotopes can be used for the remediation of groundwater, surface water, cooling water, process water and water used in nuclear fuel storage pools.

Monitoring for radionuclides in surface waters is required at nuclear facilities to fulfill regulatory compliance and to ensure the public safety. Most radionuclides are present in extremely low



concentrations and require extensive sample processing before activity determination can be performed. Usually, large sample volumes must be collected, preserved, transported, and analyzed (using a variety of evaporation, precipitation, and/or chemical separation techniques). After the samples arrive at the laboratory, sample preparation and analysis time often is several days; column extraction techniques account for a major portion of the time requirements, but they are the only acceptable alternatives now.

Patents/Commercialization/Sponsor

The 3M Company as an extension of their previous work has developed this technology with Empore™ disks for non-radioactive species, and the latter have been commercial products for some time.

There have been two patent applications and one patent granted to 3M for the new applications described here:

- U.S. Patent Application, "Self-Scintillating Sheet Material for Radioactive Species Analysis," submitted 1997.
- U.S. Patent Application, "Method for the Colorimetric Quantification of Ions in a Solution Using Ion Recognition Substrates Enmeshed in a Porous Matrix or Membrane," submitted 1996.
- U.S. Patent Number 5,637,506, "Solid Phase Extraction Using Composite Sheet for Direct Measurement of Radioactivity."

Thus, this technology has been privatized from its inception, it is a clear extension of previously commercialized technology, and the development of new technologies specifically for determinations of radioactive species has been patented. Some of the Empore™ Rad Disks that were developed and studied during the course of this work are now commercial products.



SECTION 5

COST

Introduction

The U.S. Department of Energy (DOE) is facing enormous financial burdens using current analytical methodology. The costs associated with environmental and waste management at government facilities are very high and continue to grow. Current estimates indicate that over one million analyses are required annually to support remediation efforts at DOE sites at a cost of over \$300M annually for the radiochemical analyses alone. Additionally, turnaround times (for some analyses) can be as long as 6 months. Long turnaround times slow down remediation efforts and contribute to missed milestones. Finally, many of the traditional procedures generate huge quantities of secondary wastes, which further add to DOE's problems.

The development and implementation of new analytical technologies can significantly reduce the costs associated with DOE programs. Disk solid-phase extraction technology has been proven to be highly effective for the analysis of organic compounds, wastewaters, and other aqueous samples. Disk technology improves sample throughput, reduces secondary wastes, and significantly reduces costs. In addition, commercial aqueous samplers have been developed to utilize the disk technology during automatic sampling.

Methodology

Two cost comparison studies were done to illustrate the cost effectiveness of the disk solid-phase extraction technology. The first study compares the use of a baseline analytical technique against the use of Empore™ Strontium Rad Disks for a typical effluent monitoring program such as the one at the Savannah River Site. Material and labor costs are totaled for each year of a 10-year life cycle. The present values of these annual costs are summed for both the baseline and innovative technologies, and the cost saving is presented in terms of present values. This study was prepared by the Energy and Environment Research Center at the University of North Dakota (EERC) (POC is David Murray, 701 777-5103).

For strontium analyses, the baseline technology is EPA Method 905.0, "Radioactive Strontium in Drinking Water". It measures total strontium and soluble strontium-89 and strontium-90 in drinking water. Cold strontium carrier is added to the drinking water sample and strontium-89 and strontium-90 are precipitated from the solution as insoluble carbonates. Interferences from calcium and some radionuclides are removed by one or more precipitations of the strontium carrier as strontium nitrate. Barium and radium are removed as their chromates.

From a second study, comparative information is provided for technetium-99, strontium-90, and cesium-137 analyses and is based on data obtained by the Savannah River Technology Center (SRTC) during FY 1995 through FY 1997. SRTC reviewed cost data obtained for measurement of several radionuclides during this time period using the innovative technology described herein and the baseline technology now in use at SRTC. In addition to cost savings, the second study found that the innovative technology offers the following advantages over the baseline technology:

- Using the innovative technology, much larger sample volumes are processed in the field, allowing for lower detection limits and more precise results.
- The larger uncertainties associated with the baseline methodology (because of lack of equipment sensitivity) can be avoided.
- Sample turnaround time is approximately 1 week in contrast to a baseline turnaround time of approximately 45 days. The baseline technology requires multiple technicians to perform separate lengthy chemical separations for each analyte. Critical steps of the baseline technology, such as the elution process (sample water passing through the extraction chromatography column for technetium analysis) are time consuming but cannot be shortened without sacrificing the integrity of the results.



Cost Comparison

Appendix C, Table 7 presents the results of the EERC cost comparison between the EPA technology and the innovative technology for strontium analysis. For a 10-year life cycle, this study concludes that just for Strontium-90 analysis alone a present-value savings of \$400,000 can be obtained at the Savannah River Site. Most of these savings are obtained from reduced labor costs. Savings in the areas of transportation costs and sample disposal were not included in the EERC study.

In the case of transportation cost savings, one can assume conservatively that if samples must be sent off site for assay, shipping costs will be approximately \$100 per shipment. If a shipment is made each day sampling takes place and if 5 samples per day are taken, then 150 shipments per year will be required to transport the 756 samples typically collected at a cost \$15,000. Thus an additional cost savings of \$15,000/yr can be realized by eliminating the need to transport samples off site.

Appendix C, Table 8 summarizes the baseline and innovative technology per-sample cost information obtained by SRTC during the study period (FY 1995 to FY 1997) for technetium-99, cesium-137, and strontium-90. The cost savings obtained for strontium-90 analyses via the disk solid-phase extraction technology are comparable to the savings predicted by the EERC study. In addition, comparable savings are obtainable for technetium-99 analyses. The cost of assays for cesium-137 is found to be roughly equivalent.

Cost Conclusions

The final results of the comparison study between EPA method 905.0 and the innovative technology showed that:

- The present value of using EPA method 905.0 over a 10-year period to analyze 756 samples per year for strontium-90 is \$682,000.
- The present value of using the Strontium Rad Disks for the same number of samples and analyses is \$282,000.
- The present value of the overall savings is then \$400,000.
- An additional savings of \$15,000/yr in transportation costs is possible.
- Significant savings in the cost of technetium-99 analyses are possible.
- The savings in the cost of cesium-137 analyses are less significant.

From the SRTC data, it was found that the total cost savings would be dependent upon the number of samples taken. Additional savings in the cases of cesium-137 and strontium-90 would be seen in reduced turnaround times because lengthy laboratory preparation and analyses times would not be required.



SECTION 6

REGULATORY AND POLICY ISSUES

Regulatory Considerations

The Savannah River Technology Center (SRTC) completed an environmental evaluation checklist for the National Environmental Policy Act (NEPA)/Environmental Permit before conducting the field sampling. They determined that testing was covered under the site-wide Categorical Exclusion (CX) B3.10.

Safety, Risks, Benefits, and Community Reaction

Safety. The U.S. Environmental Protection Agency (EPA) considers radionuclides to be as Class A carcinogens. Because DOE places a high value on protection of the public and the environment, DOE regularly monitors the releases of radionuclides at or near its facilities. Monitoring data are required to determine the possible dosage received from exposure. Isotopes of concern, which may be determined using this technique, include the following elements:

- Technetium, an unstable element, exists only through its production in nuclear reactions. The isotope ^{99}Tc is a weak beta emitter that may be concentrated by plants and tends to localize in the gastrointestinal tract and thyroid glands of animals and humans.
- As an analog of potassium, cesium is absorbed through the bloodstream and is considered a “whole body” concern. The cesium isotopes of concern are ^{134}Cs and ^{137}Cs , both of which are gamma-emitting radionuclides produced by activation and fission processes.
- Strontium is suspected to replace calcium in mammals. In particular, ^{90}Sr is a beta-emitting radionuclide produced during fission processes.
- Lead can be absorbed by the lungs and the digestive tract from air and water sources. After absorption, lead enters the bloodstream, where it distributes to all tissues of the body. Excessive levels of lead can damage the brain, kidneys, nervous system, red blood cells and reproductive system. The degree of harm is directly related to the level of lead in the blood (from all sources). Known effects of exposure to lead range from subtle changes in body chemistry and nervous system function at low levels of exposure, to severe toxic effects or even death at very high levels associated with acute poisoning. Some harmful effects are reversible if exposure is reduced, while other harmful effects can be permanent.

Worker and Community Safety. Worker exposure for this technology would be similar to that of alternative technologies, but the addition of autosampling techniques, along with reduced sample preparation time, may reduce worker exposure to the sample. The general public has little risk of exposure. Radionuclide concentrations in surface water are generally below maximum allowable levels.

Environmental Impact. No significant safety problems are associated with this method other than the normal precautions needed for handling radioactive materials, acids, and bases. The technology discussed herein is used as an analytical tool to determine concentrations of radionuclides in large volumes of water. Testing showed that the concentrations of radionuclides in bodies of water at SRTC are below regulatory requirements.

Socioeconomic Impacts and Community Reaction. The community has very little familiarity with this technique, but the general perception of the use of radionuclear materials is negative. At the same time, the public exhibits a great eagerness to monitor the potential for radionuclide releases and to determine the potential for radionuclide exposure to public water systems. The use of this technology is not expected to affect community safety or socioeconomic issues.



SECTION 7

LESSONS LEARNED

Implementation Considerations

This technology is used as an analytical tool, implementation considerations are limited primarily to proper placement and operation of the (auto) sampling equipment. The 3M membrane systems enable a five-fold increase in samples per day compared to conventional sample preparation procedures, and sample turnaround is reduced from weeks to days or hours. Selective capture of analytes diminishes potential interferences associated with conventional analytical sample preparation.

Technology Limitations and Needs for Future Development

In general, the use of this technology has few limitations. The following limitations were encountered during testing:

- As with all mechanical equipment (pumps, valves, etc.), general maintenance (cleaning/repair, lubrication, etc.) is required.
- One of the resins for cesium removal was designed to function in alkaline solutions and is recommended only for use in streams where the pH is greater than about 5. An alternate disk may be used below pH 5 or in saline environments, and the manufacturer should be consulted for recommended applications of the disks intended for cesium removal.
- Inlet tubs to the ISCO/ Empore™ autosamplers can become buried in mud or clogged by vegetation (stocks, leaves, etc.) and thus become unable to draw samples. Extreme declines in water levels could leave the inlet tubes exposed to air (above the water level). Autosamplers are battery powered with solar panels to regenerate the batteries. Consecutive rainy/cloudy days may result in battery drainage without the ability to regenerate the batteries.
- The ISCO/ Empore™ sampler may be used to collect a small-volume sample with high frequency, to achieve a composite sample (e.g., collect 30 mL every 30-min for a week gives a 10-L sample processed over a week) or it may be used to collect a larger sample in a short time. Because the volume delivery system is not as accurate as required, the volume of the processed water must be determined by weight.
- Although samples with high-suspended solids have been analyzed successfully, samples with high loadings of algae or other organic materials may require a standard additions calibration to correct for interferences with the liquid scintillation counting.

Technology Selection Considerations

The sampling system is most advantageous for groundwater or surface water sampling at remote locations. The total volume of sample passed through the membrane can be many liters for samples with very low analyte concentrations, and the volume can be reduced to avoid breakthrough for higher concentrations. Although the disks have high selectivity, they are best used for waters without extremely high radioisotope contaminant levels. Future work may make use of “self-indicating” disks to enable complete field determinations, but the current technology requires taking the disks to a laboratory for scintillation counting.



APPENDIX A

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APPENDIX B

RAPID FIELD SAMPLER EQUIPMENT LIST

<u>Item</u>	<u>Manufacturer</u>	<u>Catalog #</u>
Empore Disks in Disk Holders	3M	
Pre-Filters in Disk Holders	3M	
Field Sampling Stand	3M	
Disk Opener	3M	
L/S Priority Pollutant Sampling Pump	Cole-Palmer	E-07570-60
Pump heads (7021-15)	Cole-Palmer	E-07015-21
Tubing weight	Cole-Palmer	E-07570-04
Silicone tubing	Cole-Palmer	E-95802-15
Pressure gauge with couplings & tees	Cole-Palmer	Gauge: E-68002-03 Coupling: E-06349-00 Tees: E-06452-20
Tubing clips (for pressure gauges)	Cole-Palmer	E-06832-02
Tubing clips (for disk holders)	Cole-Palmer	E-06832-06
0.45 μ Capsules	Fisher	09-743-37
Ohaus Portable Toploading Balance	Fisher	01-918-14
I-Chem Environmental Cubitainer (4L)	Fisher	05-719-303B
I-Chem Environmental Cubitainer (1L)	Fisher	05-719-308



APPENDIX C

TABLES

Table 1. Summary of baseline methods

Baseline method	Operator skills required	Secondary waste generation	Potential operation concerns
⁹⁹ Tc by isotope dilution/inductively coupled plasma-mass spectrometer (ID/ICP-MS)	Knowledge of wet chemistry for sample preparation and of the use and maintenance of the ICP-MS	Acidified sample solution containing ⁹⁹ Tc and added tracer, acidified calibration solutions	Need to maintain the ICP-MS in a controlled environment
¹³⁴ Cs and ¹³⁷ Cs counted on high purity germanium (HPGe) detector	Training in gamma-spectroscopy	None	HPGe detector requires controlled environment
⁹⁰ Sr EPA method	Knowledge of wet chemistry for sample preparation, purification and separation, and of the use of the appropriate instrument (gas-flow proportional counter or liquid scintillation counter)	Acidic solutions generated during the purification and separation steps	Classical separation techniques are very labor-intensive
Pb by atomic emission spectroscopy (AES) or ICP-MS	Knowledge of the use and maintenance of the AES or the ICP-MS	Acidified sample solution, acidified calibration solutions	Need to maintain the AES or the ICP-MS in a controlled environment



Table 2. Radioisotope determinations in SRS surface waters

Volume (mL)	Baseline Methods ¹		Membrane Methods		
	Strontium (pCi/L)	Cesium (pCi/L)	Strontium (pCi/L)	Cesium (pCi/L)	Technetium (pCi/L)
5,400	8.4 ± 0.7 ²		18.0 ± 0.9	NC	0.6 ± 0.1
7,000			17.5 ± 0.8	NC	0.4 ± 0.1
3,700		1.2 ± 2.7	20.1 ± 1.1	3.8 ± 1.2	0.6 ± 0.1
5,500	7.3 ± 0.7		10.0 ± 0.6	3.3 ± 0.8	1.1 ± 0.2
1,450		9.9 ± 3.3	6.6 ± 0.9	3.8 ± 2.2	0.4 ± 0.1
6,600			1.4 ± 0.1	2.7 ± 2.5	0.64 ± 0.03
15,000		3.2 ± 1.6	1.4 ± 0.1	2.9 ± 2.5	0.05 ± 0.02
8,500			7.9 ± 0.4	2.8 ± 0.7	0.7 ± 0.1
7,000	9.2 ± 0.8	3.7 ± 2.3	13.3 ± 0.6	2.6 ± 0.7	0.6 ± 0.1
5,000			12.5 ± 0.7	3.3 ± 1.0	0.6 ± 0.1
7,000		9.9 ± 3.8	9.1 ± 0.5	2.5 ± 0.8	0.4 ± 0.1
8,000			9.0 ± 0.5	3.0 ± 0.7	0.6 ± 0.1
8,000		7.5 ± 1.7	8.0 ± 0.5	3.8 ± 0.7	0.6 ± 0.1
5,500	11.0 ± 1.2		5.1 ± 0.4	3.3 ± 0.8	0.3 ± 0.1
6,000		10.3 ± 3.5	7.7 ± 0.5	3.5 ± 0.9	0.6 ± 0.1
7,000			8.0 ± 0.5	3.4 ± 0.8	0.5 ± 0.1

¹ Historical ⁸⁹Sr and ⁹⁰Sr (1993): 10 to 15 pCi/L
 Historical ¹³⁷Cs (1993): 8.8 to 19.0 pCi/L
 Historical ⁹⁹Tc (1992 - 1994): 0.57 to 3.1 pCi/L

² Uncertainties are the 2σ errors



Table 3. ⁹⁹Tc Determinations in PGDP ground waters

⁹⁹ Tc (pCi/L)						
Sample Site	Standard Methods		Membrane Methods			
	ICP-MS ^a	DOE RP550 ^b	LSC ^c (ANL)	LSC (3M)	LSC (PGDP)	SSC ^d (ANL)
MW 66 (Test Site 1)	2069 ± 29	1901 ± 46 (N=3)	1838 ± 80 (N=4)	1979 ± 36 (N=3)	1664 ± 36 (N=2)	1995 ± 20 (N=2)
MW 155 (Test Site 2)	652 ± 12	656 ± 3 (N=3)	627 ± 26 (N=4)	657 ± 13 (N=3)	562 ± 13 (N=3)	666 ± 3 (N=3)
PZ 108 (Test Site 3)	72 ± 2	80 ± 12 (N=5)	58 ± 3 (N=2)	64 ± 4 (N=2)	N/A	N/A
MW 262 (Test Site 4)	4340 ± 60	3873 ± 116 (N=3)	3649 ± 27 (N=2)	3843 ± 73 (N=2)	N/A	3789 ± 75 (N=2)
MW 255 (Test Site 5)	11 ± 1	6 ± 2 (N=3)	N/A	3.6 ± 0.1 (N=2)	N/A	N/A

^a Inductively coupled plasma-mass spectrometry; ^b DOE Methods Compendium procedure;

^c LSC= liquid scintillation counting; ^d SSC= solid scintillation counting



Table 4. ⁹⁹Tc and Pb determinations in SRS ground waters (6/97)

⁹⁹Tc (pCi/L)						
Sample Site	Standard Method		Membrane Determinations			
	ICP-MS ^a	DOE RP550 ^b	LSC ^c (ANL)	LSC (3M)	GPC ^d (ANL)	SSC ^e (ANL)
4MA7 (Test Site 1)	4 ± 1	9 ± 1 (N=3)	< 3 (N=4)	< 3 (N=1)	7.5 ± 2.3 (N=4)	N/A
A003 (Test Site 2)	10 ± 1	< 3 (N=3)	< 3 (N=3)	< 3 (N=3)	1.6 ± 0.1 (N=3)	N/A
FSB93 (Test Site 4)	51 ± 2	47.4 ± 2.3 (N=3)	48.2 ± 1.0 (N=3)	50.3 ± 0.20 (N=3)	48 ± 2 (N=3)	60 ± 5 (N=3)
Pb (mg/L)^f						
	Standard Method (Soluble Pb)		Membrane Determinations			
21A (Test Site 3)	17.2 ± 1.7		9.6 ± 3.6			

^a Inductively coupled plasma-mass spectrometry; ^b DOE Methods Compendium procedure; ^c LSC= liquid scintillation counting; ^d GPC= gas proportional counting (A counting efficiency calibration as a function of flow rate was applied to this data); ^e SSC= solid scintillation counting.

^f A small quantity of Pb was found on the prefilters indicating that particulate Pb may be present in the sample. Pb was found at up to 5 times the detection limit on the Pb disks. No Pb breakthrough was observed.



Table 5. ⁹⁹Tc and Pb determinations at the West Valley Demonstration Project

⁹⁹Tc (pCi/L)					
Sampling Site	Standard Method		Membrane Determinations		
	ICP-MS ^a	DOE RP550 ^b	LSC ^c (ANL)	LSC (3M)	SSC ^d (ANL)
North Interceptor (Test Site 1)	762 ± 32	792 ± 39 (N=2)	675 ± 20 (N=2)	701 ± 22 (N=3)	793 ± 41 (N=3)
LLWTF Effluent (Test Site 2)	431 ± 12	466 ± 53 (N=3)	203 ± 15 (N=3)	307 ± 10 (N=3)	437 ± 18 (N=3)
LLWTF Feed (Test Site 3)	396 ± 13	385 ± 13 (N=3)	218 ± 14 (N=3)	279 ± 10 (N=3)	414 ± 41 (N=3)
Pb (mg/L)					
	Standard Method ICP-MS		Membrane Determinations		
Monitoring Well #602 (Test Site 4)	Not detected		<3 mg		

^a Inductively coupled plasma-mass spectrometry; ^b DOE Methods Compendium procedure; ^c LSC= liquid scintillation counting; ^d SSC= solid scintillation counting



Table 6: Results from GE-Morris fuel basin site

⁹⁹Tc Determination			
Sample Description	Volume (Chemical Treatment)	Detection Method	pCi/L
Tc-5 disk	1 L (0.1 M HCl)	SSC ^a	≤ 30 *
Tc-6 disk	1 L (0.1 M HCl)	SSC	≤ 23 *
Basin water		ICP-MS ^b	4 ± 1
Pretreatment for ⁹⁹Tc Determination Using Empore™ Cation Disks			
Untreated basin water	1 L (ambient)	Intrinsic Ge detector	7.65 x 10 ⁵ ¹³⁷ Cs 8.0 x 10 ⁴ ⁶⁰ Co
Treated basin water	1 L (ambient)	Intrinsic Ge detector	≤ 20 ¹³⁷ Cs ≤ 10 ⁶⁰ Co
⁹⁰Sr Determination			
Sr-1 disk	500 mL (2 M HNO ₃)	SSC	≤ 1500 *
Basin water	10 mL (2 M HNO ₃)	DOE RP500 ^c + GPC ^d	660 ± 60
* Residual ¹³⁷ Cs and ⁶⁰ Co present.			

^a SSC= solid scintillation counting; ^b Inductively coupled plasma-mass spectrometry;

^c DOE Methods Compendium procedure; ^d GPC= gas proportional counting



Table 7. Life cycle cost estimate for strontium analyses

Baseline Technology: EPA Method 905.0												
		Year 1	Year 2	Year 3	Year 4	Year 5	Year 6	Year 7	Year 8	Year 9	Year 10	
756 Determinations per Year												
Reagent Cost per Sample	\$4	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024
Sample Preparation Material	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0
Total Material Cost Per Sample	\$4	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024	\$3,024
Time per Sample	2 Hr.											
Samples per Hour	0.5											
Labor Cost per Sample(1)	\$100	\$75,600	\$75,600	\$75,600	\$75,600	\$75,600	\$75,600	\$75,600	\$75,600	\$75,600	\$75,600	\$75,600
Total Cost per Sample	\$104											
Total Annual Cost		\$78,624	\$78,624	\$78,624	\$78,624	\$78,624	\$78,624	\$78,624	\$78,624	\$78,624	\$78,624	\$78,624
Total Life cycle Cost (\$1997)	\$786,240											
Total Life cycle Present Costs	\$682,324											
Innovative Technology: 3M Empore Sr Rad Disk												
Reagent Cost per Sample	\$2	\$1,512	\$1,512	\$1,512	\$1,512	\$1,512	\$1,512	\$1,512	\$1,512	\$1,512	\$1,512	\$1,512
Sample Preparation Material	\$36	\$27,216	\$27,216	\$27,216	\$27,216	\$27,216	\$27,216	\$27,216	\$27,216	\$27,216	\$27,216	\$27,216
Total Material Cost Per Sample	\$38	\$28,726	\$28,726	\$28,726	\$28,726	\$28,726	\$28,726	\$28,726	\$28,726	\$28,726	\$28,726	\$28,726
Time per Sample	6 Min.											
Samples per Hour	10											
Labor Cost per Sample(1)	\$5	\$3,780	\$3,780	\$3,780	\$3,780	\$3,780	\$3,780	\$3,780	\$3,780	\$3,780	\$3,780	\$3,780
Total Cost per Sample	\$43											
Total Annual Cost		\$32,508	\$32,508	\$32,508	\$32,508	\$32,508	\$32,508	\$32,508	\$32,508	\$32,508	\$32,508	\$32,508
Total Life cycle Cost (\$1997)	\$325,080											
I Value for Discount Calculation		0	1	2	3	4	5	6	7	8	9	
Present Value of Total Annual Cost(2)		\$32,508	\$31,470	\$30,464	\$29,491	\$28,549	\$27,637	\$26,754	\$25,899	\$25,072	\$24,271	
Total Life cycle Present Costs	\$282,115											
Present Value of Savings	\$400,209											
Notes												
(1) \$50 Per hour Assumed Labor Rate												
(2) 3.3 Percent Discount Factor												



Table 8. Comparison of cost per sample with innovative method and SRTC baseline method

Method	Technetium-99		Cesium-137		Strontium-90	
	Cost/sample ^a	Comments	Cost/sample ^a	Comments	Cost/sample ^a	Comments
Baseline	\$500	ID-ICP/MS ^b	\$30	Resin or direct count, but processing rate is 1 sample/1000 min	\$50	Labor and materials, but requires 2 days/batch turnaround time
ISCO/3M Empore™ SPE disk	\$25/disk ^c	Material cost only, no labor costs involved	\$30/disk ^c	Processing rate is up to 10 disks/100 min	\$35/disk ^c	2 hrs/batch turnaround time

^aMaterial cost only.

^bIsotope dilution, inductively coupled plasma/mass spectrometry.

^cPrice varies with quantity purchased; prices shown reflect actual cost to SRS during this study.

