

Application of Empore™ Disk Technology to Environmental Radiochemical Analysis*

by

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The costs associated with environmental restoration and waste management at both government and private facilities are burdensome, and continue to grow. The Department of Energy estimates that over one million samples, many containing radioactive components, will be analyzed per annum to support remediation programs at its 4000 sites. The development and implementation of new analytical technologies can significantly reduce the high costs associated with these programs.

Disk solid-phase extraction technology has been proven to be highly effective for sample preparation in the analysis of organic compounds, waste waters, and other aqueous samples. Disk technology significantly improves sample throughput, while reducing secondary waste and costs. Moreover, many of the hazardous chemicals associated with traditional procedures are eliminated. This technology may be readily automated and lends itself to field applications. Through a Cooperative Research and Development Agreement, the 3M Company and Argonne National Laboratory are expanding this technology to address sample preparation and recovery of radionuclides from aqueous samples, i.e., surface, ground, and drinking waters. Disks have been developed which demonstrate high selectivity and great affinity for important radionuclides, including ^{99}Tc , $^{89/90}\text{Sr}$, and $^{226/228}\text{Ra}$. Tests results are summarized below.

The application of Empore™ disk technology to environmental radioanalytical chemistry dramatically simplifies sample preparation. Sample processing and counting-source preparation procedures may be condensed into a single step, thereby significantly improving laboratory efficiency. The preparation takes approximately 20 min of effort for a 1-liter sample and is amenable to batch processing. Samples are simply acidified and prefiltered, as appropriate, processed through an equilibrated disk under vacuum, and counted. The disks can be counted directly in a gas-flow proportional counter or in a liquid scintillation counter.

Technetium

Empore™ Anion Exchange-SR extraction disks are used to selectively extract the pertechnetate ion (TcO_4^-) from aqueous solutions. Quantitative recoveries of technetium have been observed for sample volumes up to 8 L on a 47-mm disk. Except for nitrate and iodide ions, no significant interferences have been identified. The technetium uptake efficiency remains high (>98%) from aqueous samples having aggregate anion contents in excess of 30,000 $\mu\text{g/mL}$. For example, in water from Mono Lake, CA (with an anion content of about 40,000 $\mu\text{g/mL}$), added technetium is quantitatively removed by a single membrane (47-mm disk, 1-L volume, 50 mL/min flow rate). The uptake of technetium onto the membrane remains above 98% in the presence of up to 2000 $\mu\text{g/mL}$ nitrate and 1000 $\mu\text{g/mL}$ hypochlorite. Iodide ion at 1000 $\mu\text{g/mL}$ reduces the technetium uptake to 70%. Results from the analyses of a variety of laboratory-spiked samples for ^{99}Tc exhibit good accuracy and precision with no indication of bias, as shown in Table 1.

Strontium

Empore™ Strontium Rad Disks are used to isolate strontium from samples acidified to 2 M HNO_3 . The disk is subsequently assayed for beta activity, or alternatively, ^{90}Y can be stripped and counted after sufficient ingrowth. Strontium retention is greater than 97% for 1-L samples that contain less than 3 mg total strontium and that do not contain large amounts of interfering cations. Recoveries are greater than 85% when the volume is increased to 3 L. At levels of 10,000 ppm, ammonium, potassium, calcium, and lead significantly degrade the uptake of strontium by the disk. However, at levels typical of environmental samples, these interferences are minimal. Also, Ba, Ra, and Pb are retained by the disk. Radioactive isotopes and/or daughter products of these three elements pose radiometric interferences when the disks are counted directly. Although lead may be retained by the disk, lead compounds are very insoluble, and therefore, potential problems from ^{210}Pb should be insignificant. If appropriate, an Empore™ Radium Rad Disk can be used prior to the strontium disks to remove the radium interference, or alternatively, the ^{90}Y aliquot can be purified to determine ^{90}Sr activity. Chemical recovery may be determined by instrumental analysis of pre- and post-disk stable strontium or through the use of a ^{85}Sr tracer in a matrix duplicate. The method detection limit depends on the sample volume and counting protocol but generally will be less than 1 pCi/L. Results from the analyses of performance evaluation samples and laboratory-spiked samples for $^{89,90}\text{Sr}$ indicate excellent disk performance, as demonstrated in Table 1.

Radium

Empore™ Radium Rad Disks are currently under evaluation, and an appropriate chemical separation procedure is under development. These disks exhibit a large capacity for radium; over 2000 pCi of ^{226}Ra have been loaded onto a 25-mm disk. Quantitative recoveries of radium have been observed for sample volumes up to 3 L for a 47-mm disk. However, applicable sample volumes are expected to exceed 3 L; therefore, future experiments will determine the breakthrough volume. Additionally, these disks appear to function well at a wide range of pH. No effect was observed in the performance of these disks when the matrix acid concentration was varied from deionized water through 6 M HNO_3 . Initial experiments indicate that high levels of potassium, ammonium, and sodium interfere with the uptake of radium. However, similar to the behavior exhibited by the Strontium Rad Disks, these interferences are not a problem at levels typical of environmental samples. Barium is retained by the radium disks. Results from a small set of laboratory-spiked natural samples for ^{226}Ra indicate that the Empore™ Radium Rad Disks exhibit excellent performance (see Table 1).

Table 1. Measurements by Empore™ Rad Disks

Sample	Volume, mL	Natural Samples	
		Activity, Bq/L (measured)	Activity, Bq/L (spiked)
Well water ^a + ⁸⁹ Sr	400	13.9 ± 0.3	14.3 ± 0.3
Tap water ^a + ⁸⁹ Sr	1000	14.7 ± 0.3	14.3 ± 0.3
Mississippi Riv ^b . + ⁹⁰ Sr	500	0.37 ± 0.01	0.40 ± 0.02
Runoff water ^c + ⁹⁰ Sr	500	0.41 ± 0.02	0.40 ± 0.02
Well water ^a + ⁹⁹ Tc	8000	12.4 ± 0.6	12.8 ± 0.4
Deionized water ^a + ⁹⁹ Tc	1000	19.7 ± 1.1	20.3 ± 1.1
Mono Lake water ^d + ⁹⁹ Tc	1000	26.8 ± 0.8	26.8 ± 0.6
Well water ^a + ²²⁶ Ra	1000	10.4 ± 0.2	10.9 ± 0.3
Tap water ^a + ²²⁶ Ra	500	10.8 ± 0.3	10.9 ± 0.3
Mono Lake water ^d + ²²⁶ Ra (pH = 9)	250	9.6 ± 0.4	10.9 ± 0.3
Sample	Volume, mL	Performance Evaluation Samples	
		⁹⁰ Sr Activity, Bq/L (measured)	⁹⁰ Sr Activity, Bq/L (reported) ^g
EMSL/LV water ^e	400	0.50 ± 0.02	0.56 ± 0.19
	400	0.42 ± 0.03	0.56 ± 0.19
EML water ^f	25	69.3 ± 1.2	68.6 ± 3.3
	25	71.2 ± 1.2	68.6 ± 3.3

^a Argonne National Laboratory, Argonne, IL
^b Mississippi River near Le Claire, IA
^c Gravel pit near Plainfield, IL
^d Mono Lake, CA
^e EPA program: EMSL/LV = Environmental Monitoring Systems Laboratory - Las Vegas
^f DOE program: EML = Environmental Measurements Laboratory
^g Concentration values reported by the respective performance evaluation program