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# CAPTURE AND RETRIEVAL OF PLUTONIUM OXIDE PARTICLES AT ULTRA-LOW CONCENTRATIONS USING HIGH-GRADIENT MAGNETIC SEPARATION

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# CAPTURE AND RETRIEVAL OF PLUTONIUM OXIDE PARTICLES AT ULTRA-LOW CONCENTRATIONS USING HIGH-GRADIENT MAGNETIC SEPARATION

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## ABSTRACT

A high-gradient magnetic separation system has been developed for capture and retrieval of ultra-low plutonium oxide concentrations. The application of advanced matrix materials and improved methodology has demonstrated the effective collection and recovery of submicron paramagnetic actinide particles with particle concentrations as low as $10^{-23}\,M$. Incorporation of multiple passes during recovery of magnetically captured particles improves the system mass balance. Activity balances for plutonium

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were verified with stringent sampling protocols. Collection and recovery values demonstrate that 99% of the submicron plutonium oxide particles can be accounted for when recycle loops are incorporated into capture and recovery circuits, magnetically captured particles are released by sonication, carrier fluids are organically based, and longer matrix lengths are utilized.

*Key Words*: Plutonium oxide; Actinides; High gradient magnetic separation; Dendrites; Paramagnetic; Steel wool; Mass balance; Submicron

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## INTRODUCTION

Magnetic separation has been developed and utilized in a wide variety of applications.[1–8] Selective recovery of actinides has been realized in wastewater and soil remediation, high-level waste treatment, and nuclear material recovery.[9–15] Currently, the Department of Energy (DOE, NN-20, Office of Nonproliferation and National Security) is developing separation technologies for the nondestructive capture and retrieval of actinide particles from samples of interest.[16]

Trace quantities of lanthanides and actinides emitted from nuclear processing facilities can hydrolyze under environmental conditions to yield colloidal or submicron hydrous oxide particles.[17] Colloidal material will remain suspended in water bodies due to the near neutral pH of the water and small particle size of the hydrolyzed material. Particulates of interest can also be retained on debris, clothing, or soils. High-gradient magnetic separation (HGMS) methods can nondestructively extract small paramagnetic particles from these systems for subsequent analysis.

HGMS of aqueous submicron *ferromagnetic* particles has been proven in previous experimental and theoretical works.[18–20] The extension of HGMS for submicron *paramagnetic* particles has shown diminishing capture efficiencies when targeted particle diameters are less than 0.1 $\mu$m. Additionally, HGMS modeling efforts have varied in the determination of a minimum particle diameter required for paramagnetic particle capture. Recently, calculations have indicated that particles less than 0.04 $\mu$m diameter appear to require extraordinarily high magnetic fields or particle magnetic susceptibility for retention in any HGMS process.[21] A magnetic field enhanced reversible "swing adsorption" process has been shown to improve colloidal plutonium and americium sorption on magnetite-coated polyamine–epichlorohydrin resin beads.[22] To date, there has been no reported experimental data on the paramagnetic capture of submicron actinide particles with high accountability of the material. The development of

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HGMS for the efficient collection and concentration of submicron paramagnetic particles is presented here.

## EXPERIMENTAL

A low-temperature superconducting magnet with a 3-in. warm bore and 8 T maximum field strength (Cryomagnetics Inc., Oak Ridge, TN, USA) was used for these experiments. The baseline matrix material, extruded stainless steel wool (ESSW), was obtained from MEMTEC Inc. (Deland, FL, USA) in sintered and unsintered configurations. In addition, acid etched and nickel dendrite-coated ESSW were also used and the preparation is described elsewhere.[15,16] Scanning electron micrographs (SEMs) in Fig. 1 show the differences in surface topography and geometry of the magnetically energizable matrix elements used in our studies. The figure shows images for baseline ESSW, etched ESSW, and nickel dendrite-coated ESSW materials. Matrix inserts (3/4 in. I.D., 15 or 0.76 cm

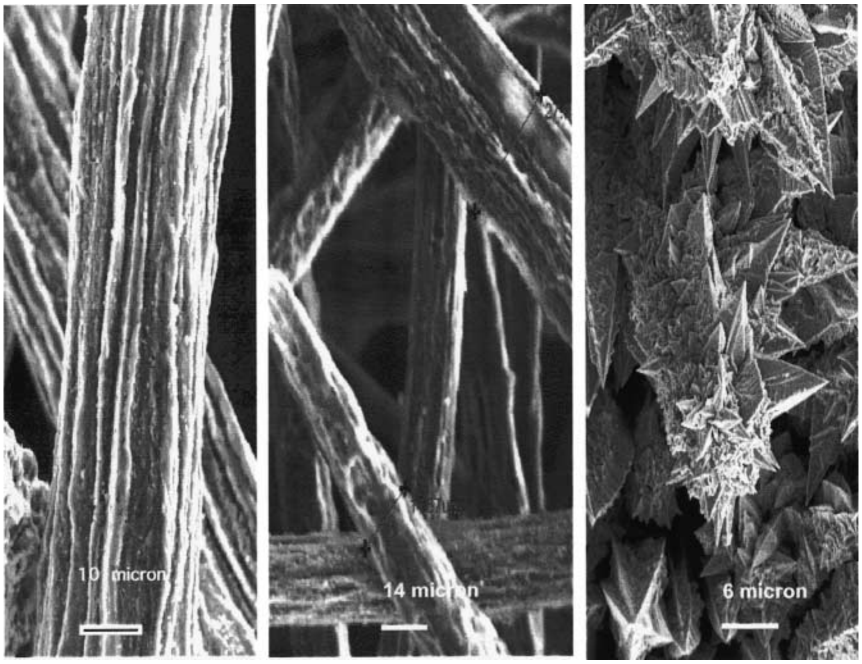

*Figure 1.* Advanced HGMS matrix materials are shown in the micrographs. From left to right the materials are as follows: ESSW, acid-etched ESSW, and nickel dendrite grown on ESSW.

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lengths) were packed with matrix material at 0.90 void fraction. Plutonium oxide ($PuO_2$) was obtained from ongoing work at Los Alamos National Laboratory. Specific size distributions of $PuO_2$ were obtained by sedimentation and decantation procedures relying upon the relationship between settling velocity and particle diameter described in Stokes Law.[23,24] Particle size distributions were verified by dynamic light scattering analysis using a Horiba LA 900 (Irvine, CA, USA). Membrane filters (0.2 $\mu$m) were obtained from Whatman Inc. (Clifton, NJ, USA) and used for secondary recovery of the magnetically captured plutonium oxide particles. Filters were submitted for radioanalysis on a Berthold Instrument (Oak Ridge, TN, USA). Liquid samples were submitted for liquid scintillation analysis (LSC) on a Packard instrument (Meriden, CT, USA) with Optifluor-O as the scintillation cocktail.

Plutonium oxide particles, 0.2–0.8 $\mu$m diameter, were suspended in a 1:1 (vol:vol) mixture of *n*-dodecane and 2-propanol. Aqueous solutions were avoided to eliminate errors from plutonium oxide dissolution and hydrolysis. Plutonium concentrations ranged from 0.40 to 10 ppb and $PuO_2$ particle concentrations ranged from $10^{-18}$ to $10^{-23}$ *M*.

A schematic of our experimental array is shown in Fig. 2. Typically, 85 mL plutonium oxide particle suspensions were pumped through the magnetized matrix at 0.5 cm/sec superficial velocity and 2 T applied field. The HGMS capture and secondary recovery circuits could be operated in a single or multiple pass (recycle loop) configuration. Secondary particle recovery from the matrix was

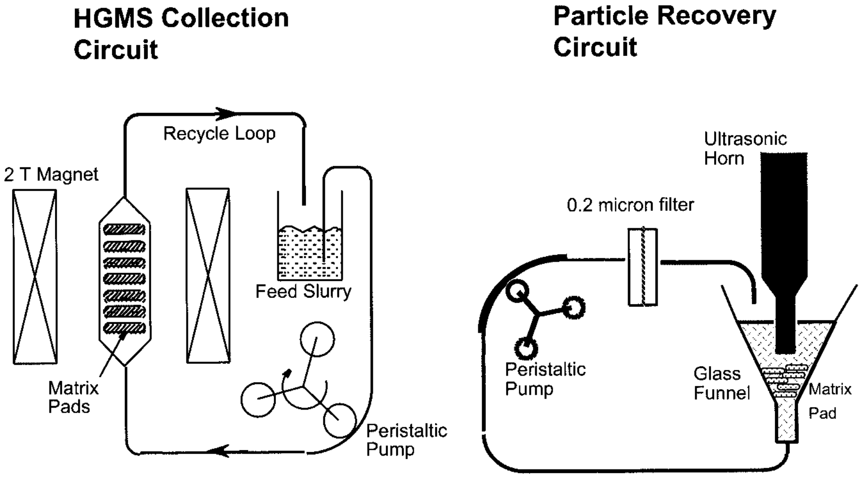

***Figure 2.*** HGMS system schematic shows the magnetic particle collection on the HGMS matrix pads and secondary particle recovery in a separate filtration loop.

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accomplished by either: gas sparging with reverse fluid flow direction at high superficial velocities and zero applied field with the matrix material remaining in the column, or removal of matrix material from the column, sonic liberation, and recovery of particles by pumping recovery fluid through a membrane filter.

Particle capture effectiveness and system mass balance were determined using the masses of the plutonium determined by the total activity from the various liquid samples and from the filter sample. System performance was evaluated using a combination of capture effectiveness and system mass balance.

## RESULTS AND DISCUSSION

The efficient HGMS capture and subsequent matrix release of plutonium oxide particles from ultra-dilute suspensions is challenging. Two areas where HGMS advances have been accomplished in this work include the successful submicron *paramagnetic* particle collection from an extremely dilute influent, and an accountability of all the particles in the recovery processes. In our experiments, a typical feed solution containing 0.8 $\mu$m diameter $PuO_2$ particles at 400 ppt plutonium would contain only 65 particles/mL available for capture. To capture successfully all available actinide particles without alteration of the particle morphology, the use of a nonaqueous carrier fluid is required to prevent solubilization. Additionally, careful experimental protocol to eliminate sample contamination and cross-contamination is demanded.

Typical HGMS systems are designed to capture and remove magnetically susceptible species from a variety of substrates where the quality of the effluent stream is important. In our application, it is critical to not only capture the particles from the carrier fluid, but also to subsequently recover and isolate the particles from the matrix material for particle analysis. Thus, an experimental design, which integrates particle mass balance closure, is a significant addition to a traditional HGMS approach.

### Capture Effectiveness

In HGMS, there are several variables affecting the separation performance.[1] The variables include both material characteristics (particle size, particle concentrations, magnetic susceptibility, carrier fluid pH, and viscosity) and separator parameters (matrix element size and spacing, applied magnetic field strength, residence time, and superficial velocity). For the application described here, the particle size, matrix fiber diameter, and particle residence time of the targeted species are the important variables.[15]

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Our HGMS capture effectiveness, $\varepsilon$, is calculated using total activity values from feed and effluent samples as:

$$\varepsilon = ([\mathrm{PuO_2}]_{\mathrm{feed}} - [\mathrm{PuO_2}]_{\mathrm{effluent}})/[\mathrm{PuO_2}]_{\mathrm{feed}} \tag{1}$$

Results are plotted logarithmically in Fig. 3 as $1 - \varepsilon$ vs. chronological run series, in order to resolve values of $\varepsilon > 99\%$. As such, values of $1 - \varepsilon$ approaching 0.001 would indicate a particle capture effectiveness of $>99.9\%$ and, conversely, values of $1 - \varepsilon = 1.0$ would indicate zero (0) particle capture.

From the results, the particle capture improves as matrix lengths increase. Referring to Fig. 3, run series 1240–1257 were conducted using a 15 cm matrix length, whereas run series 1258–1277 were conducted with a matrix length of 0.76 cm. Limitations in obtaining nickel dendrite-coated matrix material forced the use of a matrix length of 0.76 cm. In order to maintain experimental consistency, many of the HGMS experiments applied a shorter matrix length regardless of the matrix material utilized.

The variability in capture effectiveness, as shown in Fig. 3, when using a 0.76 cm matrix length was reduced when the procedure was modified to include recycling of the feed solution through the magnetized matrix. The poorest capture effectiveness when using a single pass through the matrix and a 0.76 cm matrix length was 82%. Conversely, when using multiple passes through the 0.76 cm

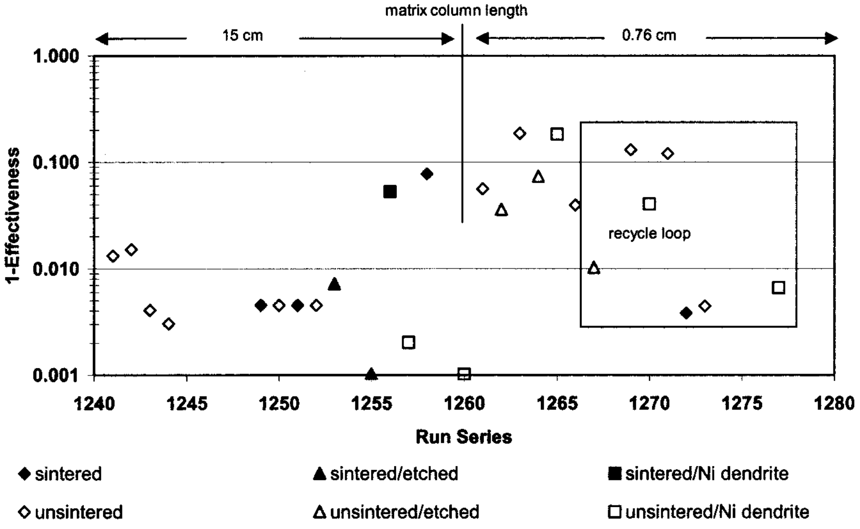

*Figure 3.* The evolution of the HGMS results is shown. The longer matrix lengths and/or increased residence time due to recycle loops improve capture effectiveness.

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matrix, the poorest capture effectiveness was increased to 88%. Generally, modified matrix materials (nickel dendrite or acid etched) performed better, based on capture effectiveness, than unmodified matrix materials. Particle capture effectiveness exceeded 90% and often was greater than 99% using the modified materials. For example, run 1260 had initial Pu concentrations of 10 ppb and effluent Pu concentration of 30 ppt, which corresponds to >99.9% capture effectiveness.

As apparent from Fig. 3, the longer matrix lengths and no fluid recycle reduce the variability and improve the magnitude of particle capture. Particle capture, when using 15 cm matrix lengths, is generally >99% $(1 - \varepsilon < 0.1)$. For example, run 1255 using sintered matrix material subjected to controlled corrosion achieved a particle capture effectiveness > 99.9%. To achieve similar particle capture values when using shorter matrix lengths, significant procedural changes were incorporated such as multiple passes of the feed solution through the magnetized matrix effectively increasing matrix length and particle residence time in the separator.

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## System Mass Balance

Most commercial applications of HGMS systems are not seriously affected by mass balance discrepancies. However, at ppt particle concentrations and with particle recovery as the primary objective, mass balance discrepancies must be kept at a minimum. Our sampling methodology allowed a stringent examination of potential areas of material losses. Ultimately, a high recovery of targeted particles proved to be as challenging as efficient particle capture.

Our HGMS system mass balance error ($\mu$) was calculated using total activity values from all liquid samples and the recovery filter [Eq. (2)]. The numerator is actually the mass balance.

$$\begin{aligned}\mu = ([\mathrm{PuO_2}]_{\mathrm{feed}} + [\mathrm{PuO_2}]_{\mathrm{filter}} + [\mathrm{PuO_2}]_{\mathrm{effluent}} + [\mathrm{PuO_2}]_{\mathrm{flush}} \\ + [\mathrm{PuO_2}]_{\mathrm{rinse}} - [\mathrm{PuO_2}]_{\mathrm{feed}})/[\mathrm{PuO_2}]_{\mathrm{feed}} \qquad (2)\end{aligned}$$

Figure 4 shows mass balance error, expressed as percent, for a series of HGMS experiments. Zero percent (0%) indicates total material accountability while negative values show activity loss and positive values indicate activity gain.

Initial efforts at recovering particles from the matrix involved a traditional backflush. Using clean carrier fluid, high superficial velocities, reverse flow, and introduction of gas bubbles captured particles were sheared off of the matrix fibers and collected for analysis. Substantial variation in particle recovery was experienced with mass balance errors greater than 20% (±) being common.

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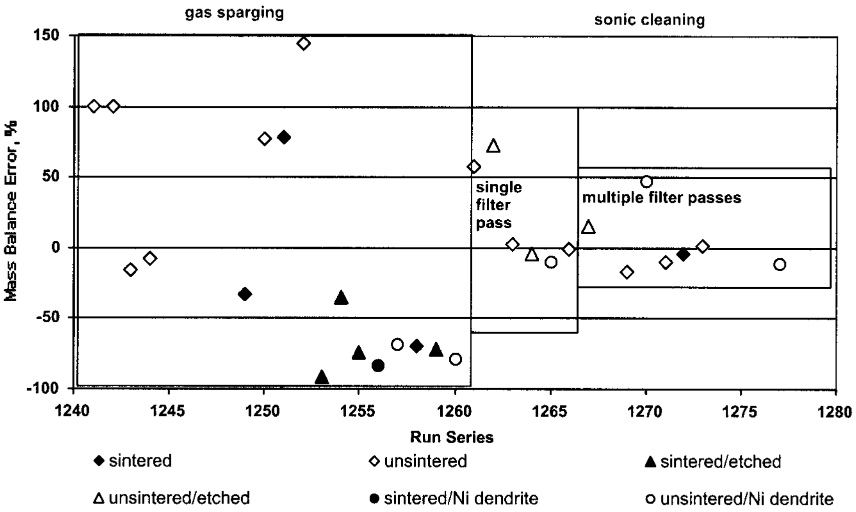

*Figure 4.* The figure shows that in the secondary recovery cycle, sonic cleaning, and multiple membrane filter passes improve mass balance.

These results were unsatisfactory and the secondary particle recovery method was reassessed.

Sonication, to liberate the magnetically captured particles from the densely packed matrix material, was integrated into the experimental procedure as an alternative to gas sparging. The matrix material was removed from the column, placed in a graduated funnel containing clean carrier fluid and into which a sonic probe could be lowered and energized. During sonication, carrier fluid was pumped through the membrane filter to achieve particle recovery. This is essentially an ex situ matrix cleaning procedure and is shown in Fig. 2.

Initial results from the ex situ procedure with a single membrane filter pass of the carrier fluid were more effective for secondary particle recovery than gas sparging. Incorporating multiple passes of the recovery fluid through the membrane filter improved mass balance errors further such that material losses generally did not exceed 15%.

## Integrated System Performance

There are two elements of importance in the actinide particle recovery application. The first is particle capture expressed as capture effectiveness, $\varepsilon$, and the second is particle recovery expressed as system mass balance error, $\mu$, as

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discussed previously. The capture effectiveness should be as close to 1 as possible and the system mass balance error should be as close to 0 as possible. We define an integrated system performance parameter, $\beta$, that combines both terms as follows:

$$\beta = \varepsilon \times \mathrm{abs}(1 - \mu) \tag{3}$$

where $\varepsilon$ and $\mu$ are expressed as fractions. This linear relationship gives equal weight to both parameters and provides a convenient method for comparing the total system performance. A zero (0) mass balance error and total particle capture would correspond to a $\beta$ of 1.0 and would indicate that all particles available were captured and recovered. Figure 5 shows $\beta$ for all experiments. The trend observed is that each procedural modification, in either particle capture or recovery, incorporated during the project lifecycle had a positive impact on the system performance. The positive impact is reflected in an increasing $\beta$.

For example, run 1273 in Fig. 5 has a reported $\beta$ approaching 1. The evolved experimental protocol for 1273 included multiple passes of the feed solution through a short matrix (0.76 cm), and multiple passes in the secondary recovery circuit. Run 1273 had an initial plutonium concentration of 1.83 ppb, and following the HGMS collection, the effluent plutonium concentrations were 0.01 ppb (our detection limits). All the particles that were extracted by HGMS were subsequently concentrated on the membrane filter generating an overall mass balance error of 0.6%. As such, >99% of the available targeted particles

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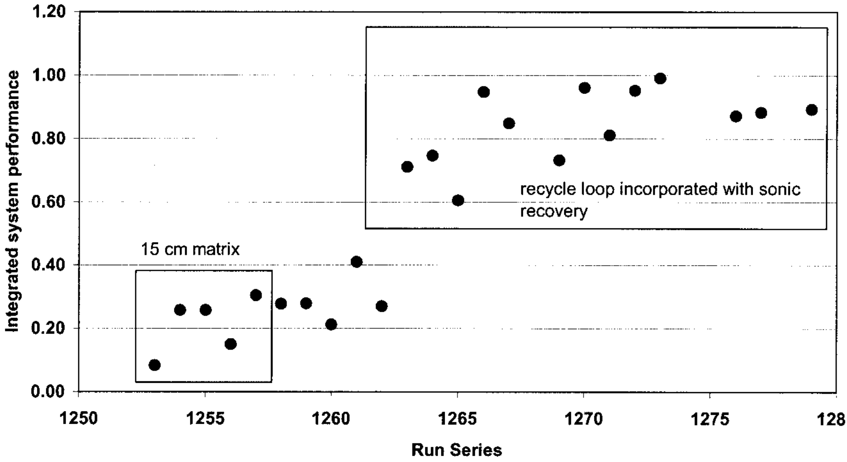

*Figure 5.* HGMS integrated system performance, defined as $\beta = \varepsilon \times \mathrm{abs}(1 - \mu)$, improves with procedural modifications.

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were captured, recovered, and available for return to the client for subsequent examinations.

## CONCLUSIONS

The application of advanced matrix materials and improved experimental methodology in HGMS has shown that $>99\%$ of targeted paramagnetic submicron actinide particles at ultra-low particle concentrations can be captured and recovered. Longer matrix lengths and/or increased residence time within the magnetized matrix due to recycle loops improve particle capture. Incorporating multiple passes of the recovery fluid substantially decreases material loss and improves system mass balance.

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