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Solid-phase extraction microfluidic devices for matrix removal in trace element assay of actinide materials

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ABSTRACT

Advances in sample nebulization and injection technology have significantly reduced the volume of solution required for trace impurity analysis in plutonium and uranium materials. Correspondingly, we have designed and tested a novel chip-based microfluidic platform, containing a 100-μL or 20-μL solid-phase microextraction column, packed by centrifugation, which supports nuclear material mass and solution volume reductions of 90% or more compared to standard methods. Quantitative recovery of 28 trace elements in uranium was demonstrated using a UTEVA chromatographic resin column, and trace element recovery from thorium (a surrogate for plutonium) was similarly demonstrated using anion exchange resin AG MP-1. Of nine materials tested, compatibility of polyvinyl chloride (PVC), polypropylene (PP), and polytetrafluoroethylene (PTFE) chips with the strong nitric acid media was highest. The microcolumns can be incorporated into a variety of devices and systems, and can be loaded with other solid-phase resins for trace element assay in high-purity metals.

1. Introduction

Quantification of trace elemental impurities in uranium (U) and plutonium (Pu) is typically required for quality control in nuclear energy and military applications of these materials [1–4]. Trace elements can also serve as signatures of material provenance in nuclear forensic analysis [5,6]. Optical spectrometric analysis methods for multiple trace elements in actinides rely on dissolution in strong mineral acids, followed by selective removal of the strongly interfering U and/or Pu matrix by anion exchange or extraction chromatography. The column effluent from this separation can be analyzed by inductively coupled plasma - optical emission spectroscopy (ICP-OES) or atomic absorption spectroscopy (AAS). A complementary analysis by inductively coupled plasma - mass spectrometry (ICP-MS) may also be performed on an intact sample, but is insufficient by itself to determine all impurities of importance to nuclear applications.

Common methods for actinide matrix removal use chromatographic resin beds that are several milliliters in volume. The column size is driven by the tens of mL of analyte solution that were traditionally required for continuous signal integration during the ICP-OES analysis. For example, Method C1647 (ASTM International) passes 250 mg of uranium and/or plutonium in nitric acid solution through a column containing 10 mL of diamyl amylphosphonate extraction chromatography resin, to generate 30 mL of analyte solution [7]. A similar large-scale method used in our laboratory requires 4 mL of strong-base anion exchange resin to process 250 mg of Pu, yielding 40 mL of trace element solution for ICP-OES analysis. These separation methods make it possible to measure > 20 trace metal contaminants at levels as low as 2 μg g-1 in actinide materials, but they generate significant quantities of radioactive liquid and solid wastes. The sample size requirement also limits use of the methods to facilities with appropriate safeguards and infrastructure for storing and handling larger quantities of nuclear materials.

With the advent of low-flow nebulization and loop injection techniques for plasma spectroscopy, it is now possible to perform comprehensive, high-sensitivity actinide impurity assay by ICP-OES using just 1 mL of column effluent [8]. Although microfluidic technology for actinide separation is still in its infancy [9–12], integrated microfluidic devices customized with solid-phase resins hold significant potential as lab-on-a-chip platforms for matrix removal in trace element assay and for related analyses of actinides. An effective microfluidic separation device would support a 10- to 100-fold reduction in sample size, with commensurate operational benefits to worker safety and waste minimization. Unlike typical microfluidic devices that perform under near-neutral pH conditions for biological and environmental applications, these devices for nuclear material separations must be chemically compatible with strong mineral acids, and must also contribute negligibly to the trace element analytical blank.

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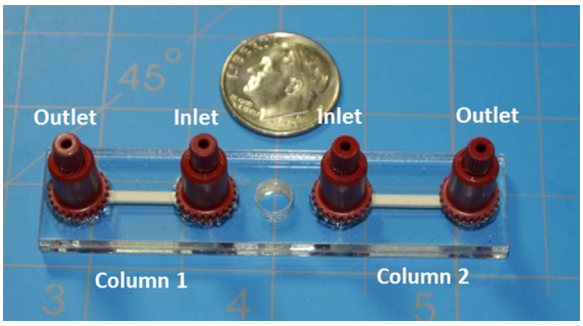

Fig. 1. Assembled 20-µL solid-phase extraction microfluidic device, filled with AG MP-1 resin (column 1) and UTEVA resin (column 2). Scale is illustrated using a US dime coin (17.9 mm diameter).

In the present study, we designed 100-µL and 20-µL solid-phase separation columns in microfluidic devices constructed of thermoplastic polyvinyl chloride (PVC, Fig. 1), and demonstrated that the microcolumns, packed by centrifugation with ion-specific resins, provide efficient actinide matrix removal from trace element impurities. Commercially-available anion exchange and chromatographic resins retained the surrogate nuclear material matrices (thorium for plutonium, and natural for enriched uranium) from strong nitric acid solutions, while trace impurities passed through quantitatively for analysis by ICP-OES. Actinides were subsequently recovered using dilute hydrochloric acid solution.

This flexible, miniaturized platform can be incorporated into more complex devices to provide complete lab-on-a-chip systems. With suitable choice of resin and reagents, the microcolumn design can be adapted for solid-phase extraction supporting trace element assay in various other sample types, such as high purity metals and meteorites [13–16].

2. Material and methods

2.1. Materials and reagents

Thermoplastic materials were purchased from McMaster (Santa Fe Springs, CA, US). Sylgard 184 polydimethylsiloxane (PDMS) was purchased from Fisher Scientific. Frits were purchased from VICI Jour (Schenkon, CH). Analytical grade macroporous anion exchange resin (AG MP-1, 200–400 mesh) was procured from BioRad (Hercules, CA, US). UTEVA resin (100–160 µm) was obtained from Eichrom (Lisle, IL, US). Nitric and hydrochloric acids were Thermo Fisher Optima grade. Multi-element ICP-OES standard solutions and thorium/uranium single element solution were custom-made by Inorganic Ventures (Christiansburg, VA, US).

2.2. Compatibility testing

Nine materials including acrylic, fluorinated ethylene propylene (FEP), polycarbonate (PC), polydimethylsiloxane (PDMS), polyetheretherketone (PEEK), polypropylene (PP), polyvinyl chloride (PVC), Teflon polytetrafluoroethylene (PTFE), and thermoset polyester (TPE) were examined for compatibility with nitric acid. A coupon of each material (15 mm x 10 mm x 1.5 mm) was submerged completely in 3 mL of 10 M HNO_3. The test coupons were removed from the acid after 24 h, and rinsed with DI water before being placed into a fresh portion of 10 M HNO_3 for an extra 7 days. The leachates from each test were analyzed by ICP-OES for trace element content.

2.3. Device design and fabrication

The microfluidic chip was designed in Solidworks (Waltham, MA, US), and the 3D patterns were imported into VisualCAD to generate G-code for chip fabrication. Each microfluidic device contains two microcolumns arrayed symmetrically around a central 5 mm diameter shaft hole (Fig. 2). Chips were fabricated from two layers of thermoplastic PVC and assembled using 467MP acrylic adhesive transfer tape (3 M, St. Paul, MN, US). The top layer of each chip contains four holes that serve as column inlets and outlets (5 mm diameter for the 100-µL column; 1.57 mm diameter for the 20-µL column). The microcolumns were machined into the bottom layer. For the 100-µL microcolumn device, the bottom layer of PVC was 3 mm thick with two extraction columns of 25 mm x 2.5 mm x 1.6 mm. For the 20-µL microcolumn device, the bottom layer of PVC was 1.5 mm thick with two engraved columns of 17.5 mm x 1.4 mm x 0.8 mm. Each patterned layer was machined using a CNC micro-milling machine (Minitech, Norcross, GA, US) at a spindle speed of 10,000 rpm and a cutting feed rate of 200 mm min-1. Tooling included two 2-flute square end mill bits with diameters of 0.06″ and 0.04″.

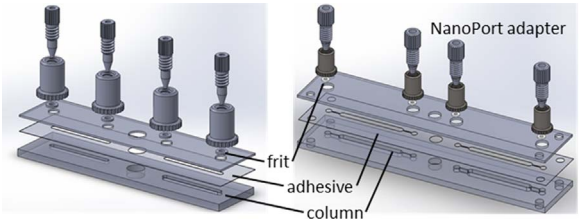

Fig. 2. Schematic design of microfluidic devices, containing two 20-µL (left) or two 100-µL (right) microcolumns. Devices were designed in Solidworks and fabricated by micro-milling.

To laminate the adhesive layer to the bottom chip, the device was passed through a GBC Catena 35 (Northbrook, IL) laminator at 240 °C. A scalpel was used to remove excess tape from the column profile. (Alternatively, the tape was pre-cut using a laser and a non-adhesive protective liner before chip assembly). After removing the adhesive protective liner, the top layer of the device was assembled with its corresponding bottom layer. The assembly was pressed by the laminator two times at 240 °C, followed by 10 min pressing at 400 pounds using a benchtop laboratory manual press (Model 4122, Carver Inc., Wabash, IN, US).

Before packing the columns, 10 µm pore size frits were inserted into the two outlet ports to contain the resin beads. The 5 mm OD frits (PEEK-encased ultra-high molecular weight polyethylene) were used for the 100-µL device, and 3 mm OD frits (PEEK-encased 316 stainless steel) were used for the 20-µL device. The resin was suspended in DI water, and transferred via a syringe into the column inlet. The device was spun on an Eppendorf 5415C centrifuge at 5000 rpm for 5 min, then a second set of frits was inserted into the inlet ports to secure the resin column. Nano-port inlet and outlet adapters (IDEX Health and Science, Rohnert Park, CA, US) were attached on the device to allow for 1/32″ OD PTFE tubing connection. Before each experiment, the column was conditioned by introducing 10 M HNO3 for a minimum of 3 or 4 min at the specified flow rate (Table 1) prior to sample introduction.

2.4. Trace element separation procedure

The system setup is depicted in Fig. 3. Sample is injected into a known volume sample loop using a syringe manually. By switching the valve from “Load” to “Inject” position, sample is pushed quantitatively onto the column. Two syringe pumps (KD scientific, Holliston, MA, US) were used to control the flow rate and deliver the acids to the microfluidic device. The 10 M and 8 M HNO3 are delivered by one syringe pump for column conditioning and trace element elution, whereas the 0.1 M HCl is delivered by another syringe pump for actinide stripping. In the case of two concentrations of HNO3 are employed, switching from 10 M HNO3 to 8 M HNO3 is performed manually. The elution sequence was controlled by a MX Series II

Table 1
Separation protocol for trace elements in Th and U using solid-phase extraction microfluidic devices.

	100 µL column		20 µL column	
	AG MP-1	UTEVA	AG MP-1	UTEVA
Flow rate, µL min^{-1}	100	100	20	20
Sample loop, µL	200	200	20	20
Conditioning, min				
10 M HNO_3	3	3	4	4
Separation, min				
10 M HNO_3	2	n/a	4	n/a
8 M HNO_3	10	12	8	15
0.1 M HCl	10	16	12	30

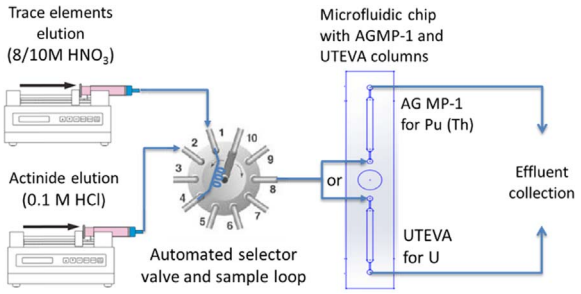

Fig. 3. Schematic diagram of solid-phase extraction microfluidic device in a flow system for the separation of trace elements from actinide matrices.

selector valve (IDEX Health and Science, Rohnert Park, CA, US), following the separation protocol as previous described [17]. A custom Labview program was written to automatically control syringe pumps and selector valve, using the process parameters listed in Table 1.

For the separation of trace elements from a uranium matrix, UTEVA resin was packed into a 100-µL microcolumn. A multi-element standard solution at 125 µg mL-1 in a 250 µg mL-1 uranium matrix was loaded onto the column through a 200-µL sample loop. A subsequent wash with the 8 M HNO3 for 12 min at 100 µL min-1 flow rate elutes the trace elements. The uranium was then stripped from the column using 0.1 M HCl for 16 min at the same flow rate. Fractions were collected every two minutes. For the U separation experiment on a 20-µL UTEVA column, a 20-µL sample loop was used. Trace elements were eluted using 8 M HNO3 at 20 µL min-1 flow rate and collected for 15 min, in two fractions representing the first 5 min and last 10 min of the load cycle. Two more fractions (15 min each) were collected after the eluent was switched to 0.1 M HCl. Each collected fraction was diluted to 4 M HNO3 acidity and a final volume of 1.2 mL for ICP-OES analysis.

For the AG MP-1 column testing, thorium was used as a surrogate for plutonium in this study. A standard solution containing 125 µg mL-1 of each trace element of interest in a 500 µg mL-1 thorium matrix was loaded onto a 100-µL AG MP-1 column through a 200 µL sample loop similarly. Trace elements were first eluted using 10 M HNO3 for 2 min at 100 µL min-1 flow rate, followed by a 10 min wash with 8 M HNO3 at the same flow rate. Finally, thorium was stripped using 0.1 M HCl for 10 min. The fractions were collected at 1 min intervals throughout. For the 20-µL AG-MP1 column study, the multi-element standard was loaded onto the column through a 20-µL sample loop. Eluents of 10 M and 8 M HNO3 were used for trace element recovery, for 4 and 8 min, respectively, at 20 µL min-1 flow rate. One fraction was collected for each of these acid solutions. The third fraction was collected for 12 min using 0.1 M HCl as the eluent. Each collected fraction was diluted to 4 M HNO3 acidity and a final volume of 1.2 mL for ICP-OES analysis. For both the AG MP-1 and the UTEVA columns, a conditioning step was performed after the thorium/uranium elution to rejuvenate the resin for the next samples separation.

2.5. ICP-OES detection

Effluent fractions containing trace elements and matrix solution were analyzed on a Thermo Fisher iCAP 6500 ICP-OES. This instrument has a compact footprint (32.7″ x 29.3″ x 23.2″), allowing it to be operated within a radiological fume hood. The ICP-OES system is equipped with a charge injection device (CID) detector for simultaneous determination of multiple elements. This instrument also has the capability of dual-viewing modes for detection, depending on the sensitivity requirements and matrices involved. In this study, axial viewing mode was employed to obtain the maximum sensitivity, as matrix effects have been minimized by the ion exchange separation. Sample introduction is performed with an Elemental Scientific (ESI, Omaha, NE, USA) microFAST sample introduction system. A total sample uptake of 1 mL was achieved using a low flowrate nebulization technique and an 800-µL sample loop. Detailed operating conditions have been described previously [8].

3. Results and discussion

3.1. Compatibility testing

Matrix separation of plutonium from the trace element impurities on AG MP-1 resin requires acid strengths up to 10 M HNO3, and up to 8 M HNO3 for uranium separations on the UTEVA resin. Materials for fabricating the microfluidic device, therefore, must be compatible with this highly corrosive chemical environment. To evaluate the material compatibility, nine commonly used materials (Table 2) for microfluidic device fabrication were evaluated for their resistance to high concentration HNO3 and for their ability to provide a low analytical background. Coupons of the nine materials were immersed in 10 M HNO3 for 24 h. Visual inspection confirmed that no material had disintegrated after the 24 h acid leaching.

The leachate from each material was diluted and analyzed for a suite of 28 elements of interest. The only elements present at greater than 10 times the ICP-OES method detection limit were calcium, iron and silicon. It is seen in Table 2 that after 24 h acid leaching, calcium was present at trace levels in the leachates of PEEK and PP, whereas iron was measured in trace levels in the leachates of FEP, PDMS, PEEK, PP, Teflon PTFE, and TPE. While only trace levels of silicon were detected in the leachate of PC and PEEK, a large amount of silicon was measured from the leachate of the acrylic coupon. PVC is the only material with no trace elements leached out after 24 h of acid contact.

The coupons recovered from the 24 h acid leaching test were rinsed with DI water and placed in a fresh portion of 10 M HNO3 for another seven days. Again, no indication of physical disintegration was observed at the end of the seven day acid leaching. However, silicon at various concentration levels was detected for all materials, with the highest in PDMS and lowest in Teflon PTFE (Table 2). Iron was measured at trace levels in the PDMS and TPE leachates, and calcium was detected only in the TPE leachate.

Table 2.
Results of material compatibility testing in strong nitric acid solution. ND=not detected.

	Calcium (µg mL^{-1})		**Iron** (µg mL^{-1})		**Silicon** (µg mL^{-1})	
Material	**24 h**	**7 d**	**24 h**	**7 d**	**24 h**	**7 d**
Acrylic	ND	ND	ND	ND	300	400
FEP	ND	ND	0.7	ND	ND	3
PC	ND	ND	ND	ND	0.9	20
PDMS	ND	ND	0.5	0.5	ND	6000
PEEK	0.2	ND	0.4	ND	0.5	5
PP	0.1	ND	0.4	ND	ND	1
PVC	ND	ND	ND	ND	ND	3
Teflon PTFE	ND	ND	0.5	ND	ND	0.5
TPE	ND	2	0.3	1	ND	9

The acid compatibility results showed that all of the materials maintained their physical integrity in the 10 M HNO_3 environment. PVC is the only material that did not contribute to the trace element sample background during the 24 h acid leaching; thus, it can be used for fabricating microfluidic devices for short-term application (i.e., disposable chips). Calcium and iron were present in the FEP, PEEK, PP, and PTFE leachates after the first 24 h leaching, but not in the additional 7-day experiment, indicating that 24 h acid leaching can be used as a means of material cleaning. PP and Teflon PTFE showed the least amount of silicon among all materials tested in the extra 7-day leaching, and no calcium or iron. Hence, PP and PTFE are most suitable for fabricating a microfluidic device for a long-term application.

PVC was selected as the initial material for a disposable device for the short-term extraction application (< 24 h) because of its favourable optical transparency, machinability and mouldability, in addition to its high chemical resistance. The microcolumn fabricated with PVC material facilitates visual confirmation of fluid movement and colour change in the resin bed during the separation process.

3.2. Trace element separation and matrix recovery

For this study, we evaluated microcolumns of two different volumes, 100 µL and 20 µL, which were integrated into microfluidic devices. A variety of chromatographic resin beads can be packed into the microcolumns. In this study, we focused on AG MP-1 anion exchange resin and UTEVA extraction chromatographic resin, to simulate Pu and U separations, respectively. For example, the 20-µL chip in Fig. 1 has been prepared with one column each of AG MP-1 and UTEVA. To evaluate the separation efficiency of the microcolumns, a multi-element standard was used to simulate the trace impurities in actinide matrices. Thorium was used as a model element to mimic the anion exchange behavior of Pu in on the AG MP-1 microcolumns [18]. This enabled evaluation of the microfluidic devices without glovebox protection, while producing less-hazardous waste. For the uranium-selective UTEVA resin, a natural uranium solution was similarly mixed with the multi-element standard.

Flow rate for elution could impact the separation. In our conventional column separation protocol, gravity flow is used for elution [17]. However, a much lower flow rate is used for the microcolumn separation to avoid backpressure build-up. For the 100-µL columns, a constant liquid flow rate of one column volume per minute was maintained, and eluted fractions were collected at 1-min intervals. For the 20-µL columns, the flow rate was also maintained at one column volume per minute; however, the trace element and matrix effluents were collected into just one or two fractions each during the strong HNO_3 and dilute HCl flushing processes, respectively. The detailed operating conditions are reported in Table 1. The volumes of eluent were selected by scaling our existing analytical method down to the size of the microcolumns.

The chromatogram in Fig. 4 illustrates the separation of trace elements from a thorium matrix on the 100-µL AG MP-1 column. The sample was introduced to the microcolumn in strong nitric acid. Thorium was retained on the column as the hexanitrato anion, while the trace elements were eluted from the column after several washes. Then, 0.1 M HCl was employed to strip the thorium from the column. The trace element profiles, measured by ICP-OES, in the raffinate fractions are plotted in aggregate in Fig. 4. The chromatogram reveals that the 28 trace elements are eluted in two distinct groupings with the HNO_3 mobile phase during the first 12 min of eluent flow, while the Th matrix is quantitatively retained on the column. Thorium is recovered from the column when the mobile phase is switched to 0.1 M HCl, in an elution band lasting from 16 to 20 min run time. These observations demonstrated the quantitative separation of trace metals from actinide matrix, and are consistent with the large-scale trace element and Pu separations routinely performed in this laboratory.

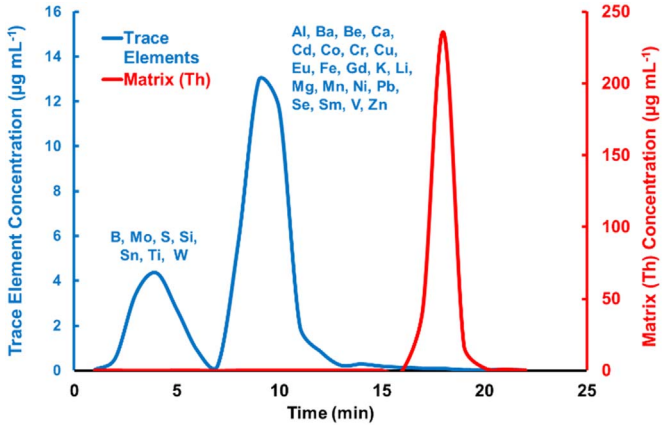

Fig. 4. Trace elements (blue) and Th matrix (red) are separated on an AG MP-1 100-µL ion exchange microcolumn. Trace elements are eluted by 10 M/8 M HNO3 and Th is stripped by 0.1 M HCl. The eluted fractions were analyzed in triplicate by ICP-OES. The blue curve represents the average concentration of the 28 elements. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

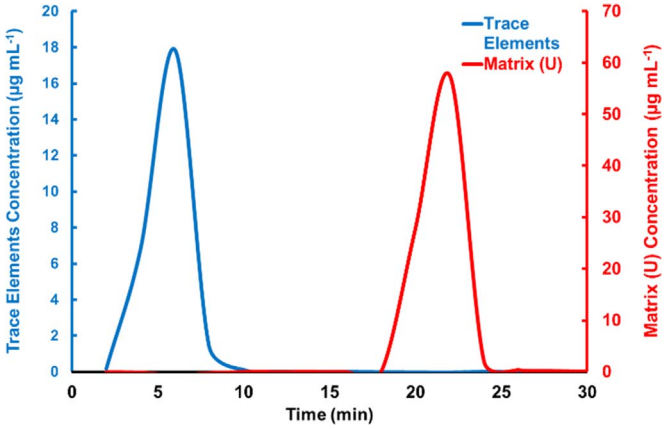

Fig. 5. Trace elements (blue) and U matrix (red) are separated on a UTEVA 100-µL extraction chromatographic microcolumn. Trace elements are eluted by 8 M HNO_3 and U is stripped by 0.1 M HCl. The eluted fractions were analyzed in triplicate by ICP-OES. The blue curve represents the average concentration of the 28 trace elements. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article)

Separation of trace elements from a uranium matrix by the 100-µL UTEVA microcolumn is similarly shown in the chromatogram in Fig. 5. In this instance, all 28 trace elements were recovered in a single band between 2 and 10 min of elution time with 8 M HNO3, while U was retained on the column as the neutral uranyl nitrato complex [19]. Uranium was stripped from the column afterwards with 0.1 M HCl, in an elution band lasting from 18 to 25 min run time.

Overall recovery of each trace element in the strong HNO3 fractions using the 100-µL microcolumn is reported in Table 3, normalized to the recovery of the thorium or uranium. Each entry in the table was obtained by summing the recovery across all fractions from the strong nitric acid washes. Th and U were not detected in the respective strong HNO3 fractions, indicating no matrix breakthrough. Nearly all elements yielded recoveries of 93–103% with the average of 98% and 99% for AG MP-1 and UTEVA column, respectively This result improves upon the acceptance criteria of +/−20% for the large-scale multi-element ICP-OES method currently used in our laboratory [1]. Potassium is the only element yielded recoveries > 120%, which was attributed to its ten times higher concentration in the multielement test standard.

Similar elution protocols were used for the 20-µL microcolumns. The results of the ICP-OES analysis in Table 3 indicate that the elution

Table 3
Percent recovery of trace elements after separation from the Th and U matrix using AG MP-1 and UTEVA resin.

	Recovery of Trace Elements (%)			
	100 µL column		20 µL column	
	AG MP–1	UTEVA	AG MP–1	UTEVA
Al	98	102	106	99
B	94	98	94	92
Ba	99	98	101	102
Be	98	99	104	105
Ca	103	109	126	102
Cd	94	94	97	96
Co	94	94	99	98
Cr	96	98	102	99
Cu	98	97	99	98
Eu	97	100	105	108
Fe	95	100	118	103
Gd	97	100	101	104
K	127	134	108	107
Li	95	94	95	86
Mg	94	97	99	94
Mn	95	98	101	102
Mo	101	93	100	100
Ni	94	95	102	99
Pb	94	94	98	96
S	99	102	102	105
Se	95	96	98	97
Si	96	102	84	99
Sm	100	103	103	107
Sn	103	94	101	101
Ti	97	96	97	98
V	96	97	96	97
W	100	93	96	96
Zn	97	102	105	100

protocol established for the 100-µL microcolumn can be scaled to the smaller 20-µL microcolumn, with no degradation of performance. That is, for both AG MP-1 and UTEVA columns, all of the ICP-OES trace elements were quantitatively eluted in the strong acid fraction, and the actinides were quantitatively recovered in dilute acid. This suggests that the centrifugation method of column packing was effective at preventing non-ideal flow patterns such as channelling and uneven band front travel.

The reduction in sample volume achieved through use of the microfluidic chips is striking. For the 100-µL AG MP-1 microcolumn, all of the trace elements were eluted with nitric acid into a combined volume of 1.2 mL for ICP-OES analysis. Meanwhile, just 1.0 mL of 0.1 M HCl was required to discharge Th from the column. In the conventional analytical approach, the total volume of acid used for trace element elution and actinide discharge was 14 mL and 10 mL, respectively. Thus, the overall solution volume was reduced by 90% using the 100-µL microcolumn. On the 20-µL microcolumns, just 0.24 mL of each effluent was generated for the trace element sample and actinide recovery. Thus, the overall effluent volume was reduced by 98%. In real-world applications, the 100-µL microcolumn can be adopted for its large capacity such as separating trace impurities from plutonium metal and oxides, while the 20-µL microcolumn can be used for samples containing moderate amount of actinides such as nuclear forensic samples.

Our previous work has shown that trace element samples at this volume and concentration can be analyzed to a sensitivity of approximately 2 $\mu g\ g^{-1}$ relative to the original actinide matrix. This makes the new platform useful for low volume actinide separation processes, replacing the traditional workflow. Furthermore, the resin inside the microcolumns can be regenerated after the actinide is stripped from the column, making the chips reusable. This offers future opportunities to integrate the microfluidic separation platform with automated systems.

4. Conclusions

In this study, we demonstrated the fabrication, assembly, and initial operation of nitric acid resistant devices containing solid-phase extraction microcolumns for actinide matrix removal in trace element assays. Separation and recovery of 28 trace elements from thorium and uranium matrices were demonstrated, with 10- to 50-fold sample volume reduction compared to conventional methods that require consumption of large actinide samples. The mass of actinide material loaded onto the columns was correspondingly reduced, from 250 mg in the historical methods, to 5–50 mg on the microcolumns. This sample size reduction has commensurate benefits to worker radiological safety and nuclear material safeguards, potentially reducing the design complexity and cost of the modern analytical chemistry facility. With successful application of the anion exchange microcolumns to thorium separation, work is now underway to validate the use of the devices for trace element analysis in plutonium.

The devices as currently configured can be applied in quality control of nuclear materials, and can be used to preserve the native nuclear and trace element signatures during nuclear forensic analysis. We envision that by changing the ion-specific resins and elution sequences described in this paper, this same platform can be used in other matrix removal applications, such as trace element analyses in high-purity arsenic [13], gallium [14], tungsten [15] and in meteoritic iron [16] samples. Additionally, this flexible microcolumn design can be incorporated into more complex devices for higher throughput or automated operations. We are currently in the process of validating the microfluidic separation method using authentic samples.

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