Automation and Methodology Development for Environmental and Biological Determination of Pu, Np, U and Tc

Qiao, Jixin

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Automation and Methodology Development for Environmental and Biological Determination of Pu, Np, U and Tc

Jixin Qiao

Radioecology and Tracer Studies
DTU Nutech

06-09-2013
### Properties of Pu, Np, U and Tc

<table>
<thead>
<tr>
<th>Nuclide</th>
<th>Isotope</th>
<th>Main origination</th>
<th>Half-life</th>
<th>Main production</th>
<th>Principal decay mode</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Pu</strong></td>
<td>$^{238}\text{Pu}$</td>
<td>Anthropogenic</td>
<td>87.7 y</td>
<td>NA and $\beta$ decay of $^{235}\text{U}$ and $^{238}\text{U}$</td>
<td>$\alpha$</td>
</tr>
<tr>
<td></td>
<td>$^{239}\text{Pu}$</td>
<td>Anthropogenic</td>
<td>$2.4 \times 10^4$ y</td>
<td>Bombardment of $^{238}\text{U}$</td>
<td>$\alpha$</td>
</tr>
<tr>
<td></td>
<td>$^{240}\text{Pu}$</td>
<td>$^{239}\text{Pu} (n, \gamma),^{240}\text{Pu}$</td>
<td>$6.6 \times 10^3$ y</td>
<td>$^{239}\text{Pu} (n, \gamma),^{240}\text{Pu}$</td>
<td>$\alpha$</td>
</tr>
<tr>
<td></td>
<td>$^{241}\text{Pu}$</td>
<td>$^{240}\text{Pu} (n, \gamma),^{241}\text{Pu}$</td>
<td>14.4 y</td>
<td>$^{240}\text{Pu} (n, \gamma),^{241}\text{Pu}$</td>
<td>$\beta^-$</td>
</tr>
<tr>
<td><strong>Np</strong></td>
<td>$^{237}\text{Np}$</td>
<td>Anthropogenic</td>
<td>$2.4 \times 10^6$ y</td>
<td>NA and $\beta$ decay of $^{235}\text{U}$ and $^{238}\text{U}$</td>
<td>$\alpha$</td>
</tr>
<tr>
<td><strong>U</strong></td>
<td>$^{234}\text{U}$</td>
<td>Natural</td>
<td>$2.4 \times 10^6$ y</td>
<td></td>
<td>$\alpha$</td>
</tr>
<tr>
<td></td>
<td>$^{235}\text{U}$</td>
<td>Natural</td>
<td>$2.5 \times 10^5$ y</td>
<td></td>
<td>$\alpha$</td>
</tr>
<tr>
<td></td>
<td>$^{236}\text{U}$</td>
<td>Anthropogenic</td>
<td>$2.3 \times 10^7$ y</td>
<td>$^{235}\text{U}$ neutron activation (NA)</td>
<td>$\alpha$</td>
</tr>
<tr>
<td></td>
<td>$^{238}\text{U}$</td>
<td>Natural</td>
<td>$4.5 \times 10^9$ y</td>
<td></td>
<td>$\alpha$</td>
</tr>
<tr>
<td><strong>Tc</strong></td>
<td>$^{99}\text{Tc}$</td>
<td>Anthropogenic</td>
<td>$2.1 \times 10^5$ y</td>
<td>$^{235}\text{U}$, $^{239}\text{Pu}$ fission product</td>
<td>$\beta^-$</td>
</tr>
</tbody>
</table>
Sources of Pu, Np, U and Tc in the environment

Pu isotopes, $^{237}$Np, $^{99}$Tc, $^{236}$U

Nuclear power plants
Nuclear weapons testing
Nuclear reprocessing plants
Nuclear accidents
Nuclear medicine
<table>
<thead>
<tr>
<th>Source term</th>
<th>$^{238}\text{Pu}$, Bq</th>
<th>$^{239}\text{Pu}$, Bq</th>
<th>$^{240}\text{Pu}$, Bq</th>
<th>$^{241}\text{Pu}$, Bq</th>
<th>$^{237}\text{Np}$, Bq</th>
<th>$^{240}\text{Pu}/^{239}\text{Pu}$ atomic ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nuclear weapons testing</td>
<td>$3.3 \times 10^{14}$</td>
<td>$7.4 \times 10^{15}$</td>
<td>$5.2 \times 10^{15}$</td>
<td>$1.7 \times 10^{17}$</td>
<td>$3.9 \times 10^{13}$</td>
<td>$\sim 0.19$</td>
</tr>
<tr>
<td>Burn up of SNAP-9A</td>
<td>$6.3 \times 10^{14}$</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Thule, Greenland, 1968</td>
<td>-</td>
<td>$1 \times 10^{13}$</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Palomares, Spain</td>
<td>-</td>
<td>$5.5 \times 10^{10}$</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Chernobyl, 1986</td>
<td>$3.0 \times 10^{13}$</td>
<td>$2.6 \times 10^{13}$</td>
<td>$3.7 \times 10^{13}$</td>
<td>$5.5 \times 10^{15}$</td>
<td>-</td>
<td>$\sim 0.39$</td>
</tr>
<tr>
<td>Sellafield reprocessing plant</td>
<td>$1.2 \times 10^{14}$</td>
<td>$6.1 \times 10^{14}$</td>
<td>$2.2 \times 10^{16}$</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>La Hague reprocessing plant</td>
<td>$2.7 \times 10^{12}$</td>
<td>$3.4 \times 10^{12}$</td>
<td>$1.2 \times 10^{14}$</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
Sources of Pu, Np, U and Tc in the environment

**Sources of $^{99}$Tc and $^{236}$U in the environment**

<table>
<thead>
<tr>
<th>Source term</th>
<th>$^{99}$Tc released, Bq</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sellafield nuclear reprocessing plant</td>
<td>$1.72 \times 10^{15}$</td>
</tr>
<tr>
<td>La Hague nuclear reprocessing plant</td>
<td>$1.54 \times 10^{14}$</td>
</tr>
<tr>
<td>Global weapons fallout (1940s-1970s)</td>
<td>$1.40 \times 10^{14}$</td>
</tr>
<tr>
<td>Nuclear accident in Chernobyl</td>
<td>$7.5 \times 10^{11}$</td>
</tr>
<tr>
<td>Estimated nuclear accident in Fukushima</td>
<td>$&gt;2.5 \times 10^{11}$</td>
</tr>
<tr>
<td>Estimated medical application ($^{99}$Mo-$^{99m}$Tc generator)</td>
<td>$&lt;2 \times 10^{10}$</td>
</tr>
<tr>
<td>Estimated nuclear power plants</td>
<td>$&lt;1 \times 10^{10}$</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Source term</th>
<th>$^{236}$U released, Bq</th>
</tr>
</thead>
<tbody>
<tr>
<td>Natural</td>
<td>$8.4 \times 10^{10}$</td>
</tr>
<tr>
<td>Anthropogenic</td>
<td>$2.4 \times 10^{15}$</td>
</tr>
</tbody>
</table>
Significances of Pu, Np, U and Tc determination

1) Environmental risk assessment and monitoring

2) Nuclear emergency preparedness

3) Routine occupational health monitoring

4) Nuclear Forensics

5) Nuclear decommissioning and waste disposal

6) Radioecology and tracer studies
1) Levels are very low and vary with location or sample type
2) Often coexist with matrix elements (Ca, Mg, Al, V, Ru, Mo…) and other interfering radionuclides (Th, Am, Cm…)
Traditional analytical methods for Pu and Np

Pre-concentration:
- 8-16 batchwise
- Fairly straightforward

Chemical purification:
- Lengthy
- High labor intensity
- High consumption of resin
- Organic waste

Detection:
- Long counting time
- High detection limit

Leachates

Dry, ashing
Ash
Centrifugation
Co-precipitation
Valence adjustment
Anion exchange chromatography + solvent extraction

Matrix/U
Th
Pu/Np

Electro-deposition
Alpha spectrometry

1 day
1-2 day
1-10 day
Our objectives

1. Rapid determination of Pu, Np, U and Tc
2. Automation of the analytical procedure

Specific focuses:

i. Chemical purification
   - Protocol simplification and optimization
   - Automation

ii. Detection
   - Mass spectrometry (ICP-MS, AMS)
Automation techniques

- Vacuum box
- HPLC
- Flow injection/Sequential injection

Final strategies

Flow/Sequential injection

Carrier

SP

HC

R1

R2

SV

E

S

W

Eluates

Extraction or anion exchange chromatography

ICP-MS/ AMS
Methods Development-Pu and Np

Environmental Samples:
- 0.5-200 g of soil, sediment or seaweed
- 50-200 L of seawater

Parameters optimized:
- Resin type (TEVA, AG1, AG MP-1M)
- Column size (1-20 mL)
- Washing solution (1-8 M HNO₃)
- Elution solution (NH₂OH·HCl-HCl, 0.1-1.0 M HCl)
- Flow rate (1-5 mL/min)

Performance evaluation:
Chemical yields; ²³⁷Np/²⁴²Pu chemical yield;
Decontamination of U; Method reability; Sample throughput
Auto-uint no.1---Sequential injection

Automatically handle 9 samples!
Work overnight!

## Selected results for soil analysis

<table>
<thead>
<tr>
<th>Method</th>
<th>Analyte</th>
<th>Resin</th>
<th>Chemical yield of $^{242}$Pu, $Y_{Pu}$ (%)</th>
<th>Chemical yield of $^{237}$Np, $Y_{Np}$ (%)</th>
<th>Ration of $Y_{Np}/Y_{Pu}$</th>
<th>$^{239}$Pu measured (Bq/kg) *</th>
<th>$^{240}$Pu measured (Bg/kg)**</th>
<th>Decontamination factor ***</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extraction chromatography 1, 2</td>
<td>Pu</td>
<td>TEVA (2mL, 0.7 × 5 cm)</td>
<td>97.7 ± 3.4</td>
<td>-</td>
<td>0.14 ± 0.01</td>
<td>0.09 ± 0.01</td>
<td>7.5 × 10^4</td>
<td>2.5 × 10^4</td>
</tr>
<tr>
<td></td>
<td>Np &amp; Pu</td>
<td>TEVA (2mL, 0.7 × 5 cm)</td>
<td>88.1 ± 3.4</td>
<td>85.7 ± 3.9</td>
<td>0.97</td>
<td>0.14 ± 0.01</td>
<td>0.09 ± 0.01</td>
<td>1.0 × 10^4</td>
</tr>
<tr>
<td>Anion chromatography 3, 4</td>
<td>Pu (Np)</td>
<td>AG 1-X4 (50-100mesh),</td>
<td>103.0 ± 5.2</td>
<td>84.8 ± 5.3</td>
<td>0.75</td>
<td>0.14 ± 0.02</td>
<td>0.09 ± 0.01</td>
<td>3.9 × 10^3</td>
</tr>
</tbody>
</table>
|                               |         | (2mL, 0.5 × 10cm)         |                                         |                                         |                           |                               |                           | 10 g of soil was used in each analysis. *The reference value is 0.140 ± 0.008 Bg/kg. **The reference value is 0.098 ± 0.006 Bg/kg. *** The relative standard deviations were in all instances better than 10%.

Optimized chemical purification for Pu and Pu simultaneous determination

Sample solution, with Pu (IV) and Np(IV) in 8M HNO₃ medium

Sequence 1: Wash with 100 mL of 8M HNO₃, 1.2 mL/min
Sequence 2: Wash with 100 mL of 9M HCl, 1.2 mL/min
Sequence 3: Elute with 40 mL of 0.5M HCl, 1.2 mL/min

Matrix (Ca, Mg, Fe, Pb...) Am, U

Load, 1.2 mL/min

2 mL AGM P-1M

Sequence 1

Sequence 2

Sequence 3

Th

Pu and Np

ICP-MS

Loading: 0.2 hr
Rinsing: 2.8 hr
Pu elution: 0.5 hr
Measurement: 0.5 hr (including sample preparation)

Total: 3.5 + 0.5 = 4 hr

Comparison: >2 days using traditional method

<table>
<thead>
<tr>
<th>Item</th>
<th>Extraction chromatography</th>
<th>Anion exchange chromatography</th>
</tr>
</thead>
<tbody>
<tr>
<td>Price of resin</td>
<td>☹️ High (e.g. 5600 €/500 g)</td>
<td>☑️ Relatively Low (e.g. 330-1000 €/500 g)</td>
</tr>
<tr>
<td>Chemical yields</td>
<td>☑️ 80-100% (Pu), ☼ 40-80% (Np&amp;Pu)</td>
<td>☔️ 80-100% (Pu), ☒ 70-90% (Np&amp;Pu, AG MP-1M resin)</td>
</tr>
<tr>
<td>Separation time</td>
<td>☑️ 1.5 hr/sample</td>
<td>☔️ 2.5-3.5 hr/sample</td>
</tr>
<tr>
<td>Decontamination</td>
<td>☑️ High (1-10 × 10^4 for 238U)</td>
<td>☒ Medium (1-10 × 10^3 for 238U)</td>
</tr>
<tr>
<td>Accuracy</td>
<td>☑️ High (RSD ≤ 5%)</td>
<td>☒ Medium (RSD ≤ 10%)</td>
</tr>
<tr>
<td>Consumption of chemicals</td>
<td>☑️ Low (e.g. 10 mL of conc. HNO₃/sample)</td>
<td>☔️ High (e.g. 80 mL of conc. HNO₃/sample)</td>
</tr>
<tr>
<td>Recommendation</td>
<td>Pu determination using TEVA resin</td>
<td>Pu &amp;Np simultaneous determination using AG MP-1M resin</td>
</tr>
</tbody>
</table>

Methods Development-Pu and Np

**Biological samples:**
- 1-5 L urine

**Parameters optimized:**
- Co-precipitation techniques (Ca$_3$(PO$_4$)$_3$, Fe(OH)$_3$, MnO$_2$, etc.)
- Decomposition of organic matter (acid digestion/dry ashing)
- Washing solution (0.2-1 M HNO$_3$)
- Elution solution (0.025-0.5 M HCl)

**Performance evaluation:**
- Chemical yields; $^{237}$Np/$^{242}$Pu chemical yield ratio;
- Method reability; Sample throughput
Auto-unit no.2---LOV bead injection

- LOV: Lab-on-valve; HC: Holding Coil; PP: Peristaltic Pump; PV: Pinch Valve; SP: Syringe Pump

- Column size: 5 mm i. d. x 42 mm long (ca. 0.82 mL)

Analytical procedure for urine analysis

1 L of human urine

- Ca$_3$(PO$_4$)$_2$ co-precipitation
- Fe(OH)$_3$ co-precipitation
- MnO$_2$ co-precipitation
- Evaporation

Sample pre-concentration

- Organic matter decomposition
- Dry ashing
- Acid digestion

Column separation

TEVA extraction chromatography

Measurement

ICP-MS
<table>
<thead>
<tr>
<th>Group no.</th>
<th>Pre-concentration method</th>
<th>Organic matter decomposition</th>
<th>Valence adjustment reagents</th>
<th>Operation time</th>
<th>²⁴²Pu, %</th>
<th>²³⁷Np, %</th>
<th>²³⁷Np/²⁴²Pu</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ca₃(PO₄)₂ co-precipitation</td>
<td>Dry ash</td>
<td>Ascorbic acid / conc.HNO₃</td>
<td>13 hr</td>
<td>84.7 ± 5.7</td>
<td>80.9 ± 10.7</td>
<td>0.95</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Acid digestion</td>
<td></td>
<td>8 hr</td>
<td>46.8 ± 4.1</td>
<td>8.3 ± 5.4</td>
<td>0.18</td>
</tr>
<tr>
<td>2</td>
<td>Fe(OH)₂/Fe(OH)₃ co-precipitation</td>
<td>Dry ash</td>
<td>Fe/K₂S₂O₅ / conc.HNO₃</td>
<td>6 d</td>
<td>84.3 ± 15.6</td>
<td>73.3 ± 33.0</td>
<td>0.87</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Acid digestion</td>
<td></td>
<td>5.5 d</td>
<td>80.3 ± 9.9</td>
<td>77.9 ± 10.9</td>
<td>0.97</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Acid digestion</td>
<td></td>
<td>6 hr</td>
<td>51.3 ± 0.2</td>
<td>57.5 ± 8.8</td>
<td>1.12</td>
</tr>
<tr>
<td>3</td>
<td>MnO₂ co-precipitation</td>
<td>Acid digestion</td>
<td>Fe/K₂S₂O₅ / conc.HNO₃</td>
<td>6 hr</td>
<td>88.4 ± 8.0</td>
<td>91.4 ± 10.0</td>
<td>1.03</td>
</tr>
<tr>
<td>4</td>
<td>Ca(OH)₂/Fe(OH)₂/Fe(OH)₃ co-precipitation</td>
<td>Acid digestion</td>
<td>Ascorbic acid / conc.HNO₃</td>
<td>6 hr</td>
<td>87.3 ± 6.6</td>
<td>51.2 ± 1.6</td>
<td>0.59</td>
</tr>
<tr>
<td>5</td>
<td>Evaporation</td>
<td>Dry ash + acid leaching</td>
<td>Fe/K₂S₂O₅ / conc.HNO₃</td>
<td>1.5 d</td>
<td>75.5 ± 2.6</td>
<td>81.1 ± 3.6</td>
<td>1.07</td>
</tr>
</tbody>
</table>
Methods Development-Pu, Np and U

Environmental Sample:
- 10 L of seawater

Parameters optimized:
- Resin type (TEVA, UTEVA)
- Washing solution (1-4 M HNO₃)
- Decomposition of organic matter

Performance evaluation:
Chemical yields; \(^{237}\text{Np}/^{242}\text{Pu}\) chemical yield ratio;
Method reability; Sample throughput
Auto-unit no.3---Dual-column sequential injection

Flexible control the connection of two columns!

1. Sample loading
2. Rinse with 20 mL 3 mol/L HNO₃
3. Rinse with 20 mL 1 mol/L HNO₃
4. Rinse with 20 mL 1 mol/L HNO₃
5. Rinse with 20 mL 6 mol/L HCl
6. Elute Pu/Np with 20 mL 0.1 mol/L NH₂OH·HCl-2 mol/L HCl
7. Elute U with 20 mL 0.025 mol/L HCl

---

Add conc. HCl to pH=2, add tracer $^{242}$Pu; Add 0.5 g Fe and 5 g $\text{K}_2\text{S}_2\text{O}_5$, $\text{N}_2$ bubbling for 20 min.; Add 10% $\text{NH}_3\cdot\text{H}_2\text{O}$ to pH 8-9; Add 40 g NaCl, wait 0.5-1 h, discard the supernatant.

**Fe(II) hydroxide co-precipitate**

Dissolve with 40 mL aqua regia; Digest under 200 °C for 2 h; Filtrate with GF/A filter paper; Add conc. $\text{NH}_3\cdot\text{H}_2\text{O}$ to pH 8-9; Centrifuge and discard the supernatant.

**Fe(III) hydroxide co-precipitate**

Dissolve with diluted HCl; Add 500 mg $\text{K}_2\text{S}_2\text{O}_5$; stir for 20 min.; Add conc. $\text{NH}_3\cdot\text{H}_2\text{O}$ to pH 8-9; Centrifuge and discard the supernatant.

**Fe(II) hydroxide co-precipitate**

Dissolve with 1-2 mL of conc. HCl; Add conc. HNO₃ to a final concentration of 3 mol/L HNO₃.

---

**TEVA + UTEVA**

7. Elute U with 20 mL 0.025 mol/L HCl

---

**U**

---

4. Rinse with 20 mL 1 mol/L HNO₃
5. Rinse with 20 mL 6 mol/L HCl
6. Elute Pu/Np with 20 mL 0.1 mol/L NH₂OH·HCl-2 mol/L HCl

---

**Pu/Np**
## Selected results for Pu/Np/U seawater analysis

### Typical analytical performance

<table>
<thead>
<tr>
<th>Analytical time, h</th>
<th>Chemical yield, %</th>
<th>DU</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$^{242}$Pu</td>
<td>$^{237}$Np</td>
</tr>
<tr>
<td>8</td>
<td>73.6 ± 9.8</td>
<td>73.9 ± 5.6</td>
</tr>
</tbody>
</table>

### Method application with the use of AMS measurement

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>$^{236}$U/$^{238}$U, $\times 10^{-8}$</th>
<th>$^{238}$U, µg/L</th>
<th>$^{236}$U, atom/L, $\times 10^8$</th>
<th>Measured value, mBq/L</th>
<th>Expected value, mBq/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>North Atlantic-1</td>
<td>8.88 ± 1.33</td>
<td>2.76 ± 0.41</td>
<td>(6.21 ± 0.93) × 10^8</td>
<td>&lt;0.001</td>
<td>&lt;0.005</td>
</tr>
<tr>
<td>North Atlantic-2</td>
<td>2.03 ± 0.30</td>
<td>2.17 ± 0.33</td>
<td>(1.11 ± 0.17) × 10^8</td>
<td>0.18</td>
<td>1.02</td>
</tr>
<tr>
<td>Roskilde Fjord-1</td>
<td>1.40 ± 0.21</td>
<td>1.65 ± 0.28</td>
<td>(6.88 ± 1.03) × 10^7</td>
<td>&lt;0.001</td>
<td>0.03</td>
</tr>
<tr>
<td>Roskilde Fjord-2</td>
<td>1.65 ± 0.25</td>
<td>1.65 ± 0.28</td>
<td>(5.85 ± 0.88) × 10^7</td>
<td>0.16</td>
<td>1.18</td>
</tr>
</tbody>
</table>
Environmental Sample:
• 200 L of seawater

Parameters optimized:
• Resin type (TEVA, UTEVA, AG MP-1M)
• Selection of Redox regents
• Decontamination of interferences

Performance evaluation:
Chemical yields; $^{237}\text{Np}/^{242}\text{Pu}$ chemical yield ratio;
Method reability; Sample throughput
Auto-uint no.4---Flow injection

Simultaneously handle 4 samples!
## Summary

<table>
<thead>
<tr>
<th>Objectives</th>
<th>Achievement</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Rapid determination of Pu, Np, U and Tc</td>
<td>• Environmental solids: 2-5 h/sample</td>
</tr>
<tr>
<td></td>
<td>• Large volume seawater: 1-2 days/sample</td>
</tr>
<tr>
<td></td>
<td>• Biological samples: 6 h/sample</td>
</tr>
<tr>
<td>2. Automation of the analytical procedure</td>
<td>• Sample pre-concentration: batchwise</td>
</tr>
<tr>
<td></td>
<td>• Chemical purification: automated</td>
</tr>
<tr>
<td></td>
<td>1. Auto-unit no.1: sequentially 9 samples</td>
</tr>
<tr>
<td></td>
<td>2. Auto-unit no.2: automated column packing</td>
</tr>
<tr>
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<td>3. Auto-unit no.3: automated dual connection</td>
</tr>
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<td>4. Auto-unit no.4: simultaneously handle 4 samples</td>
</tr>
<tr>
<td></td>
<td>• Measurement: automated</td>
</tr>
</tbody>
</table>
Conclusions and perspectives

Innovation of the previous work:
• Automatic
• Rapid and simple
• No need of Np isotopic tracer
• Low consumption of resins
• High sample throughput
• Low labor intensity

On-going projects:
• Tracer application studies of Pu and $^{236}$U
• Multi-radionuclide determination (Pu/Np,U, Th, Am) in environmental samples
Thank you!

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