NAMP Young Investigators Series: Environmental Radiochemistry

Dustin May
Brennen Ferguson
Frances Zengotita

National Analytical Management Program (NAMP)
U.S. Department of Energy Carlsbad Field Office

TRAINING AND EDUCATION SUBCOMMITTEE
Meet the Presenters...

**Dustin May** is a Ph.D. candidate in Human Toxicology at the University of Iowa working with Michael Schultz. He is an NRC Radiochemistry Fellow studying naturally occurring radioactivity in Iowa groundwater and its impact to public health, specifically focused on less well-understood radionuclides, polonium-210 and lead-210. He is also the manager of the State Hygienic Laboratory’s radiochemistry department. May earned a B.S. in Chemistry also from the University of Iowa in 2006. He expects to graduate in December 2018.

Contact Information: dustin-may@uiowa.edu

Presentation: **Polonium-210 and lead-210 in groundwater**
Meet the Presenters...

**Brennan Ferguson** is a Ph.D. student in Environmental Engineering and Earth Sciences at Clemson University working with Brian Powell. She is doing multi-scale experimental research on organic ligand facilitated dissolution of uranyl-phosphate minerals as part of a DOE Established Program to Stimulate Competitive Research (EPSCoR) task force. Ferguson has a B.A. in Chemistry and Environmental Studies from Alfred University in 2015 and expects to complete her Ph.D. work in December 2020.

Contact Information: bofergu@g.clemson.edu

Presentation: *Uranyl-phosphate mineral dissolution in the presence of organic ligands*
Meet the Presenters...

Frances Zengotita is an undergraduate student pursuing a dual degree in Chemistry (B.S.) and English (B.A.) at Florida International University. She is a Department of Energy Fellow, a former Nuclear Regulatory Commission Scholar and a Ronald E. McNair Fellow under the mentorship of Dr. Hilary Emerson. Her research focuses on laboratory experiments to update risk assessment models on the behavior of the actinide series elements under high ionic strength conditions relevant to the WIPP. Zengotita expects to graduate in Spring 2019.

Contact Information: fzeng002@fiu.edu
Presentation: Nd$^{3+}$ Sorption to WIPP-Relevant Minerals
Polonium-210 and lead-210 in groundwater

Dustin May
University of Iowa

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TRAINING AND EDUCATION SUBCOMMITTEE
Terrestrial NORM is Present in Groundwater

- Naturally Occurring Radioactive Material (NORM)
- Two primordial decay series of concern
  - Uranium series
  - Thorium series
- Persistent in environment
  - Parent radionuclide half-lives are billions of years
Groundwater is the major source of drinking water in Iowa
Some NORM is Regulated in Public Drinking Water

- Safe Drinking Water Act (SDWA) regulates contaminants in public drinking water
- Maximum Contaminant Level (MCL) for NORM based on excessive lifetime cancer risk
- $^{210}$Po & $^{210}$Pb are not regulated directly
- Private drinking water wells are not regulated

<table>
<thead>
<tr>
<th>Nuclide</th>
<th>MCL</th>
<th>Excessive Lifetime Cancer Risk</th>
</tr>
</thead>
<tbody>
<tr>
<td>Radium-226(^{a})</td>
<td>5 pCi/L (185 mBq/L)</td>
<td>6.78x10^{-5}</td>
</tr>
<tr>
<td>Radium-228(^{a})</td>
<td>5 pCi/L (185 mBq/L)</td>
<td>1.89x10^{-4}</td>
</tr>
<tr>
<td>Uranium(^{b})</td>
<td>30 μg/L (740 mBq/L)</td>
<td>4.49x10^{-5}</td>
</tr>
<tr>
<td>Gross Alpha Particles</td>
<td>15 pCi/L (555 mBq/L)</td>
<td>N/A</td>
</tr>
<tr>
<td>Beta/Photon Emitters</td>
<td>4 mrem/year</td>
<td>N/A</td>
</tr>
</tbody>
</table>

\(^{a}\) Enforced as Radium-226 + Radium-228  
\(^{b}\) Risk calculated based on assumed natural activity ratio (U-234:U238:U235, 1:1:0.046)
# Polonium-210 & Lead-210 Are Hazardous

<table>
<thead>
<tr>
<th>Po-210</th>
<th>Pb-210</th>
</tr>
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</table>
| • Alpha-emitter  
• 138 day half-life  
• Can exist as inorganic ions or organometallic complexes  
  – Organometallic complexes more retained, ~50%  
  – Po-210 is often found in a colloidal form in water  
• Accumulates in reticuloendothelial tissues and erythrocytes  
• Risk-based MCL = 1.1 pCi/L | • Beta-emitting radionuclide  
• 22.2 year half-life  
  – Decays to Bi-210 and then Po-210  
• Retained by body from 5-60%  
  – Dependent on age and diet  
  – Younger, higher retention  
  – Fasted, higher retention  
• Accumulates in erythrocytes and bone  
• Risk-based MCL = 2.2 pCi/L |
Limited studies performed on $^{210}\text{Po}$

- Some studies have been done on $^{210}\text{Po}$ in groundwater
  - Seiler, et al. - Nevada
  - Harada, et al. – Florida
  - Ruberu, et al. – California
  - Jacobus, et al. – Minnesota

- Studies found $^{210}\text{Po}$ in groundwater
  - Limited by scope, geographic focus
  - Areas very different geologically
Polonium-210 and lead-210 pose unknown risks

- Lack of wide-scale data examining $^{210}$Po and $^{210}$Pb in groundwater
- Alpha and beta MCLs too high to address potential risk
- Prevalence of NORM in private wells is unknown
- Higher potential carcinogenicity of $^{210}$Po and $^{210}$Pb relative to other NORM
Aim of these studies

- Identify drinking water resource types (private wells; surface supply; deep aquifer) and hydrogeological character (well depth/age, geological deposit, aquifer) in which elevated levels of regulated NORM and unregulated $^{210}\text{Po}$ are found.
Study Design

• Samples collected from all over the state of Iowa
  – 119 samples from public water supplies
  – 39 samples private wells
• Samples collected in representative manner
  – Based on drinking water withdrawals
• Analyzed for uranium-series radionuclides, various water quality parameters
  – TSS, TDS, Chloride, Alkalinity, Nitrate, Ammonia, Phosphorus
FY16 Public Well Sites
FY18 Public Wells Sites
Methods and Quality Control

• Analyses performed using a variety of methods
  – EPA 900.0 (Gross Alpha/Beta)
  – EPA 903.0 ($^{226}$Ra)
  – Eichrom methods (U, $^{210}$Pb)
  – In-house adapted method for $^{210}$Po analysis
    • $\text{MnO}_2$ pre-concentration, auto-deposition on nickel disc

• Method performance was verified using appropriate quality controls
  – Laboratory control samples, matrix spikes, duplicates, reagent blanks
Radiological Parameters Vary Across Aquifers
Gross Alpha Positively Correlates with Well Depth
Po-210 Distribution is Different from Gross Alpha
Elevated $^{210}\text{Po}$ Well Below Alpha MCL
Gross Beta Relationship with Well Depth is Less Clear
$^{210}\text{Pb}$ Not Related to Well Depth

![Graph showing activity vs. well depth](image-url)
Highest $^{210}$Pb Found at Low Beta Levels
Conclusions

• The Dakota aquifer appears different
  – Higher $^{210}$Po, U activities
  – Mean gross alpha activity higher than beta activity

• Gross alpha/beta screening is inadequate for $^{210}$Pb, $^{210}$Pb
  – $^{210}$Po shows significant negative correlation with well depth, higher at lower gross alpha activities
  – $^{210}$Pb also higher at lower gross beta activities

• All $^{210}$Po, $^{210}$Pb less than the theoretical MCLs
  – But, $\sim 7\%$ of $^{210}$Po > $1/2$ MCL
Future Work

• **Private Well Study**
  – Analyses to be completed this summer
  – Comparison to active public water supplies

• **Follow up on Dakota aquifer anomalies**
  – How is this aquifer different?
  – Are these differences widespread? reproducible?
Acknowledgements

- Thank you to:
  - Michael Schultz, PhD
  - Claire Hruby, PhD
  - Michael Wichman, PhD
  - Amanda Carl
  - Lindsey Tirado

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- State Hygienic Laboratory
- CHEEC
- NRC
References

- Cutler Prior, J.B., Janice; Howes, Mary; Libra, Robert; VanDorpe, Paul, *Iowa’s Groundwater Basics*. 2003, Iowa Department of Natural Resources: Iowa City, IA.
Uranyl phosphate mineral dissolution in the presence of organic ligands

Brennan Ferguson
Clemson University
Dangers of Uranium

- Uranium contaminates 120 sites in 36 states
  - Mining and milling
  - Nuclear Energy and Weapons
  - Natural Processes
- Public health concerns
- $\text{UO}_2^{+2}$ - soluble
System of Interest

- **Uranyl-phosphate:**
  - Stable under oxidizing conditions
  - Precipitates faster and less soluble than non-phosphate minerals
  - Can be dissolved by organic ligands: citrate, EDTA, acetic acid, bicarbonate

- **Citrate:**
  - Produced by plant roots to mobilize nutrients
  - Increases uranium accumulation in plants

Chernikovite: A Uranyl-Phosphate Precipitate

- Chemical Formula: 
  \((H_3O)(UO_2)(PO_4)\cdot3H_2O\)

<table>
<thead>
<tr>
<th>Mineral</th>
<th>Chemical Formula</th>
<th>Log (K_{sp})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chernikovite</td>
<td>((H_3O)(UO_2)(HPO_4)\cdot3H_2O)</td>
<td>-22.73</td>
</tr>
<tr>
<td>Autunite</td>
<td>(\text{Ca}[(UO}_2\text{)(PO}_4\text{)]_2\cdot4\text{H}_2\text{O})</td>
<td>-48.36</td>
</tr>
<tr>
<td>Uranyl Orthophosphate</td>
<td>((UO_2)_3(PO_4)_2\cdot4\text{H}_2\text{O})</td>
<td>-49.36</td>
</tr>
</tbody>
</table>

Uranyl-Phosphate Precipitate

- **Synthesis:**
  - Mix $\text{UO}_2(\text{NO}_3)_2$ and $\text{Na}_2\text{HPO}_4$
  - Yellow precipitate forms immediately
  - For one experiment, added citrate to $\text{Na}_2\text{HPO}_4$ solution

- **Characterization:**
  - X-ray diffraction – chernikovite
  - Scanning electron microscope (SEM) with electron dispersive spectroscopy (EDS)

<table>
<thead>
<tr>
<th></th>
<th>Wt%</th>
<th>σ</th>
</tr>
</thead>
<tbody>
<tr>
<td>U</td>
<td>52.7</td>
<td>0.2</td>
</tr>
<tr>
<td>O</td>
<td>24.7</td>
<td>0.2</td>
</tr>
<tr>
<td>C</td>
<td>15.8</td>
<td>0.2</td>
</tr>
<tr>
<td>P</td>
<td>5.5</td>
<td>0.1</td>
</tr>
<tr>
<td>Na</td>
<td>1.4</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Motivation

• **Specific:** to examine the kinetics and extent of citrate facilitated dissolution of chernikovite
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• Expanding work and collaboration: micromodels, plant uptake, and field scale studies

Motivation

• Specific: to examine the kinetics and extent of citrate facilitated dissolution of chernikovite ($\text{UO}_2\text{HPO}_4\cdot4\text{H}_2\text{O}$)

• Expanding work and collaboration: micromodels, plant uptake and field scale studies

• **Broader Impact:** to gain a better understanding of the fate of uranyl-phosphate precipitates in the environment

Research Questions

1. What is the relationship between **citrate** concentrations and **dissolved uranium** from chernikovite?
2. When does the reaction reach **steady state**?
3. Do **citrate and phosphorous** in solution **sorb** to the chernikovite surface?
4. Does the citrate affect the **remaining solid**?
Experiments

- Batch dissolution – varying citrate concentrations
- Continuous stirred-tank reactor (CSTR) – varying citrate concentrations and flow rate
- Isotope sorption study with $^{32}$P and $^{14}$C
- Post dissolution analysis of precipitate – Raman, theoretical simulations, x-ray diffraction (XRD)
Dissolved Uranium Increases with Citrate

Dissolution of chernikovite in a batch experiments for 12 hours with 12 different concentrations of citrate at pH 4
Continuous Stirred-Tank Reactor (CSTR) Experiments

- Enable measurements of rapid reactions
- Minimizes transport phenomena
- Experiments
  - Varied citrate concentration: no citrate, 0.1 mM, 1 mM, and 10 mM
  - Pretreatment: Wash for 10 reactor volumes (RV) with 10 mM NaCl, citrate for 15 RV
  - Varied flow: Wash, 1 mL/min, 0.5 mL/min, 0.25 mL/min
  - Constant pH of ~4
Dissolution of chernikovite in a continuously stirred tank reactor with four different concentrations of citrate (denoted by marker color) and three different flow rates (denoted by marker shape) at pH 4
After 24 hours, a 0.1 mM citrate and chernikovite batch system was spiked with $^{32}$P and $^{14}$C labeled citrate.
Raman - Structure Changes

Post CSTR Chernikovite and Theoretical Simulations

- \( \text{PO}_4^{-3} \) stretching: 986 – 1020 cm\(^{-1}\)
- \( \text{UO}_2^{+2} \) stretching: 821 – 867 cm\(^{-1}\)
- Downshift in \( \text{UO}_2^{+2} \) peak with increasing citrate concentration
- Theoretical simulation: 1:2 uranyl to citrate ratio with middle carboxylic group

![Graph showing Raman shift with varying concentrations of citrate](image)
XRD - Structure Changes

Chernikovite synthesized with 10 mM citrate

10 mM citrate CSTR

0 mM citrate

Chernikovite reference
Research Questions → Research Findings

1. What is the relationship between citrate concentrations and dissolved uranium from chernikovite?
   a) The presence of citrate increases the concentration of mobile uranium.
   b) Citrate surface saturation or reprecipitation hinders the dissolution.

2. When does the reaction reach steady state?
   Steady state dissolution of chernikovite was achieved within 30 min but only after the precipitate was pre-treated with citrate to achieve a more uniform chemical environment for dissolution.
3. Do citrate and phosphorous in solution sorb to the chernikovite surface?
Phosphorous and citrate in solution exchanges quickly with the phosphorous and citrate on the mineral surface, with possible re-precipitation of phosphate.

4. Does the citrate affect the remaining solid?
Citrate alters the structure of the remaining chernikovite.
Broader Impact

- Uranyl-phosphate minerals may not be as immobile as originally thought
  - Soil rich in organic matter
  - Bacteria processes
  - Plant exudates
References

- National Research Council 2010 Science and Technology for DOE site Cleanup: Workshop summary
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Nd$^{3+}$ SORPTION TO WIPP-RELEVANT MINERALS

FRANCES ZENGOTITA
FLORIDA INTERNATIONAL UNIVERSITY
Nuclear Waste clean-up

- Large amounts of radioactive waste began to accumulate in the 1940s as a byproduct from WWII and then the Cold War.

- A deep geologic repository was ultimately recommended as the best option for long-term disposal by the National Academy of Sciences.

- During the 1960s, the Chihuahuan desert of southeastern New Mexico was chosen to be an appropriate site.
Repository Storage for Transuranic Waste

What is TRU Waste?
TRU Waste (Transuranic Waste) – radioactive waste containing >100 nCi/g of alpha from elements greater than or equal to atomic # 92 (U)

How much waste is at the WIPP*?
- 90,600 m³ CHTRU
- 360 m³ RHTRU

*received through 11,900 shipments as of 2014
The WIPP is a deep geologic repository for the permanent disposal of radioactive waste that is a byproduct from the nation’s nuclear defense program.

Only plant in the world to handle and permanently dispose of transuranic waste.
In the most likely WIPP Release scenario, human intrusion can lead to direct and/or long-term brine release (US DOE 1995, US DOE 1996, Perkins et al., 1999).

Once the brine is released, it may proceed through the Rustler formation (the most transmissive layer) and pose a potential threat to the environment (Perkins et al., 1999)
Lack of Experimental Sorption Data for Trivalent Actinides/Lanthanides for Dolomite

Previously, Log$K_d$’s for dolomite highly variable, reported from 3.4 – 6

- **Brady et al. 1999**: limited residence time reactor measured $K_d$’s, exceeded Nd solubility for pH 6 – 8, limited pH range for Am(III) (pH 3 – 6), limited conditions (0.05 and 0.5 M NaCl)

- **Perkins et al. 1999**: intact-core experiments, no breakthrough recorded after many months of injection, possible precipitation of Am(III)

- **Brush and Storz 1996**: batch sorption experiments in brines, no pH reported for Am(III) $K_d$’s, limited Nd(III) $K_d$’s for atmospheric CO$_2$ in 0.05 M NaCl
OBJECTIVES

- The focus of this research is to investigate sorption processes of neodymium to dolomite minerals from the Rustler formation above the WIPP repository.

- Relevance: These data will decrease uncertainty in the WIPP risk assessment models by decreasing the current range of sorption parameters for the trivalent actinides.

- Investigated the effects of
  1. Ionic strength (Borkowski et al., 2009; Emerson et al., 2018)
  2. Ligands (Emerson et al., 2017-18; Roach et al., 2008)
  3. Microbes (Zengotita et al., 2017)
Neodymium Background

So why are we using Nd for this work?

- Nd\(^{3+}\) exhibits similar chemical behavior to Am\(^{3+}\) as it exists in the same oxidation state (Silva and Nitsche, 1995)
- Nd solubility is ~50 ppb at pH 8.5
  - Strong carbonate complexation occurs
  - Modeled in 0.1M solution
- Solubility in 5.0 M NaCl has been measured from 715 to 0.1 ppb (5x10\(^{-6}\) to 1x10\(^{-9}\) M) at pH 8.5 in over and under-saturation experiments highlighting that we still do not fully understand the high IS system (Borkowski et al., 2009).

Nd solubility in 0.1 M NaCl + atmospheric CO\(_2\)
Experimental Design: Mini Columns

Initial Conditions

- 2.2 cm column with (1 cm of dolomite + fittings) (Top figure)

- Kohlen Pump (Bottom figure) pushes electrolyte background + Nd into mini-column

- Effluent is collected in 13x100 mm polystyrene tubes in Eldex fraction collector

Experimental Design inspired by:

Experimental Design: Batch Experiments

- **Initial Conditions**
  - Variable concentrations of dolomite 0.5, 1.0, 5.0 g/L
  - Background electrolyte of 0.01 to 5 M total ionic strength [3 mM NaHCO₃ + NaCl] + 20 pbb Nd + ligands

- **Sampling Protocol**
  - Variable time measurements: 15 minutes to 60 days
  - 15 minute settling, centrifugation, or filtration prior to sampling

End over end mixer with variable solid to liquid ratio batch sorption experiments
High Ionic Strength Systems

➤ Why high Ionic Strength?
➤ The WIPP repository that contains the nuclear waste is a salty layer (up to 7.4 M).

➤ High ionic strength systems are not as well understood (Borkowski et al., 2009).

➤ What is $pC_H$?
➤ $pC_H$ values normalize the ionic strength in the solution in terms of $H^+$ ion concentration (Borkowski et al., 2009).

➤ The pH measurement is not straightforward in high ionic strength solutions due to changes in activity.

**Relevant Equations**

Eqn. 1: $\Delta pH = 0.1868[I] + 0.073$

Eqn. 2: $\Delta pH = pCH - pH$

Eqn. 3: $I = \frac{1}{2} \sum c_i z_i^2$
Gran Titrations at Variable IS

\[ p_H = A + p_{cH} \]

\[ y = 0.1696x - 0.0377 \]
\[ R^2 = 0.9884 \]

LEGEND:
Blue Our data
Navy (Rai et al., 1995)
Results: Variable Ionic Strength

Sorption (as $K_{d,con}$ in mL/g) with respect to ionic strength for 5 g/L dolomite and initially 20 ppb Nd (Emerson et al., 2018)
It is likely that the removal of Nd increases as ionic strength increases and affects mineral dissolution and precipitation processes. Previous research (Morse et al., 2002; Lucero et al., 1998) has shown that Nd is removed by (1) a fast adsorption process followed by (2) a slow co-precipitation process.
Why are the results from both experiments different?

- Different removal processes in different systems due to the reaction times
- Two reactions are occurring in batch experiments (1) fast adsorption (2) slow process with co-precipitation
- Equilibrium is not reached in the mini-column due to the short reaction time, i.e. co-precipitation does not occur. (Emerson et al., 2018)
**Biocolloid Transport of Nd**

**Sub-objective:** To observe the behavior of *Chromohalobacter* and its effect on the mobility of Nd in the presence of dolomite minerals.

We considered:
(1) Co-transport of Nd with microbes and
(2) Re-mobilization of Nd adsorbed to dolomite by microbes (Zengotita *et al.*, 2017).

**WHY *CHROMOHALOBACTER***?
- Isolated from near the WIPP site,
- Can thrive in high salt concentrations which are relevant to WIPP-related conditions,
- Very little experimental data exists on the behavior of *Chromohalobacter* species for WIPP-related conditions

(Dittrich, T.M., *et al.*, 2017)
Results: Co-transport of Nd with Biocolloids

Results for Column with initial injection of Chromohalobacter + Nd + brine

- **Unfiltered (left):** Highlights good correlation of Nd and Microbes
- **Filtered (right):** Suggest majority of aqueous neodymium associated with microbes (<3% dissolved)
  - *(Zengotita et al., 2017)*

**LEGEND:**
- **Blue Nd unfiltered recovery**
- **Red Nd filtered recovery**
- **Green microbe recovery**

*Note change in y-axis between results*
Results: Remobilization of Nd by Biocolloids

Results for column with initial injection of Nd

- Strong sorption onto the dolomite
- Microbial recoveries show no association with Nd
- No remobilization

Summary

- The microbes were unable to pull Nd off the dolomite.
  - (Zengotita et al., 2017)

Results for column with initial injection of 20 ppb Nd + brine followed by injection of Chromohalobacter + brine only (only unfiltered data presented)
Effect of EDTA on Nd Transport

Sub-objective:
The main focus of this task is to investigate the binding effect of the EDTA on Nd in the aqueous state and observe the potential removal of the contaminant on the surface of the dolomite.

Why EDTA?
- EDTA concentrations in the WIPP could reach 0.3 mM!
- EDTA is used in nuclear decontamination chemistry because of its ability to bind radionuclides and remove them from contaminated surfaces (Roach et al., 2008).
- It is persistent in the environment which makes it relevant in studies of radioactive waste disposal.
- Forms stable complexes with cationic metals (for example: Nd$^{3+}$) (Rizkalla et al., 1989)
Results: Effect of EDTA on Nd Transport

- **Initial Conditions**
  - 1000ppb Nd
  - 5 g/L dolomite
  - 5mg/L EDTA
  - IS: 0.1M

*30 & 60 day both at equilibrium

**So... what does this graph mean?**

- At 24 hours, very little sorption of Nd to dolomite with EDTA
- Around the 30 day period, the dolomite started to compete with the EDTA in respect to Nd sorption.
- The 60 day period demonstrates that the EDTA was able to complex the Nd and out-compete the dolomite

Note: The complexation that is adsorbing to the dolomite is the Nd-carbonate
Conclusions

These experiments updated parameters for risk assessment models on the environmental behavior of the actinide series elements.

1. In the absence of ligands, strong sorption of Nd to dolomite occurs from 0.1 – 5.0 M IS.
2. Ionic strength has an impact on Nd removal processes in the absence of flow potentially due to its effect on mineral dissolution and co-precipitation processes.
3. Microbes can mobilize Nd if they interact with Nd first but cannot remobilize the Nd from dolomite. However, different microbes may interact more strongly.
4. EDTA reduces sorption to dolomite ($K_{d,\text{cond}}$).

Why is this relevant?

- These data help us to better understand the potential for transport of the actinide elements from the WIPP and allow us to identify which parameters may have the greatest impact on mobility.
- The results suggest that the WIPP should consider the environmental conditions (based on the salt formation, high IS systems), presence of EDTA (found in storage and usage) and microbes. These data demonstrate that there are multiple potential transport vectors for actinides in the storage of nuclear waste.
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Questions for our presenters??
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- Young Investigators Series: Novel Uranium Nanostructures

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