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Actinide Solubility and Speciation in the WIPP

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Florida International University – October 20, 2015

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Overview

- **Role and Need for Nuclear Repositories**
  - Overall Concept
  - International Updates
  - US Approach and Current Status
- **WIPP TRU Repository Concept**
  - Design
  - Current Status
  - Path Forward
- **WIPP Safety Case: Dissolved Actinide Concentrations**
  - Overall Approach
  - Oxidation State Distribution and Redox Control
  - Solubility of Actinides
  - Colloidal Contribution and Microbial Effects
- **Concluding Observations**

Nuclear Repository Updates

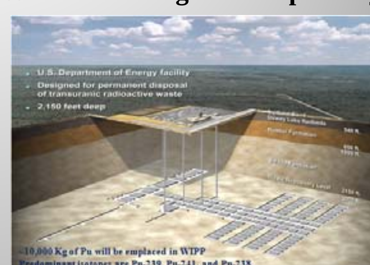
- Repository Concepts
- International
- US/EM
- WIPP

Repository Concepts: Strategic Elements

Key Concepts for the Geologic Disposal of Nuclear Waste

- Geologic isolation
- Favorable thermodynamics and chemistry
  - o Reducing conditions
  - o Reactive redox control
  - o Mildly alkaline pH
- Cost is an issue
- Transparent process with good societal receptiveness is very critical (political support)

WIPP – Existing TRU Repository



HLW?

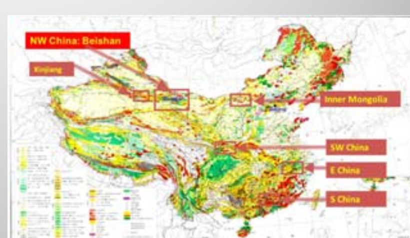
Repository Updates: International Programs
Europe and Canada

- **Repository projects in a few countries are advanced**
  - **Sweden: SKB**
    - **application in 2011 for a site licence at Forsmark (in granite) for a repository for spent fuel (in review)**
  - **Finland: Posiva**
    - **application in 2012 for a construction license for a repository for spent fuel in Olkiluoto (approved by STUK)**
    - **expected to apply for an operating licence for the repository (also in granite) in 2020**
  - **France: Andra**
    - **has moved to an industrial phase and has submitted its license application (in Clay) in 2015**
- **Other countries are in a step-wise siting process**
  - **Switzerland, Canada, and the UK**

Repository Updates: International Programs – cont.
China, Korea and Japan

China

- **Beishan region selected in 2015 (granitic site) – down-selected from 6 sites**
- **3 candidates evaluated to 1 repository (by 2050)**



Stepwise siting process in Japan and Korea

Status of Repository Science in the US

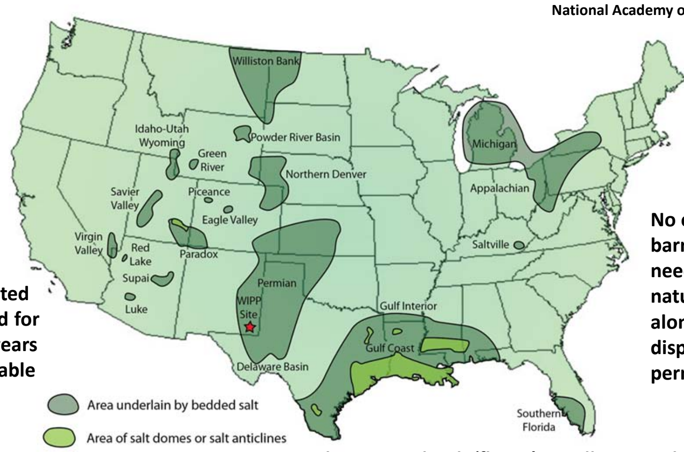
- Decision to de-commingle defense and commercial nuclear waste (2015)
- Task Force on EM Technology Development (Jan. 2015 report) concluded EM likely not able to complete remaining mission without the infusion of new technologies considering technical uncertainties and risks associated with the to-go work (3% of EM budget allocation is recommended)
- Path forward to WIPP recovery is under development (March 2017 restart?)
- Yucca Mountain Project is still "not an option"
- Program responsibility for used fuel and HLW waste disposition lies with DOE Office of Nuclear Energy (NE), they are to study various options for geologic disposal
  - Salt, crystalline (granites) and sedimentary (clay/shale) rocks
  - Repositories and/or deep boreholes
- EM and NNSA are the owners of the waste
- International cooperation is a key to progress

Why is Salt an Ideal Disposal Medium for the US?

There is a wide geographic distribution of salt with many potential sites.

"The great advantage is that no water can pass through salt. Fractures are self healing…."

National Academy of Sciences, 1957



Salt has existed underground for millions of years and has a stable geology.

No engineered barriers are needed – the natural barrier alone makes disposal in salt permanent.

Salt at great depth 'flows.' It will encapsulate waste and isolate it from the surface for eons.

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Waste Isolation Pilot Plant (WIPP)

WIPP is a permanent disposal facility for TRU waste

- **Located in southeast New Mexico**
- **First certified in 1998, first shipment in 1999, recertified every 5 years**
- **Operated by U. S. Department of Energy (DOE)**

Regulated by U. S. Environmental Protection Agency (EPA) and New Mexico ED

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Types of Waste


Contact-handled (CH)

- Large volume
  - ~169,200 m^3 capacity
- No shielding required
- Stacked on floor of waste rooms

Remote-handled (RH)

- Small volume
  - ~7,000 m^3 capacity
- Contains short-lived gamma emitters
- Shielding required
- Emplaced in horizontal boreholes in waste room walls


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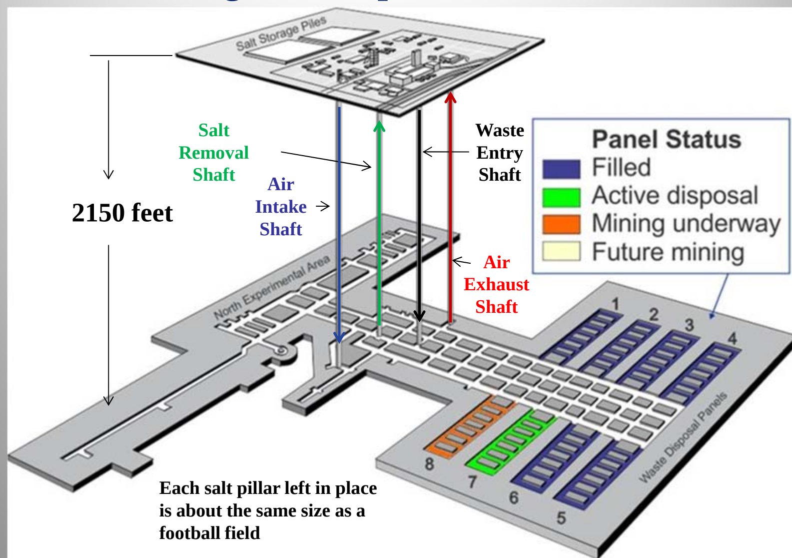
WIPP Underground Operations



WIPP Status

WIPP, although currently not operating, is certified to operate and is in the process of being recertified (3rd time) by the EPA.

Tentative Recertification Schedule:

October 10, 2014 – Notice of Availability; Official opening of public comment period
June 16-17, 2015 – Stakeholder Meetings in Carlsbad and Albuquerque
Summer 2015 – EPA and DOE reach agreement on parameter changes to run the PABC-2014
December 2015 – PABC-2014 completed
August 25, 2016 – EPA makes completeness determination
February 25, 2017 – EPA recertifies WIPP

11,894	shipments received
33,000	type B packages unloaded
91,000	cubic meters of TRU waste disposed
14,200,000	loaded miles
24	storage sites cleaned

* Through February 4, 2014

Safety Incidents that Shut Down WIPP

Salt Truck Fire (February 5, 2014)

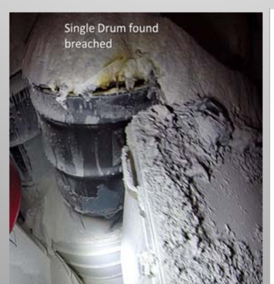
At approximately 11 AM, there was an underground fire in the WIPP when a 29 year-old salt hauler vehicle caught fire near the salt shaft. All 86 people in the mine at the time were able to exit safely but six were treated for smoke inhalation. This led to a shutdown of the WIPP to review/address safety issues raised by this incident.

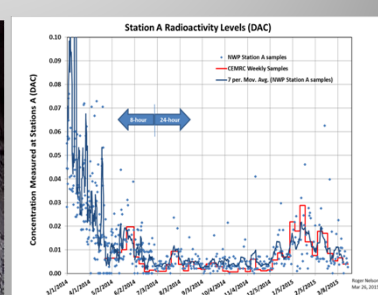
Safety Incidents that Shut Down WIPP

Actinide Release Incident (February 14, 2014)

At approximately 11 PM, a CAM alarm signaled the presence of airborne contamination in the WIPP underground. This was later found to be a release from a single container (LANL waste stream MIN-02) failure in WIPP room 7 in panel 7 (shown on the bottom) that led to a measurable but small release of Pu/Am from the WIPP. This release event has been extensively investigated and the path forward to resume operations is being developed.

WIPP Panel 7. room 7





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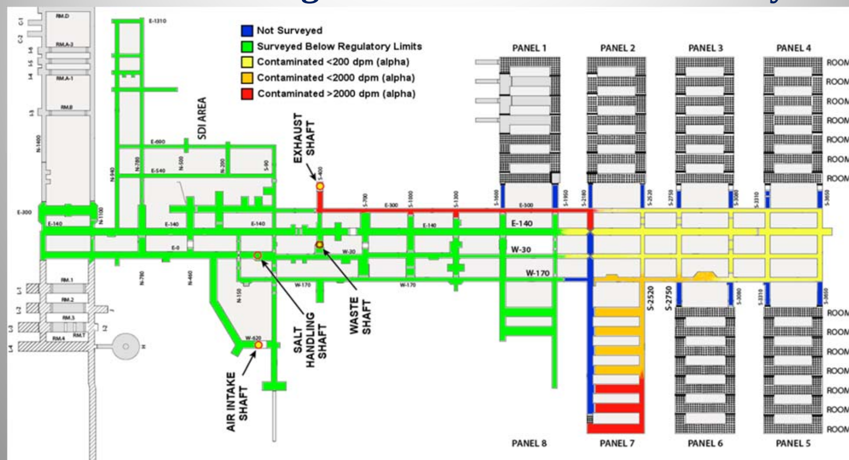
WIPP Underground Contamination Survey



Path forward is being finalized and operation is expected to resume within two years

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Actinide Solubility and Speciation In the WIPP

- ❑ Redox and Oxidation State
  - Example reduction of Pu by Fe
- ❑ Actinide Solubility
  - Example of Thorium solubility in brine
- ❑ Microbial Effects and Colloidal Contributions
  - Examples of sequential filtration results and Biosorption

Why is Actinide Research Needed?

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Actinide Research: Motivation and Application

WIPP-specific goals

- **Support ongoing recertification (next is CRA-2014)**
- **Support model changes and updates under consideration (2-year program plan)**
- **Measure needed/planned parameter updates**

Rationale for the work beyond WIPP

1) **Groundwork for all WIPP repository options (including an expanded mission?)**
2) **Possible development of Salt-based repositories for HLW**

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Importance of Actinides and Oxidation State Distribution in the WIPP

Overall Release: **Pu ~ Am >> U > Th >> Np, Cm and fission products**

Oxidation State: **III > IV >> VI > V**

Oxidation State Distribution of Key Actinides in WIPP Performance Assessment

Actinide	Oxidation State				Speciation Data used in Model Predictions
	III	IV	V	VI	
Uranium		50%		50%	Thorium for U(IV), 1 mM fixed value for U(VI)
Plutonium	50%	50%			Am/Nd for Pu(III) and thorium for Pu(IV)
Americium	100%				Americium/neodymium

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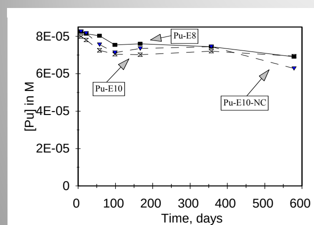
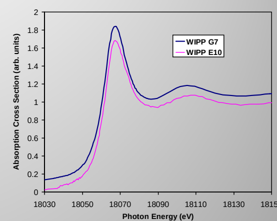
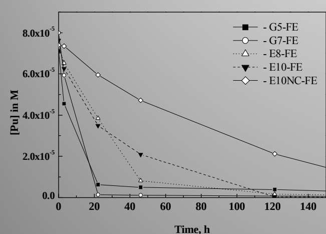
Plutonium Oxidation State in Brine 1990s Experiments

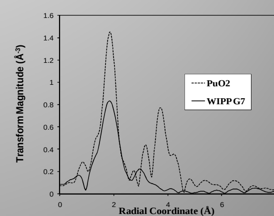


No reductant





Fe Added



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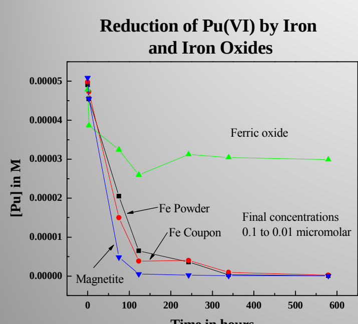
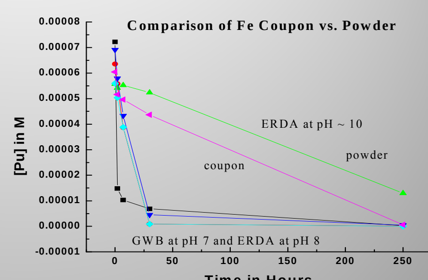
Plutonium Oxidation State Distribution in Brine ACRSP Experiments (Current/ongoing)





Pu(V/VI) reduction was always observed when reduced Fe, Fe(0/II), was present

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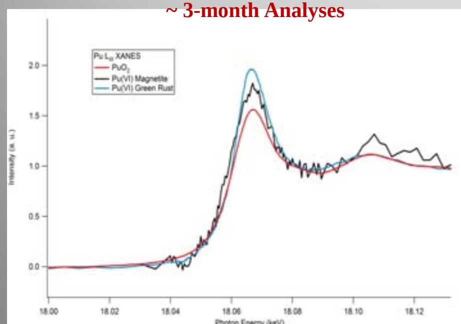
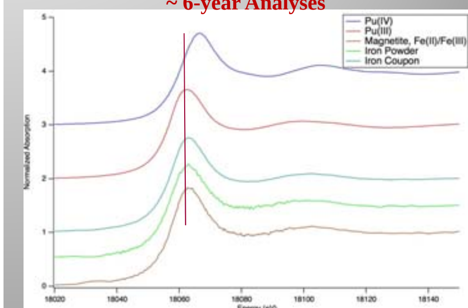
Pu(V/VI) Reduction by Lower-Valent Fe in Brine

Final Pu oxidation state is mostly Pu(III) and Pu(IV) colloid based on TTA extraction, ~ no extractable Pu(IV)

~ 3-month Analyses



~ 6-year Analyses



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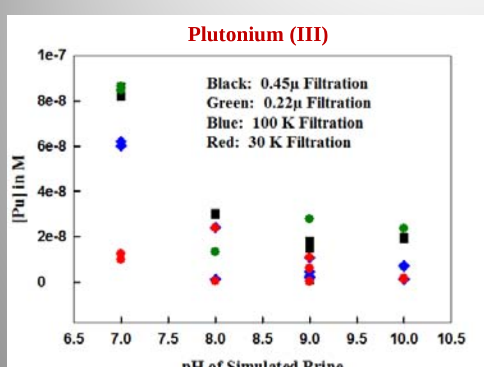
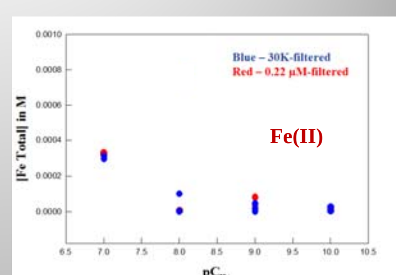
Plutonium (III) Association with Iron Colloids





Long-term Pu studies with iron show colloidal enhancement well above the >10 nm operational definition of intrinsic plutonium colloids and correlates with the [Fe] present in solution. states

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Predominance of Pu(III) under Anoxic Conditions

Qualitative Redox Indicators for Iron Interactions with Plutonium under Anoxic Conditions

Experiment	Description	[a] Oxidation State of Pu Solid	[b] $[Fe]_{total}$ in mM (%Fe^{2+} in solution)	[c] E_h Measured (± 3 mV)
PuFe23OX	ERDA-6 brine at pH ~9 with excess magnetite	~87% Pu(III), rest Pu(IV)	0.12 (25%)	-122 mV
PuFeCE8	ERDA-6 brine at pH ~8 with Fe coupon	~100 % Pu(III)	ND	ND
PuFeCE10	ERDA-6 brine at pH ~ 9.6 with Fe coupon	~100% Pu(III)	0.27 (100%)	ND
PuFeP	ERDA-6 brine at pH~9 with excess Fe powder	~100% Pu(III)	0.18 (100%)	-175 mV
PuFeC	ERDA-6 brine at pH ~ 9 with Fe coupon	~90% Pu(III), rest Pu(IV)	0.18 (58%)	-110 mV
PuFeG7	GWB brine at pH ~6.7 with Fe coupon	~ 100% Pu(III)	12.62 (97%)	-210 mV

a. Pu(III) content established by XANES analysis of solids
b. Fe(II) content established by analysis using FerroZene®
c. E_h measurement made using an Orion combination ORP electrode
ND – not determined

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Summary: Pu Redox in the WIPP

- **Reducing conditions are generated due to anoxic corrosion that leads to Fe^{2+} production**
- **Pu(III) predominates in solid and aqueous phase for Pu-242 and the presence of reduced iron – the long-term role of Pu(III) is not understood**
- **Radiolysis and the presence of organics may shift this redox to Pu(IV) even under Fe-dominated anoxic conditions (still under investigation)**

Pu(V/VI)

↓ **Rapid (aqueous)**

Pu(IV)

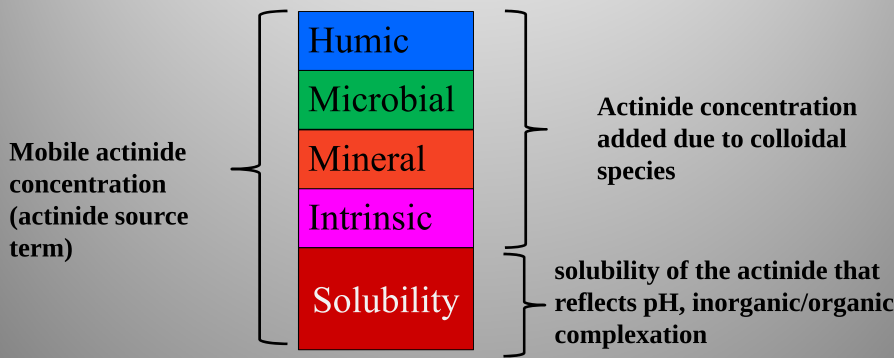
⇅ **Slow (solid phase?)**

Pu(III)

Mobile Actinide Concentration

In the WIPP concept, colloidal species contribute to the source term in dissolved brine release (DBR) release scenarios

- **Colloidal transport is not a significant issue**
- **Structure and physical properties are not important**



Approach to Actinide Concentrations in the WIPP

1. **Assess and track actinide inventory**
   a) **Pu, Am, Cm, U, and Th are potentially important**
   b) **Cm and Np is eliminated as a consideration**
   c) **Available chelating/complexing ligands**
2. **Assign oxidation state distribution by expert opinion**
   a) **Built-in conservatism**
   b) **Fe/microbially-induced redox environment that is reducing**
   c) **Support with WIPP-specific data**
3. **Establish effective solution concentration**
   a) **Model/measure actinide solubilities using redox-invariant analogs**
   b) **Account for colloidal contribution by process-specific enhancement factors: intrinsic, bio, inorganic, HA**
   c) **Assign an uncertainty distribution based on literature data review**

Inventory (Panels 1-4 and Projected)

Amount (in kilograms) of Key Waste Package Components and Actinides Present in the WIPP					
Isotope/Material	Panel 1	Panel 2	Panel 3	Panel 4	WIPP (total projected)
Radionuclide (in kg)					
Am-241	34.63	9.21	7.65	3.58	203
Pu	2571.00	1405.00	441.10	341.60	12,000
Pu-239	2416.00	1306.00	404.80	311.10	9,130
U	22160.00	6844.00	1504.00	1723.00	226,000
U-238	22090.00	6802.00	1487.00	1704.00	191,000
Np-237	0.58	1.26	0.98	3.37	32.5
Emplaced/Waste Materials (in kg)					
Iron based alloys	3,327,871	4,922,035	4,559,000	4,380,000	49,100,000
Aluminum based alloys	5,459	17,730	9,859	9,437	457,000
Other alloys	46,793	121,526	89,370	26,130	5,795,000
MgO – Eng. Barrier	4,482,000	6,667,000	6,437,000	6,445,000	51,430,000
Cellulosics	706,141	477,213	548,280	833,066	4,650,000
Plastic	522,688	876,399	890,640	1,065,040	9,510,000

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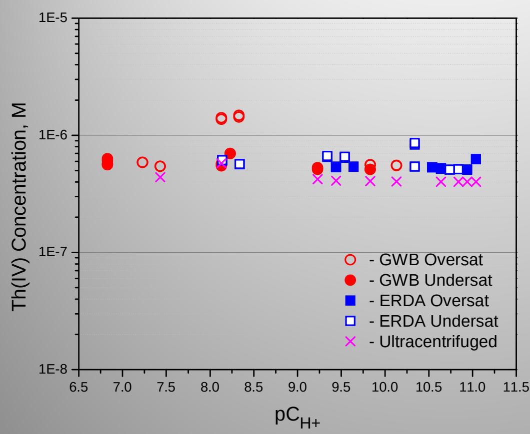
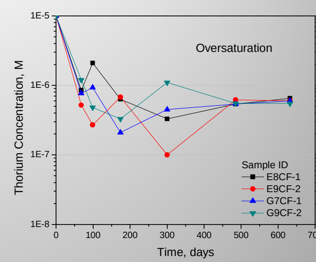
Thorium Solubility Studies: Experimental Approach

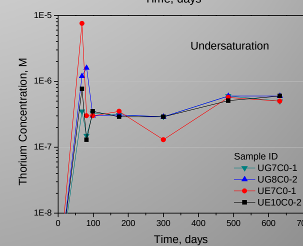
Brine Systems:
- GWB and ERDA-6 Brine – Bracketing approach
- Anoxic conditions in Glovebox
- pH range of 7-12

General Approach:
- Long-term (over 4 years) data
- Routinely filtered to 100K MWCO (~20 nm)
- ICP-MS analysis to measure concentration
- Effects of organics and carbonate evaluated

An(IV): Solubility of Thorium in WIPP







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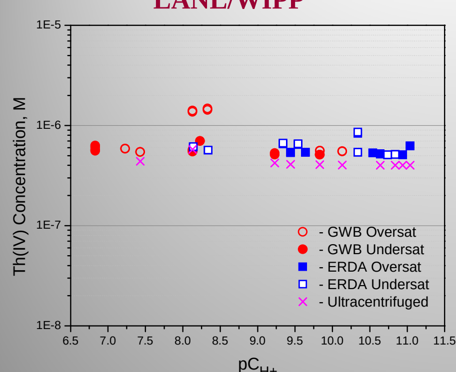
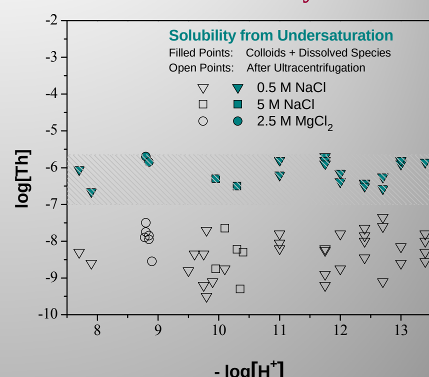
Intrinsic Colloids: Thorium Data

LANL/WIPP



INE/Germany



Why are these different?

1) **Brine Composition and degree of saturation – Mg colloidal content**
2) **Time – colloidal fraction decreases with time**

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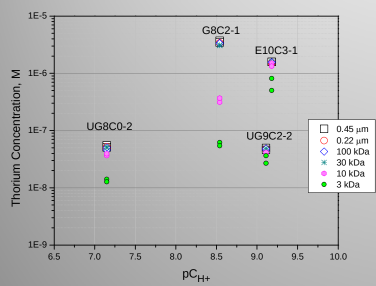
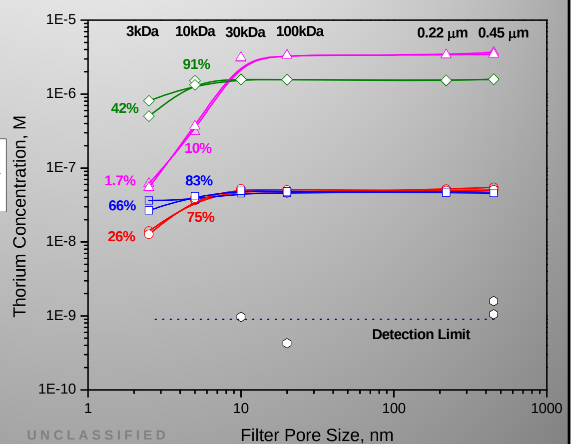
Effect of Filtration on Thorium Concentrations

~ no effect to 10 nm, 6 nm data matches ultracentrifugation results





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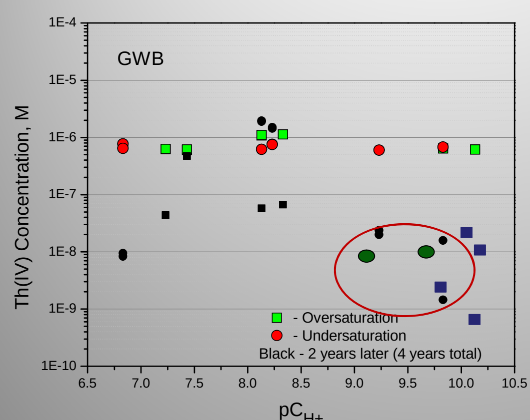
Results of Th(IV) solubility measured after 4 years of equilibration



Altmaier et al., 2004 – 5 M NaCl ultracentrifugation data

Biosorption experiments with *Chromohalobacter sp*.

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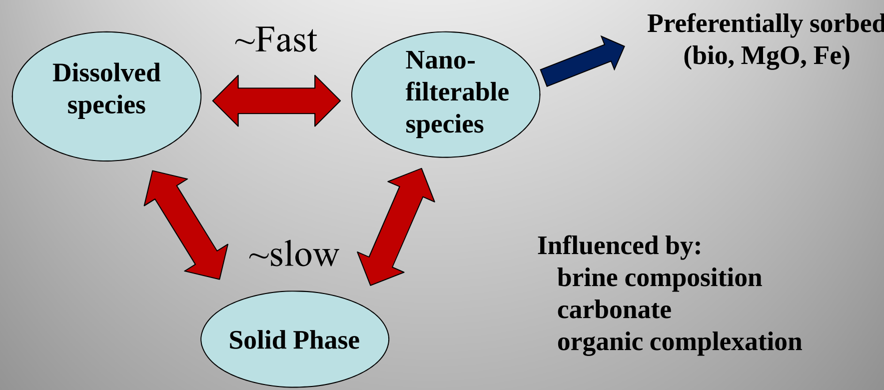
Summary of Observations: Thorium Brine System



Influenced by:
brine composition
carbonate
organic complexation

A similar mechanism was proposed in Altmaier, Neck, Fanghanel. Radiochimica Acta ***92****(9-11), 537-543 (2004).*

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Assumptions about Intrinsic/Mineral Colloids ~ 1995 (first license application)

- **The tendency to colloid formation is element-specific (e.g. all Pu oxidation states have the same tendency to form colloids)**
- **Plutonium is the only element that will form intrinsic colloids (other actinides where eliminated based on literature search)**
- **Intrinsic colloid enhancement factor is defined as a maximum potential colloid concentration that is added to the predicted solubility**
- **Iron oxides are the only species that form mineral colloids (screened ~20 minerals)**
- **~10 nm size definition was proposed**
- **Enhancement factor was in the end defined by the overall analytical uncertainty in measuring [Pu] – no colloidal fraction was directly measured – this is 10^{-9} M.**

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Intrinsic Colloids in High Ionic-Strength Brine WIPP Experiments

- **Long-term Studies (4-6 years)**
- **Simulated brine systems (GWB and ERDA-6)**
- **UO_2^{2+}, Nd^{3+}, Th^{4+}, Pu^{3+} (Fe-dominated system)**
- **Sequential filtration and some ultracentrifugation**
- **pC_{H+} from 7-12**

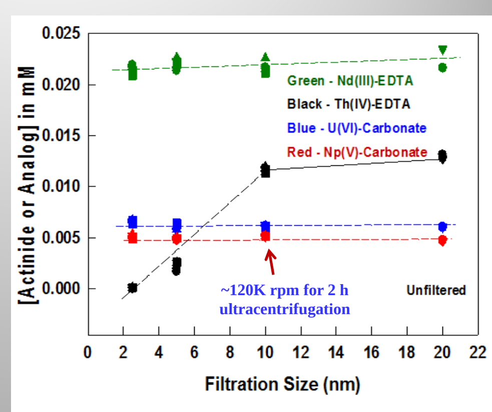
WIPP Simulated Brine Composition

Ion or property[a]	GWB Brine Composition[b]	GWB after reaction with MgO (phase 5), halite, and anhydrite[c]	ERDA-6 Brine Composition[d]	ERDA-6 after reaction with MgO (phase 5), halite, and anhydrite[c]
$B(OH)_x^{3-x}$ (see footnote f)	158 mM	186 mM	63 mM	62.3 mM
Na^+	3.53 M	4.77 M	4.87 M	5.30 M
Mg^{2+}	1.02 M	0.330 M	19 mM	136 mM
K^+	0.467 M	0.550 M	97 mM	96.0 mM
Ca^{2+}	14 mM	11.1 mM	12 mM	11.6 mM
SO_4^{2-}	177 mM	216 mM	170 mM	182 mM
Cl^-	5.86 M	5.36 M	4.8 M	5.24 M
Br^-	26.6 mM	31.3 mM	11 mM	10.9 mM
Total Inorganic C (as HCO_3^-)	Not reported	0.379 mM	16 mM	0.455 mM
pH	Not reported	8.82	6.17	8.99
Ionic Strength (M)	7.44	6.44	5.32	5.99

a - ions listed represent the total of all species with this ion.
b – From Snider 2003a
c – From Brush and Domski 2013a
d – From Propielak et al., 1983
f – Boron species will be present in brine as boric acid, hydroxyl polynuclear forms ($B_3O_3(OH)_4^-$, and/or borate forms (e.g. $B_4O_7^-$)

Experimental Approach Sequential Filtration

- **Long-term (multi-year) actinide solubility experiments were re-analyzed to determine size distribution of species**
- **Sequential filtration used in most cases (Amicon ultrafilters) at 0.45 μM, 0.22 μM, 100 kDa (20 nm), 30 kDa (10 nm), 10 kDa (5 nm) and 3 kDa (2.5 nm)**
- **Technique demonstrated in GWB brine systems with "truly" dissolved species**



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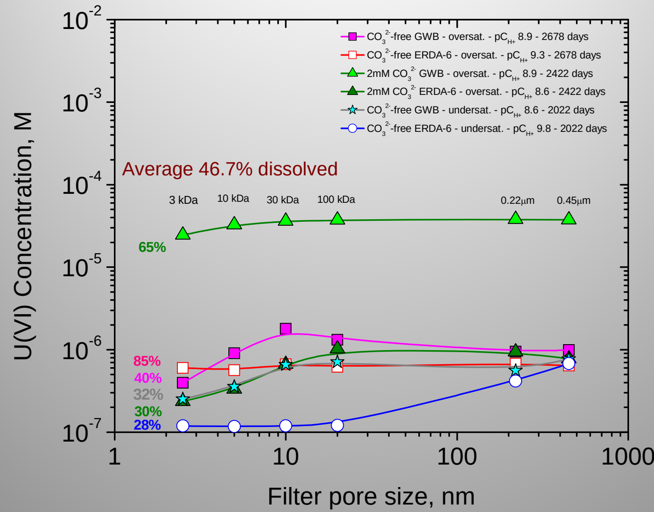
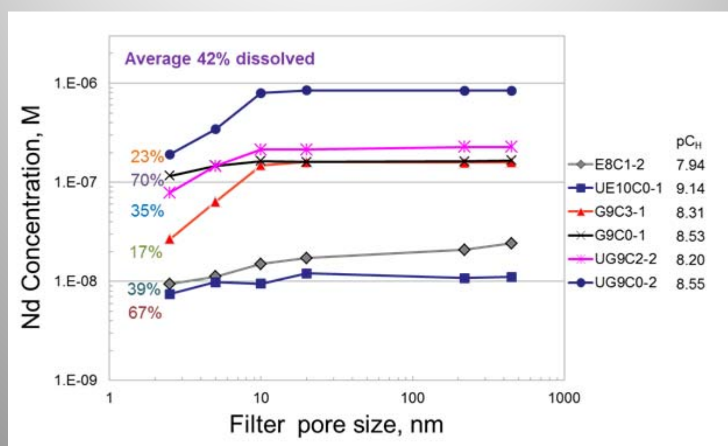
Sequential Filtration Results for U(VI) in Brine



Sequential Filtration Results for Nd^{3+} in Brine



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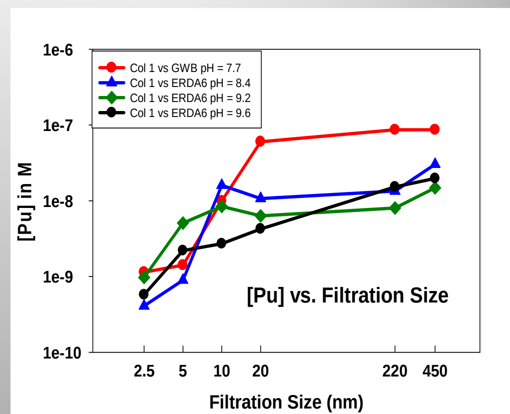
Sequential Filtration Results for Pu^{3+} in Brine Size distribution at various pH

- Much smaller effect noted at >10 nm filtrations
- Approaching 90% of Pu in solution is between 2.5 and 10 nm in size



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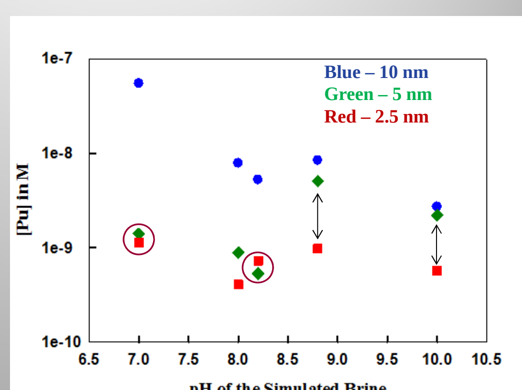
Sequential Filtration Results for Pu^{3+} in Brine < 10 nm filtration fraction

- Overall solubility is quite low
- pH trend is noted, < 5 nm fraction grows with pH



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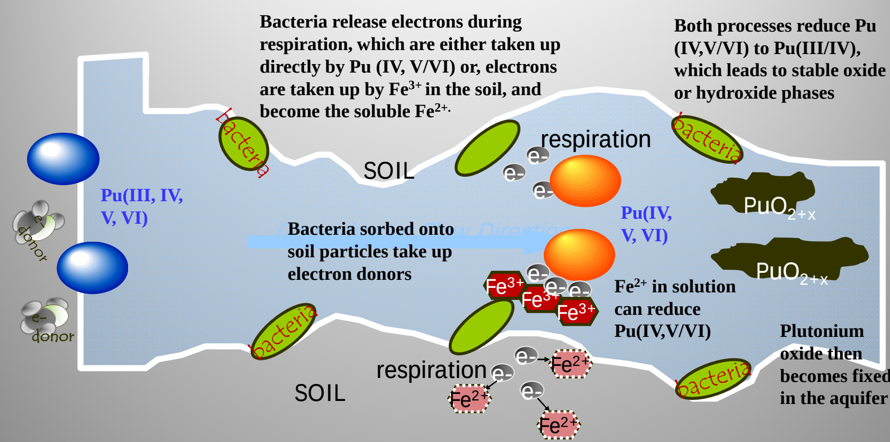
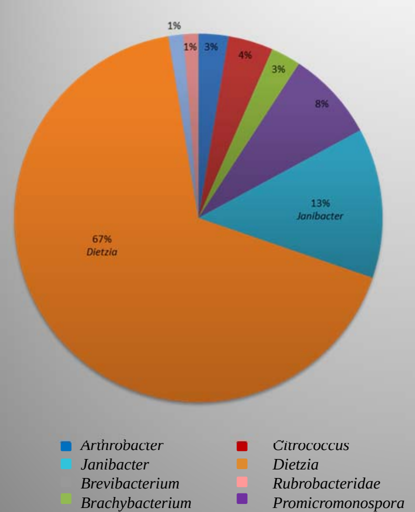
Microbial Interactions lead to Multiple Effects on Actinide Speciation that Affect Mobility



- No bioreduction noted under WIPP-specific conditions
- Measured toxicity was observed and the subject of ongoing investigations

COMMUNITY STRUCTURE IN WIPP WASTE

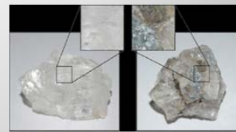


- Low diversity
- All DNA sequences originate from only one phylum, *Actinobacteria*
- Organisms in this phylum are known to survive extreme conditions:
  - Desiccation
  - UV and g irradiation
  - Low temperatures
  - High salt
  - High pressure

Biosorption - Microorganisms

Bacteria

Chromohalobacter sp.
isolated from area groundwater, borehole seep, WIPP and Gorleben halites; strain used tolerates pC_{H+} 5-9, 0.9-4.3 M NaCl; 0.3-0.5 x 1.5-2 µm size

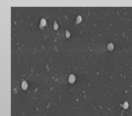


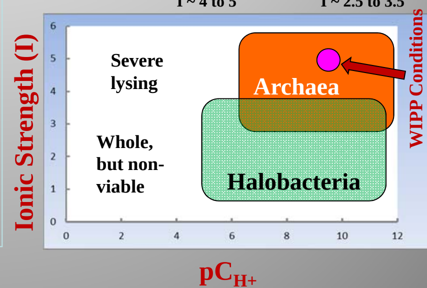
"Pure" Halite vs. Clayey Salt

Optimum Growth: Archaea: pC_{H+} = 8-10, I ~ 4 to 5; Bacteria: pC_{H+} = 7-9, I ~ 2.5 to 3.5

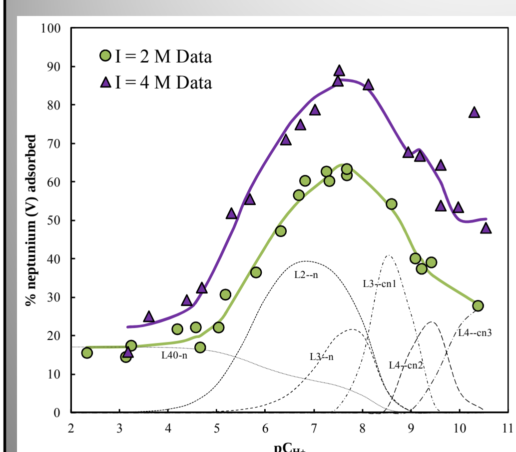
Archaea

Halobacterium noricense
isolated from incubations of halite in generic media and in WIPP brines; detected in other subterranean salts worldwide (including Germany); requires 2.5-5 M NaCl, tolerates pC_{H+} 6-10; 0.3-1.5 x 0.3-1.5 µm size





Titration Results: *Chromohalobacter* sp.



Speciation scheme that accounts for Np-carbonate species adsorption

Calculated Neptunium (V) Binding Constants

	Reaction	log K I = 2 M	log K I = 4 M
1	$NpO_2^+ + R\text{-}L_4^x \leftrightarrow R\text{-}L_4NpO_2^{x+1}$	3.01 ± 0.1	3.04 ± 0.1
2	$NpO_2^+ + R\text{-}L_2^{x-1} \leftrightarrow R\text{-}L_2NpO_2^x$	3.71 ± 0.1	3.86 ± 0.1
3	$NpO_2^+ + R\text{-}L_3^{x-1} \leftrightarrow R\text{-}L_3NpO_2^x$	4.11 ± 0.1	4.91 ± 0.2
4	$NpO_2CO_3^- + R\text{-}L_3^{x-1} \leftrightarrow R\text{-}L_4NpO_2CO_3^{x-2}$	8.32 ± 0.1	8.71 ± 0.1
5	$NpO_2(CO_3)_2^{3-} + R\text{-}L_4^{x-1} \leftrightarrow R\text{-}L_4NpO_2(CO_3)_2^{x-4}$	10.89 ± 0.1	11.86 ± 0.1
6	$NpO_2(CO_3)_3^{5-} + R\text{-}L_4^{x-1} \leftrightarrow R\text{-}L_4NpO_2(CO_3)_3^{x-6}$	11.86 ± 0.1	12.73 ± 0.1

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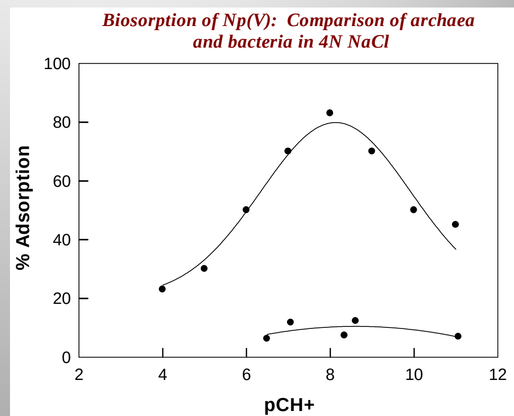
Biosorption of Np(V) Towards *H. noricense* - Results

- **Speciation: Np(V) exists as NpO_2^+ aquo species (~2 μM) until pC_{H+} ~ 9 where carbonate complexation occurs.**
- **Sorption onto archaea is relatively featureless and about 10% of comparable bacterial absorption.**
- **Np(V) is stable and reversibly sorbed**
- **Relatively low toxicity (in contrast to soil bacteria)**



Summary of Biosorption Data at the Expected WIPP pC_{H+} (Normalized to biomass of 10 g/L)

- **Np(V)**
  - *Archaea*: ~ 10% sorption
  - *Bacteria*: 70-85% sorption
- **Nd(III)**
  - *Archaea*: ~ 25% sorption
  - *Bacteria*: 50-65% sorption
  - EDTA reduced sorption in both cases by ~ 40%
- **Th(IV)**
  - *Archaea*: ~ 10-20% (no EDTA) and ~0% (EDTA) sorption
  - *Bacteria*: ~ 80% (no EDTA) and 57% (EDTA) sorption
  - At $pC_{H+} > 9$, cannot differentiate between sorption and precipitation, due to colloids and "induced" precipitation

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Summary of "Colloid" Results

Intrinsic colloids

- Nano-filtrable species are almost always observed, most are smaller than 10 nm
- Need to investigate structure and long-term stability

Mineral colloids

- Little to no Mg-derived colloids are noted
- Fe colloids are observed

Biocolloids (biosorption)

- Strong sorption noted for halobacteria (typical of soil bacteria)
- Much lower sorption noted for archaea – we do not yet have an explanation for this
- Strong coupling between precipitation and bioassociation observed at the higher pHs considered (pC_{H+} >9)

Humic Colloids

- Humics are not stable in brine systems

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Oxidation-specific "Mobile" Actinide Concentrations

Calculated Oxidation-Specific Solubility of Actinides in Equilibrated WIPP Brine (GWB – high Mg brine, ERDA-6 – NaCl brine) [Brush and Domski, 2013]

Actinide Oxidation State, and Brine	Actinide/ Analog Used	CRA-2004 PABC (M)	CRA-2009 PABC (M)	CRA-2014 PA (M)
III, GWB	Am/Nd	3.87×10^{-7}	1.66×10^{-6}	2.59×10^{-6}
III, ERDA-6	Am/Nd	2.88×10^{-7}	1.51×10^{-6}	1.48×10^{-6}
IV, GWB	Th	5.64×10^{-8}	5.63×10^{-8}	6.05×10^{-8}
IV, ERDA-6	Th	6.79×10^{-8}	6.98×10^{-8}	7.02×10^{-8}
V, GWB	Np	3.55×10^{-7}	3.90×10^{-7}	2.77×10^{-7}
V, ERDA-6	Np	8.24×10^{-7}	8.75×10^{-7}	8.76×10^{-7}

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Conclusions and Observations

- **International programs are moving forward, but at a very slow and somewhat sporadic pace.**
- **In the United States, the Salt repository concept, from the perspective of the long-term safety case, remains a viable option for nuclear waste management despite the current operational issues/concerns.**
- **Current model/PA prediction (WIPP example) are built on redundant conservatisms. These conservatisms are being addressed in the ongoing and future research to fill existing data gaps.**
  - **Redox control of plutonium by Fe(0, II)**
  - **Thorium (analog) solubility studies in simulated brine**
  - **Contribution of intrinsic and biocolloids to the mobile concentration**
  - **Clarification of microbial ecology and effects**
- **There is no downside to establishing a strong scientific basis for a repository concept. This is a necessary and critical aspect of the regulatory process.**

Thank you for your attention

Acknowledgments

LANL Actinide Team:

Don Reed:	Pu chemistry, Np and Th biosorption studies
Marian Borkowski:	Thorium, Neodymium chemistry
David Ams:	Np sorption towards halobacteria
Jef Lucchini:	U chemistry and Nd biosorption studies
Danielle Cleveland:	ICP-MS analysis of Nd, Th, Pu and Np
Julie Swanson:	Microbial characterization and growth
Karen Simmons:	Microbial growth and DNA Analysis
Michael Richmann:	EQ3/6 speciation calculations modeling
Tim Dittrich:	Colloid transport and sorption studies

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