Radiochemistry Webinars
Nuclear Fuel Cycle Series

- *Advanced Partitioning Technologies in the U.S.*

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A URS-led partnership with B&W and AREVA

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UNIVERSITY of CALIFORNIA · IRVINE

NM STATE UNIVERSITY CARLSBAD

Meet the Presenter...   *Dr. Jenifer Braley*

Dr. Braley joined the faculty at the Colorado School of Mines (CSM) in the fall of 2012 after two years at Pacific Northwest National Laboratory. During her undergraduate research, she studied the solid-state synthesis of *f*-block elements at Colorado State University with Professor Peter Dorhout. In 2006 she worked with Dr. Ken Nash at Washington State University, where she examined the fundamental solution chemistry of the *f*-elements relevant to solid-liquid and liquid-liquid separations chemistry. While completing graduate school, she participated in an internship at Eichrom Technologies with Dr. Phil Horwitz, and bolstered her understanding of extraction chromatographic (solid-liquid) separations. She is currently the Program Chair for the American Chemical Society (ACS) Division of Nuclear Science and Technology, and a member of the ACS Younger Chemist Committee. Since joining the faculty at CSM, she has developed coursework and leads a research group that educates students on the fundamental and applied concerns of nuclear chemistry and radiochemistry (including the nuclear fuel cycle, nuclear forensics, and radioisotope production). As a member of the Nuclear Engineering Program at CSM, she actively engages the U.S. Geological Survey 1 MW TRIGA Nuclear Reactor at the Denver Federal Center to accomplish research and educational goals. She is a 2010 recipient of a DOE Innovations in Fuel Cycle Research award, 2013 recipient of a DHS SCUREF Junior Faculty award, and 2014 recipient of a DOE Early Career award.

Phone: 303-273-3996
Email: jbraley@mines.edu

Advanced Partitioning Technologies in the U.S.

Professor Jenifer C. Braley
Colorado School of Mines

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

TRAINING AND EDUCATION SUBCOMMITTEE

Learning Objectives

- Relevant nuclear chemistry
- Various nuclear fuel cycle options
- Knowledge of current U.S. nuclear fuel cycle policy
- Ability to define advanced partitioning
- Understand motivations for advanced partitioning
- Acquire cursory understanding of various partitioning techniques
- Learn different U.S. approaches to advanced partitioning strategies
  - Solvent extraction emphasis

Outline

- Nuclear reactions in a reactor
- Types of nuclear fuel cycles
- Current U.S. nuclear fuel cycle policy
- Motivations for advanced partitioning
- Various techniques for accomplishing advanced partitioning goals
- Quick overview of solvent extraction
- Solvent extraction strategies considered in the U.S.

Nuclear Fuel Cycles - Generic
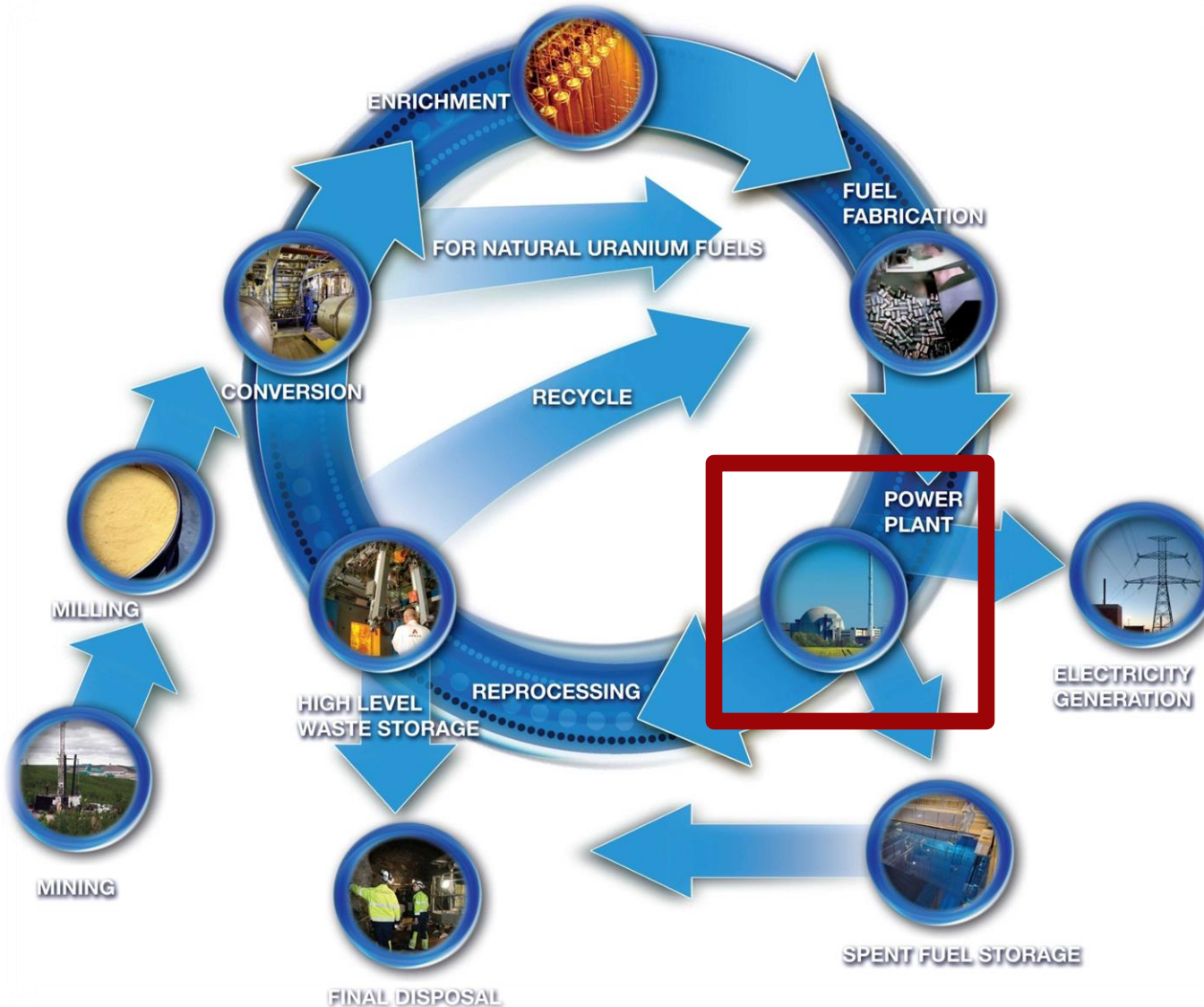
Nuclear Chemistry in Nuclear Reactors

Nuclear Fission
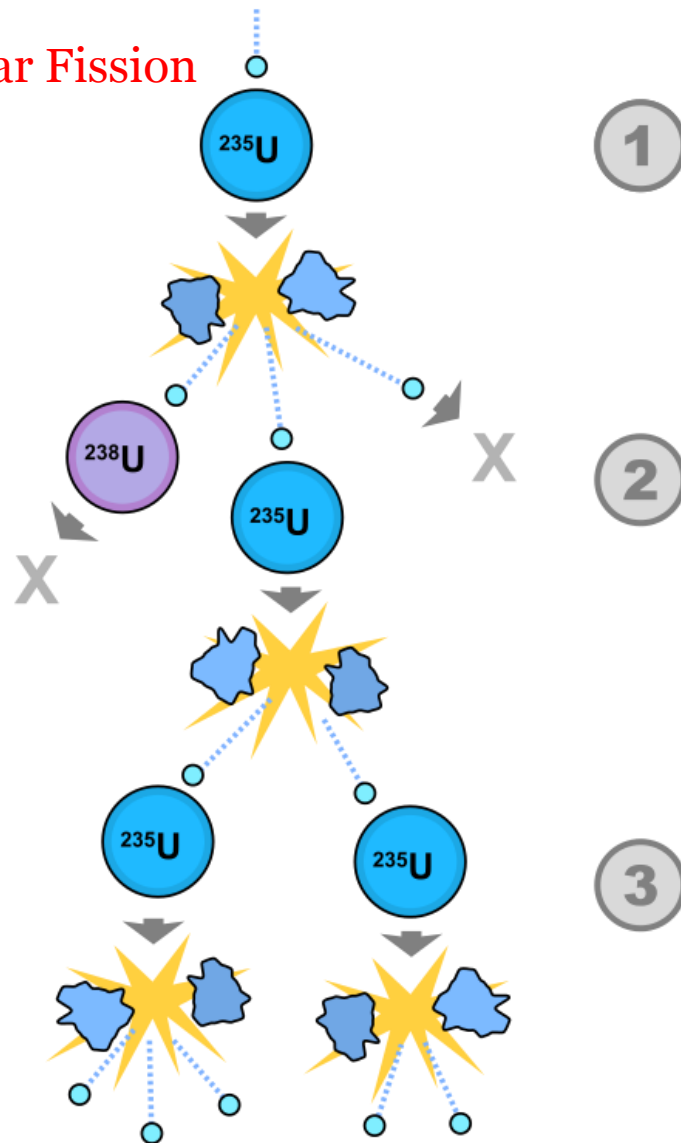
Neutron Absorption/Capture

$$^{238}U + n \quad ^{239}U \quad \beta \quad ^{239}Np$$
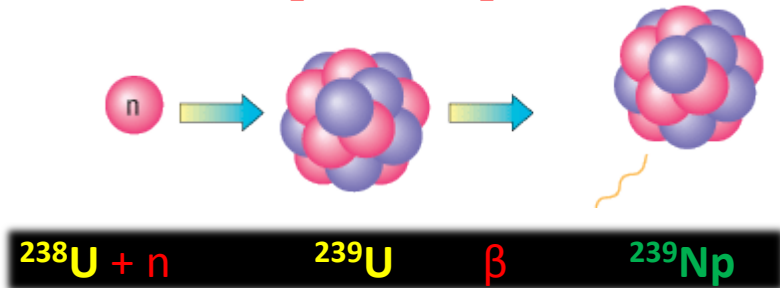
- The fission process releases energy and produces "fission products"
  - Second-row transition metals
  - Lanthanides

- The neutron capture process competes with the fission process and produces heavier actinides
  - Neptunium
  - Plutonium
  - Americium
  - Curium

Nuclear Fuel Cycle

ENRICHMENT

FUEL FABRICATION

FOR NATURAL URANIUM FUELS

CONVERSION

REC

POWER PLANT

MILLING

HIGH LEVEL WASTE STORAGE

REPROCESSING

ELECTRICITY GENERATION

MINING

FINAL DISPOSAL

SPENT FUEL STORAGE

OPEN

- No recycling step
- Fuel is sent directly to a geological repository

Nuclear Fuel Cycle **Partially**

- One recycling step
- Fuel is eventually sent to a geological repository

Nuclear Fuel Cycle

CLOSED

- Some recycling

- All actinides are fissioned to shorter lived fission products

- Requires use of advanced, fast reactor technology
- Harder neutron spectrum

Decay of used nuclear fuel

- Hazard of Nuclear Waste from 1 ton IHM of typical PWR fuel relative to the amount of uranium needed to make 1 ton fuel.
- A hazard index of 100 means the used nuclear fuel is equally hazardous as the natural uranium.

Slide courtesy Professor Mikael Nilsson, UC - Irvine

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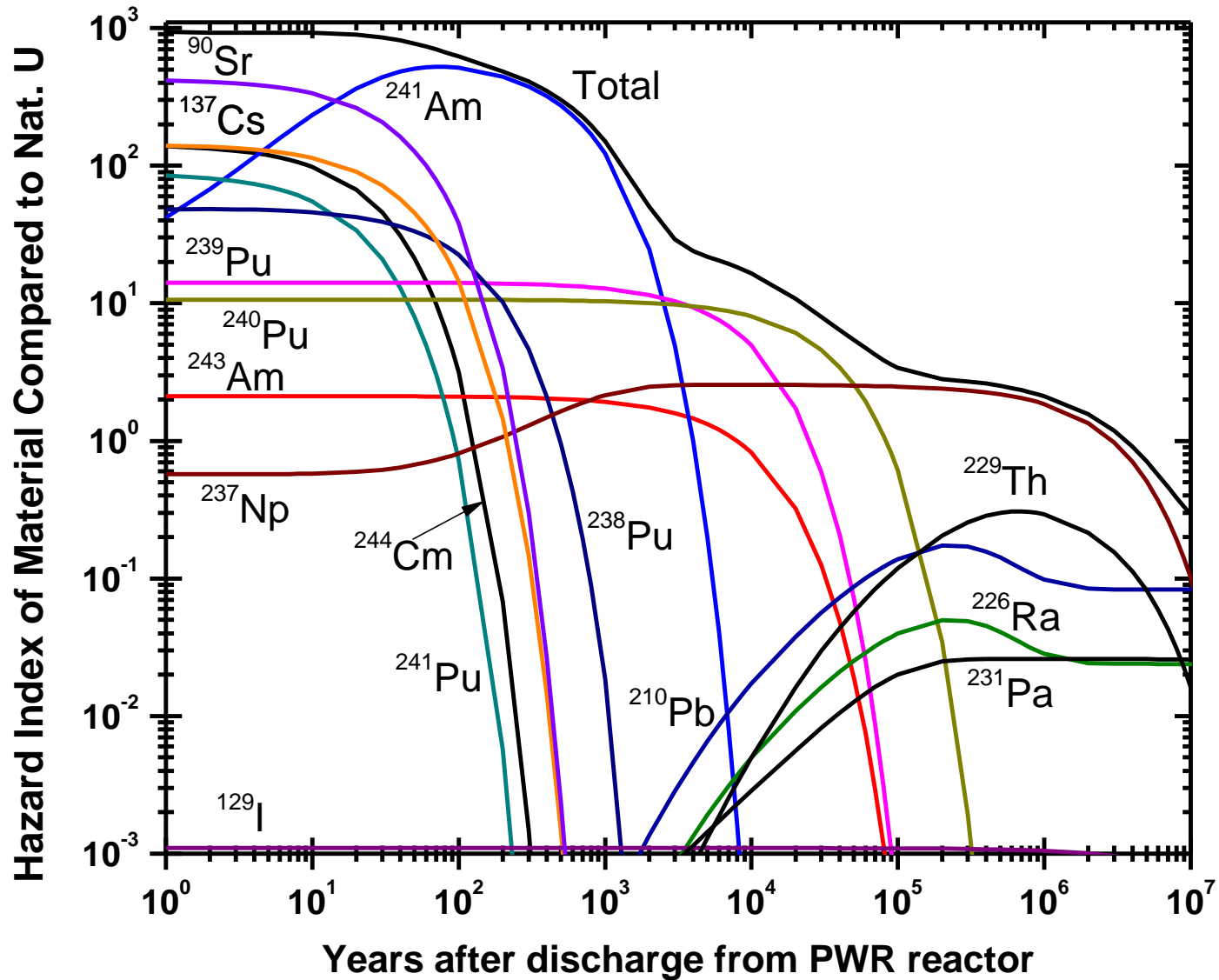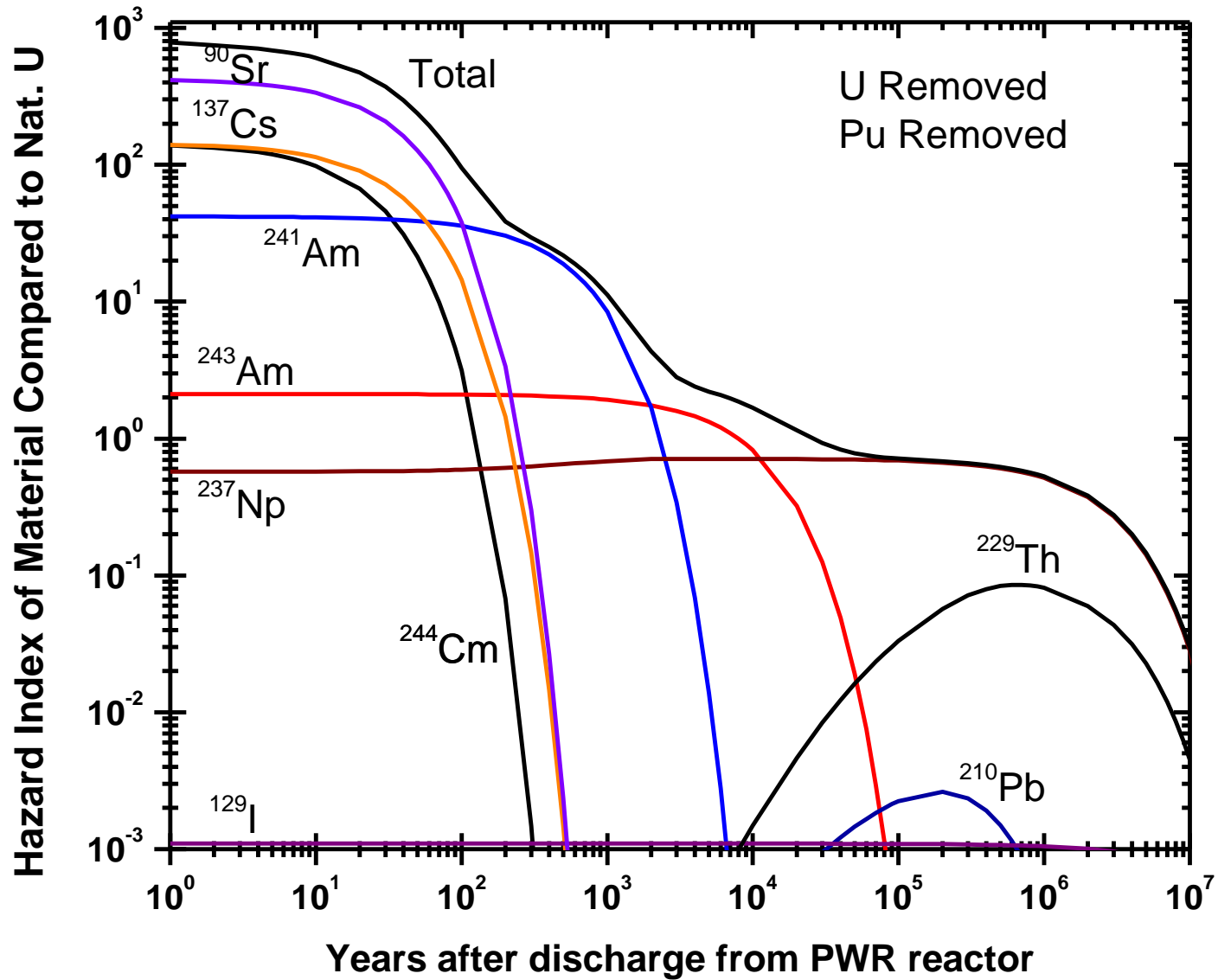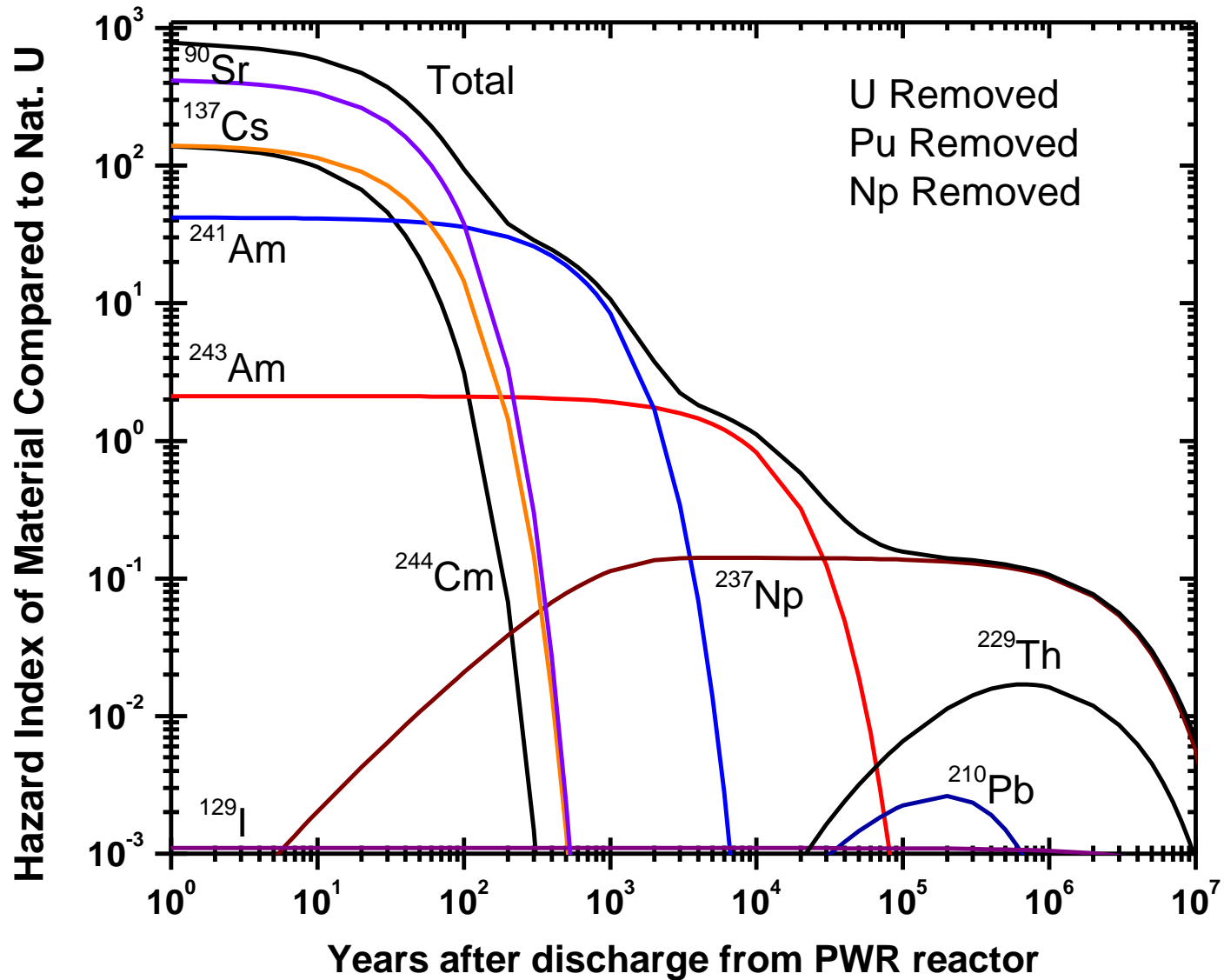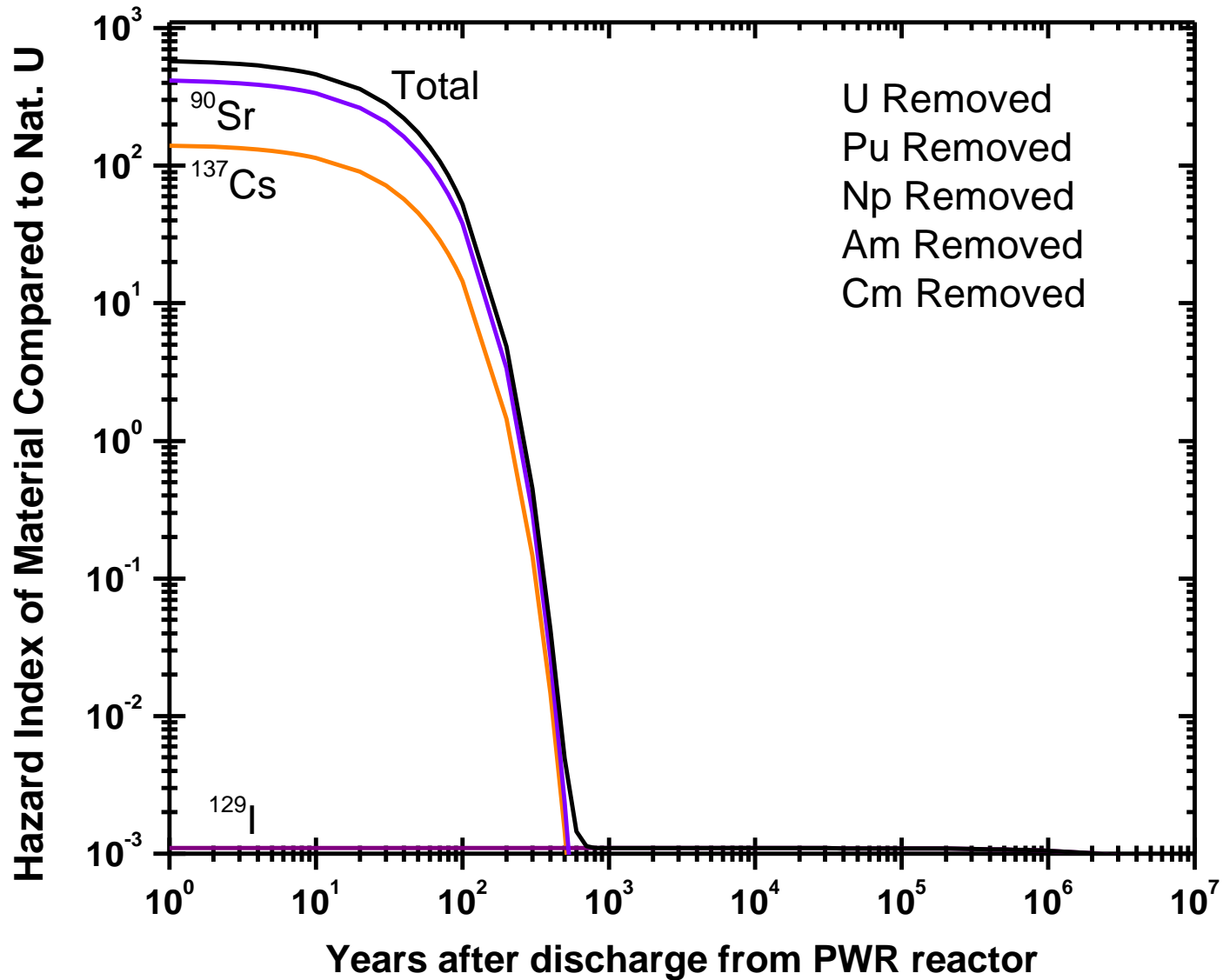
Used Nuclear Fuel in the U.S.
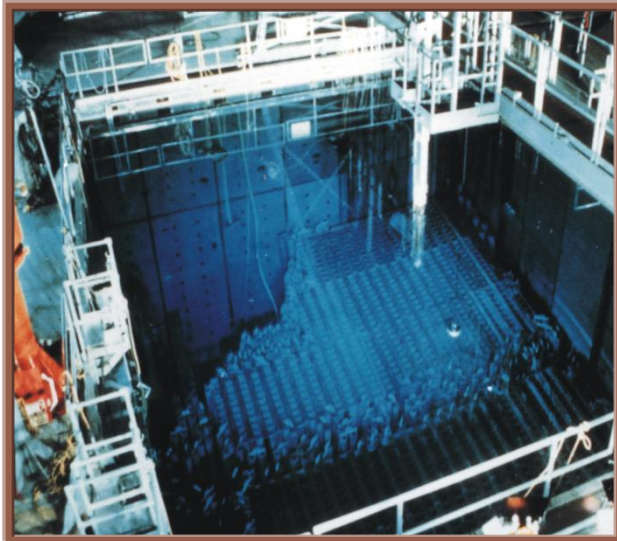
- 72 plant sites with used fuel + DOE sites
- 39 states with used fuel
- 69,500 metric tons of used fuel currently in US
- 135,000 metric tons of used fuel projected by 2035 (excluding new builds)

Key
- Period of Extended Operation
- Recently shut-down plants
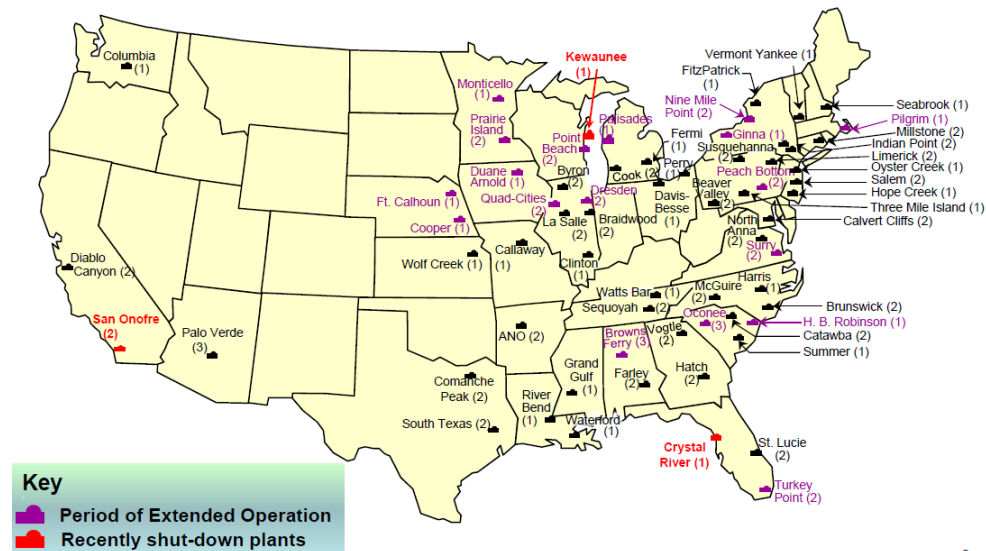
U.S. Fuel Cycle Policy

- The current US approach is a once-through fuel cycle
  - It is likely that most of the ~70,000 MT of current used fuel will be directly disposed in a geologic repository, beginning around mid-century
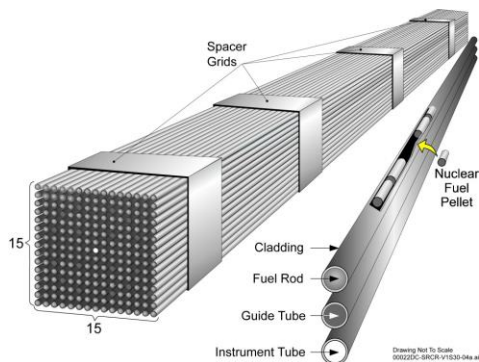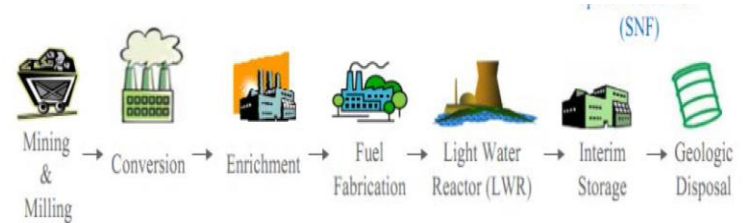
- The US is evaluating advanced (closed) fuel cycles for potential deployment also around mid-century
  - The closed fuel cycle, if implemented, would likely process future generated UNF
  - At current UNF generation rates, 2000 MT/yr, there will be adequate fuel available

- No decisions have been made on future fuel cycles in the US, nor which technologies will be employed, if a closed fuel cycle is selected

Advanced Fuel Cycle Strategies – Why?

Review Slide

The once-through fuel cycle utilizes only about 0.7% of the uranium

■ **Multiple recycle of U, Pu and TRUs in fast thermal reactors can significantly raise uranium utilization**
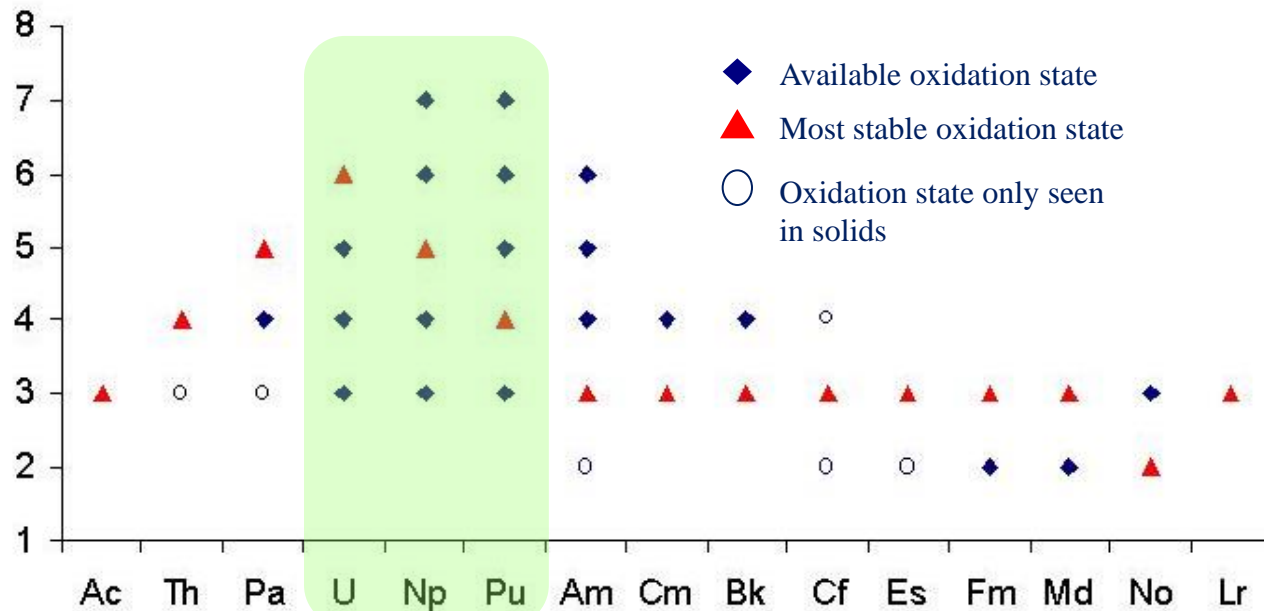

^{241}Am ^{242m}Am ^{243}Am ^{244}Cm ^{245}Cm

^{238}Pu ^{239}Pu ^{240}Pu ^{241}Pu ^{242}Pu ^{237}Np
[Inner core: Pu enrich. 19.6 wt.%]

TRU compositions considered in core design studies , NEA/OECD-7077, 2012

■ **Partitioning and Transmutation of TRU can also improve repository performance**
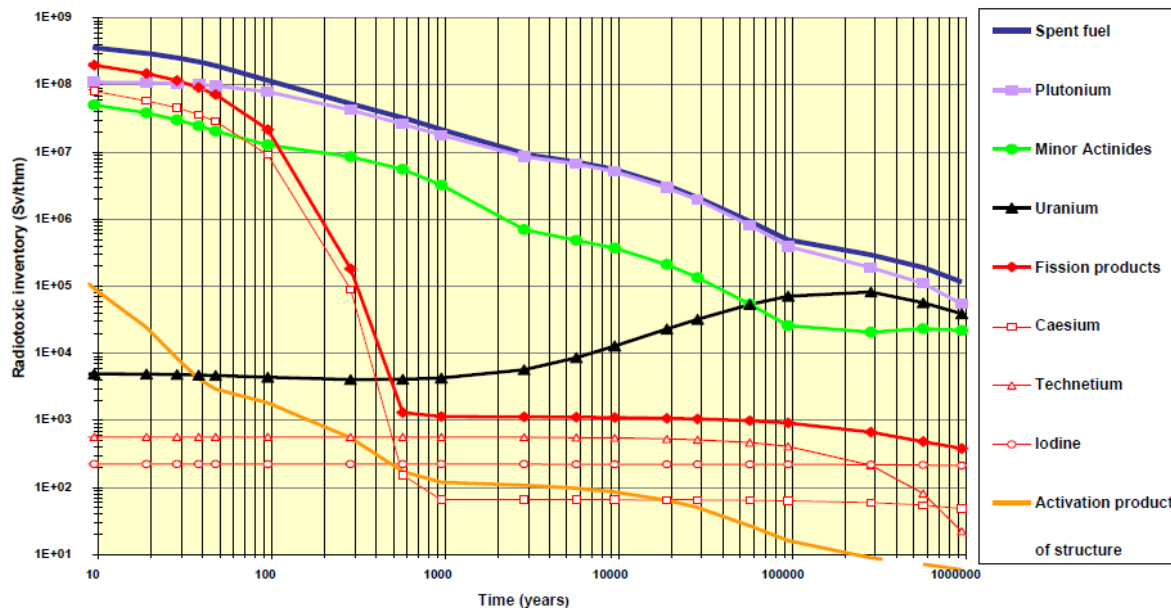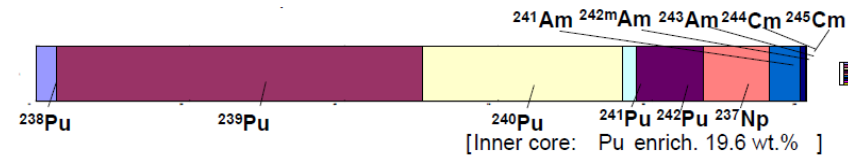
Radiotoxic inventory of UOx used fuel in Sv/ton heavy metal, NEA/OECD-7077, 2012

What is advanced partitioning?

***Advanced partitioning* is loosely defined as the post nuclear fuel irradiation separations steps that enable advanced fuel cycle technology**
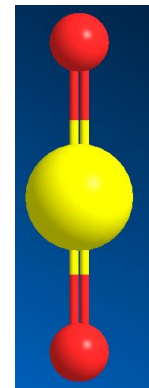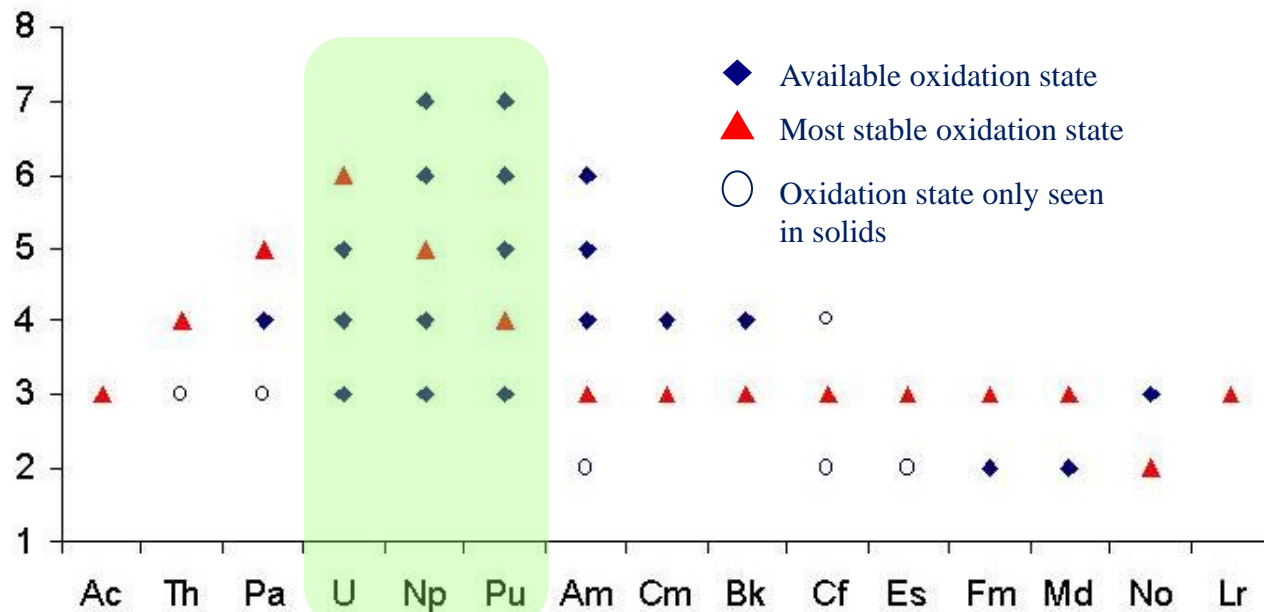
Relevant f-element features

- Early actinides have multiple oxidation states available in solution

Nash, K.L.; Braley, J.C.;Chemistry of Radioactive Materials in the Nuclear Fuel Cycle, (2010) in Advanced separation techniques for nuclear fuel reprocessing and radioactive waste treatment, Woodhead Publishing Series in Energy: Number 2, K.L. Nash, G.L Lumetta Eds., pp 1-22.

Relevant f-element features
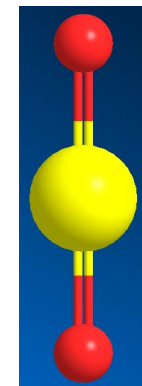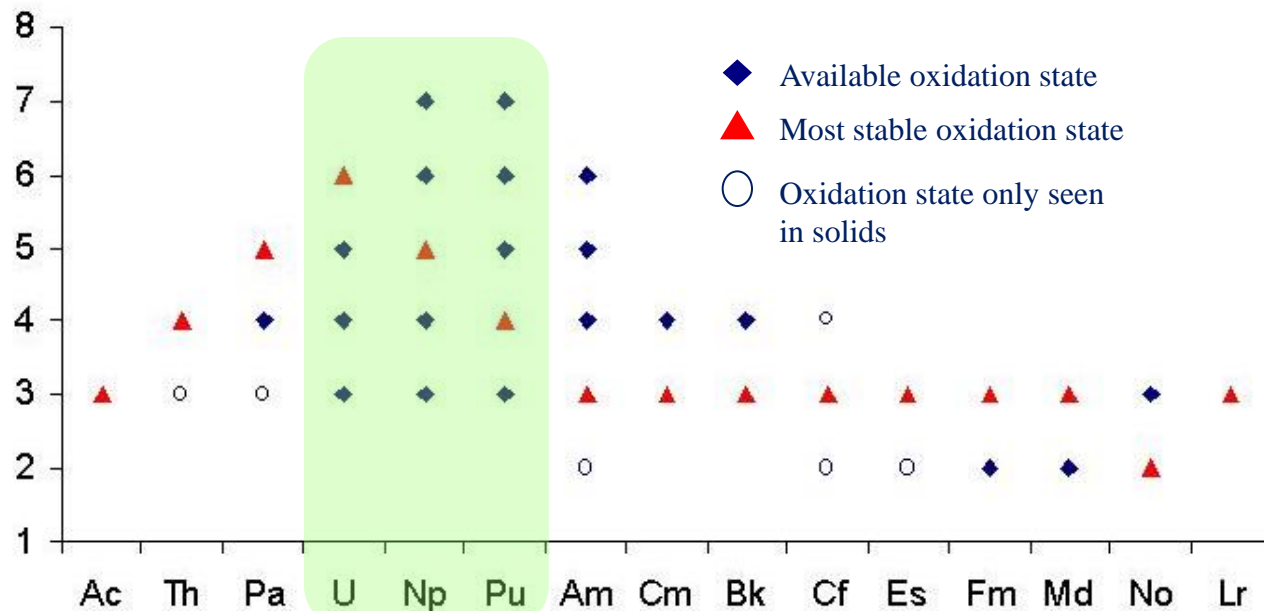
- Early actinides have multiple oxidation states available in solution
- Geometric Restrictions $-An(V)O_2^+$, $An(VI)O_2^{2+}$
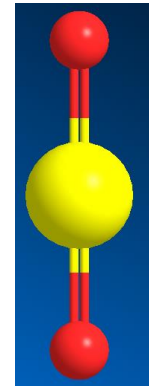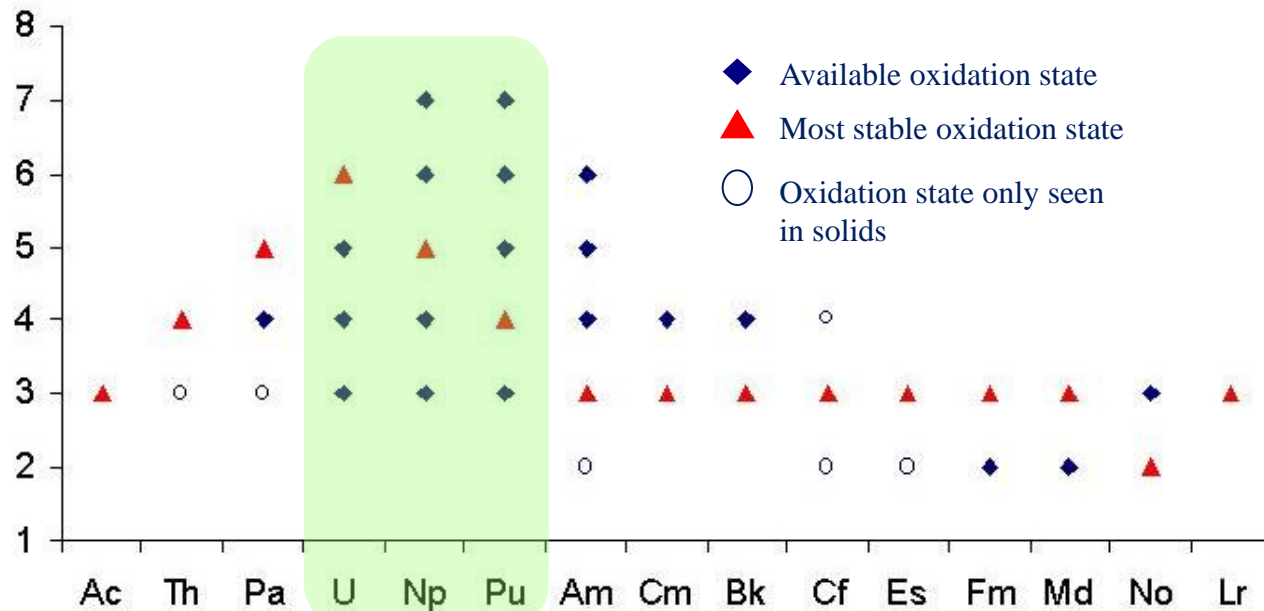
Relevant f-element features

- Early actinides have multiple oxidation states available in solution
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Relevant f-element features
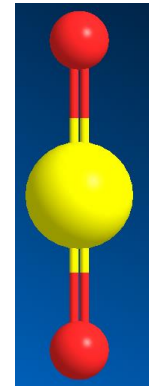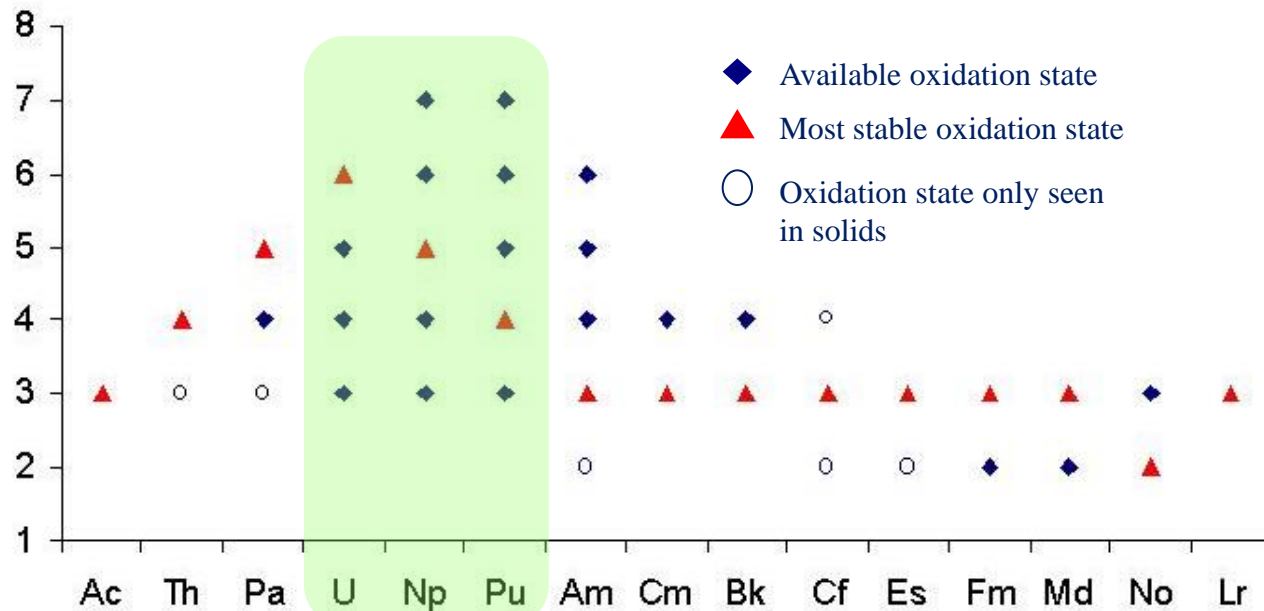
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- Electrostatics – Mostly electrostatic bonding
  – An can participate in some covalent bonding

Relevant f-element features

- Early actinides have multiple oxidation states available in solution
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- Effective Charges – V (2.2) < III (3.0) < VI (3.3) < IV (4)
- Electrostatics – Mostly electrostatic bonding
  - An can participate in some covalent bonding
- Cationic Radii – Decrease by 20% across the series

Challenges for SX processing of used nuclear fuel

- High levels of radioactivity
  - Remote handling and monitoring
  - Degradation

- Acidic / aggressive environment
  - Solvent cleanup
  - Solvent losses
  - Monitoring and adjustment of phases

Nash, K.L.; Braley, J.C.; *Challenges for Actinide Separations in Advanced Nuclear Fuel Cycles*, (2010) in Nuclear Energy and the Environment, ACS Symposium Series volume 1046, C. M. Wai, B, J. Mincher Eds, pp. 19-38.

An(III)/Ln(III) Separation

- Reasons for An(III)/Ln(III) Separation:
  - 1/3 of FP are Ln(III)
  - An(III) : Ln(III) = 1:60
  - Large neutron capture cross sections
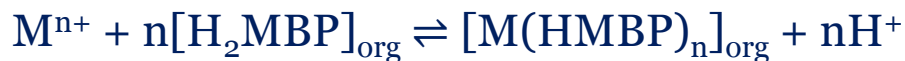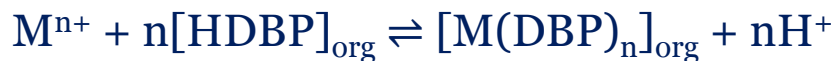 - Chemically and physically very similar

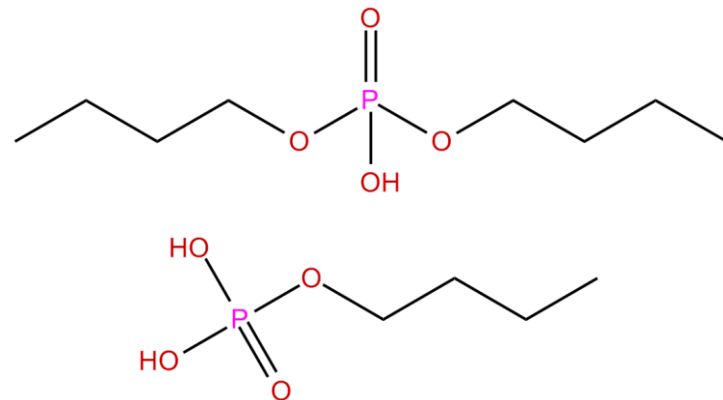# Extraction Mechanisms

Solvating Mechanism

$$M^{n+} + nNO_3^- + 2[TBP]_{org} \rightleftharpoons [M(NO_3)n(TBP)_2]_{org}$$
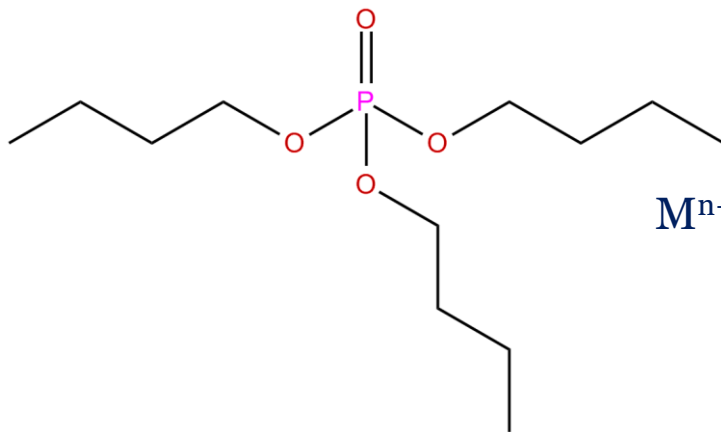
Extraction – high nitric acid
Stripping – low nitric acid

Cation Exchange Mechanism

$$M^{n+} + n[HDBP]_{org} \rightleftharpoons [M(DBP)_n]_{org} + nH^+$$

$$M^{n+} + n[H_2MBP]_{org} \rightleftharpoons [M(HMBP)_n]_{org} + nH^+$$

Extraction – low acid
Stripping – high acid

The recycle challenge is in separating minor actinides!

- Separating Am/Cm from lanthanides is a difficult challenge
  - Most partitioning and transmutation schemes include separation of either americium alone or americium and curium together
  - Chemistry of Am & Cm are very similar to lanthanides
    - Most lanthanides are trivalent, Cm is trivalent and Am is most stable as Am(III)
    - Slight differences in bonding characteristics (5f softer than 4f)
  - Several methods have been used to separate Am/Cm from lanthanides in lab scale studies or applications, but there has yet to be a large-scale demonstration of any technology
    - Most methods utilize extractants with limited radiolytic stability, extraction conditions at relatively high pH (3-4), and/or very complex chemistry involving lipophilic extractants and aqueous soluble complexants (plus buffers!)
- A number of technologies have been developed and tested, but not deployed…
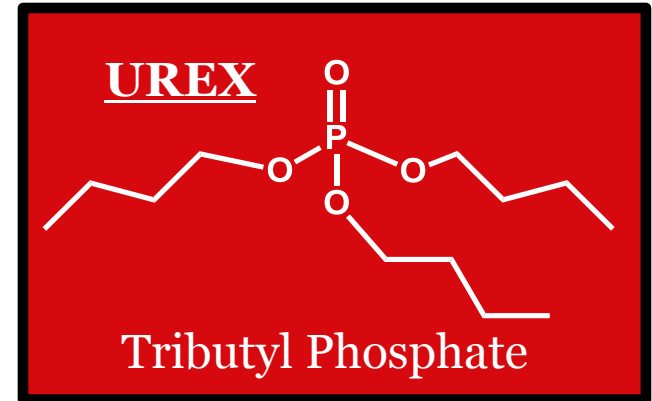- Separating Am alone (from Cm and Ln) is an even greater challenge

Slide courtesy Dr. Bruce Moyer, ORNL

Sigma Team for Minor Actinide Separations

Feed types: *Dissolver solution; UREX (CoEX) raffinate; or TRUEX (DIAMEX) strip solution*
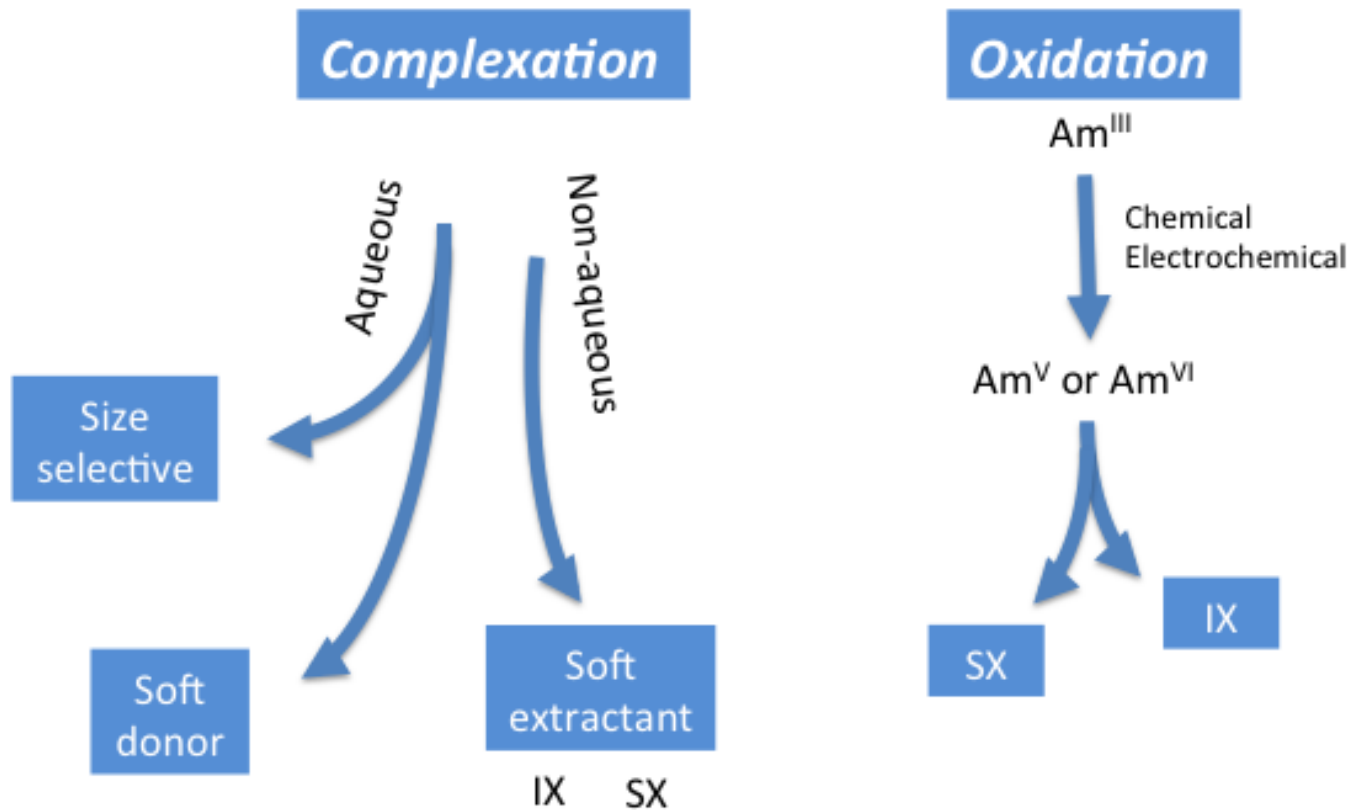
Complexation

Aqueous

Non-aqueous

Size selective

Soft donor

Soft extractant

IX   SX

Oxidation

Am^{III}

Chemical
Electrochemical

Am^V or Am^{VI}

SX

IX

Recent U.S. approaches

Global Nuclear Energy Partnership

URanium EXtraction

UREX → U, Tc

UREX

Tributyl Phosphate

- Selective recovery of U and Tc

- Redox chemistry of the aqueous phase can be manipulated to adjust actinide recovery

- Rest of fission products and actinides continue to the next separation step

Recent U.S. approaches

Global Nuclear Energy Partnership

URanium EXtraction

| UREX | → | U |
| | → | Tc |

Extractants Used

| FPEX | → | Cs/Sr |
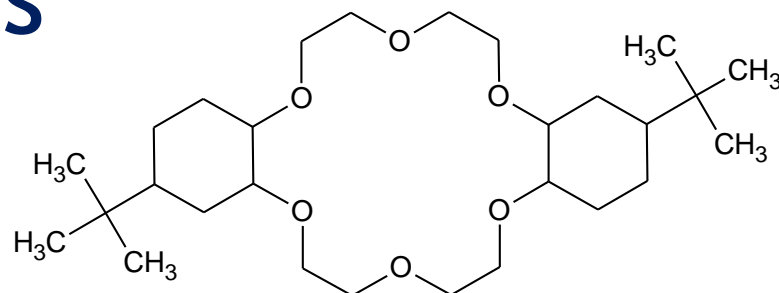
DtBuCH18C6
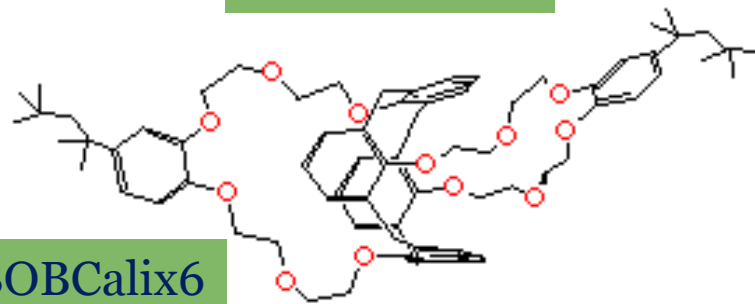
BOBCalix6

- Selective separation of Cs and Sr from the rest of the fission products and actinides.

- Based on highly specific extracting agents
  - 4,4',(5')-Di-(t-butyldicyclo-hexano)-18-crown-6
    - DtBuCH18C6 for Sr extraction
  - Calix[4]arene-bis-(tert-octobenzo-crown-6)
    - BOBCalix6 for Cs extraction

Recent U.S. approaches

Global Nuclear Energy Partnership

URanium EXtraction → **UREX** → U, Tc

Extractants Used → **FPEX** → Cs/Sr

TRansUranium EXtraction → **TRUEX** → Non-RE FPs

TRUEX

C_8H_{17}
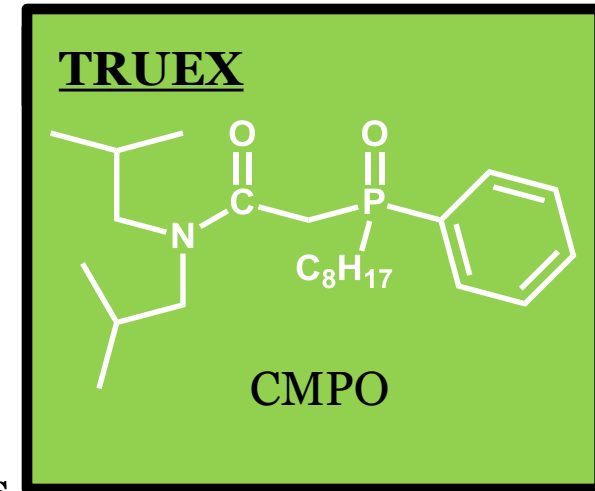
CMPO

- Remaining transuranics, Am^{3+}, Cm^{3+} are extracted together with the lanthanides

- The extraction reagent used is octyl(phenyl)-N,N-diisobutylcarboylmethyl-phosphine oxide (CMPO) with some TBP added

Recent U.S. approaches

Global Nuclear Energy Partnership

URanium EXtraction → **UREX** → U, Tc

Extractants Used → **CCD-PEG** → Cs/Sr

TRansUranium EXtraction → **TRUEX** → Non-RE FPs

TALSPEAK → Ln(III)
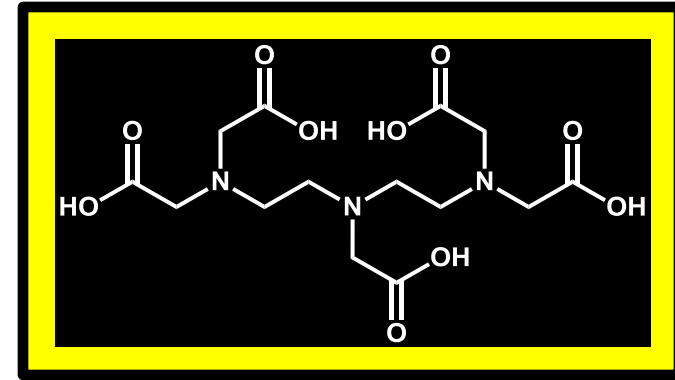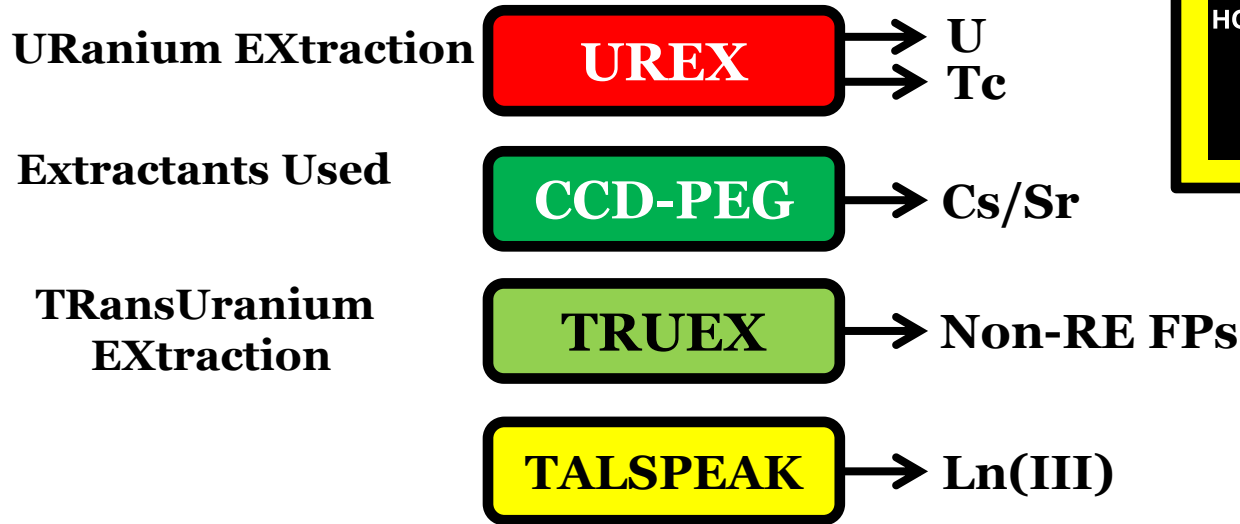
DTPA
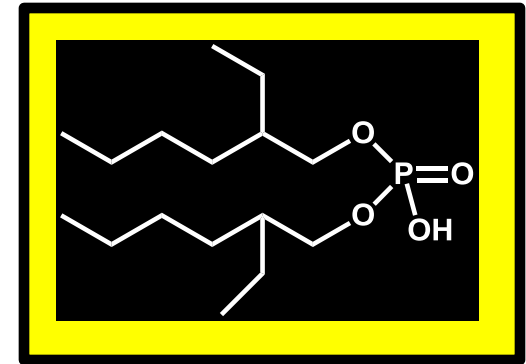
HDEHP

- **T**rivalent **A**ctinide **L**anthanide **S**eparation by **P**hosphrous **E**xtractants and **A**queous **K**omplexants

- Am and Cm are selectively held back in the aqueous phase while all the lanthanides are extracted

- Combination of organic extracting reagents and aqueous complexing agents increase the complexity of the system.

Recent U.S. approaches

Global Nuclear Energy Partnership

Review Slide

- UREX chemistry supports the separations requirement of recovering uranium and potentially other actinides
- FPEX chemistry was eventually decided to be too complex while providing too little benefit
  - Was dropped
- TALSPEAK chemistry is very complex and had several unknown equilibria
  - This will be considered later in the webinar
  - Advanced TALSPEAK was developed to address these issues
- To compress fuel cycle, combination of TALSPEAK with TRUEX (TRUSPEAK process), and other modifications, have been considered
  - Advanced TRUSPEAK and ALSEP
- An ideal process would potentially remove all of the actinides (U-Am) in one or two recycling
  - Would probably require use of hexavalent Am

TALSPEAK

Trivalent Actinide Lanthanide Separation Using Aqueous Komplexants
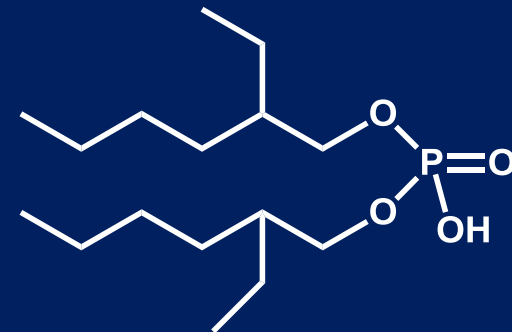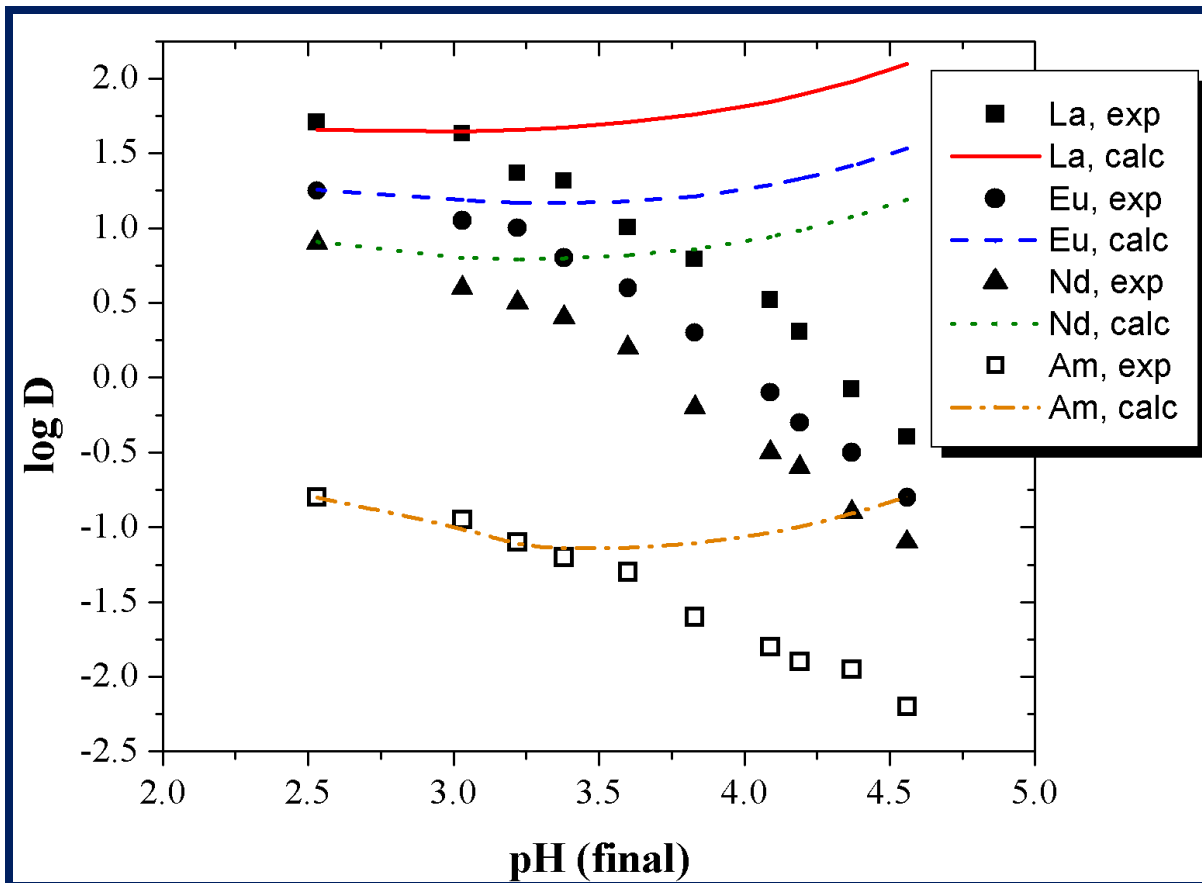
Aqueous
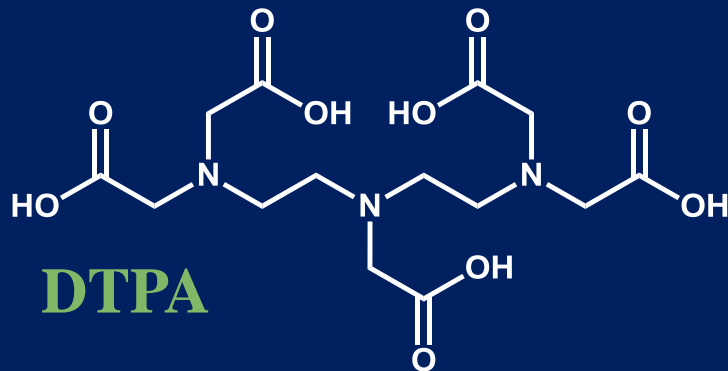
DTPA
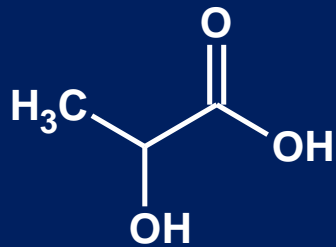
Lactic Acid

Am/Cm

Organic

Lanthanides

HDEHP

Unexplained Chemistry

$$D = \frac{\overline{[M(AHA)_3]}}{[M^{3+}] + [ML^{2+}] + [ML_2^{+}] + [ML_3] + [MR^{2-}] + [MHR^{-}]}$$
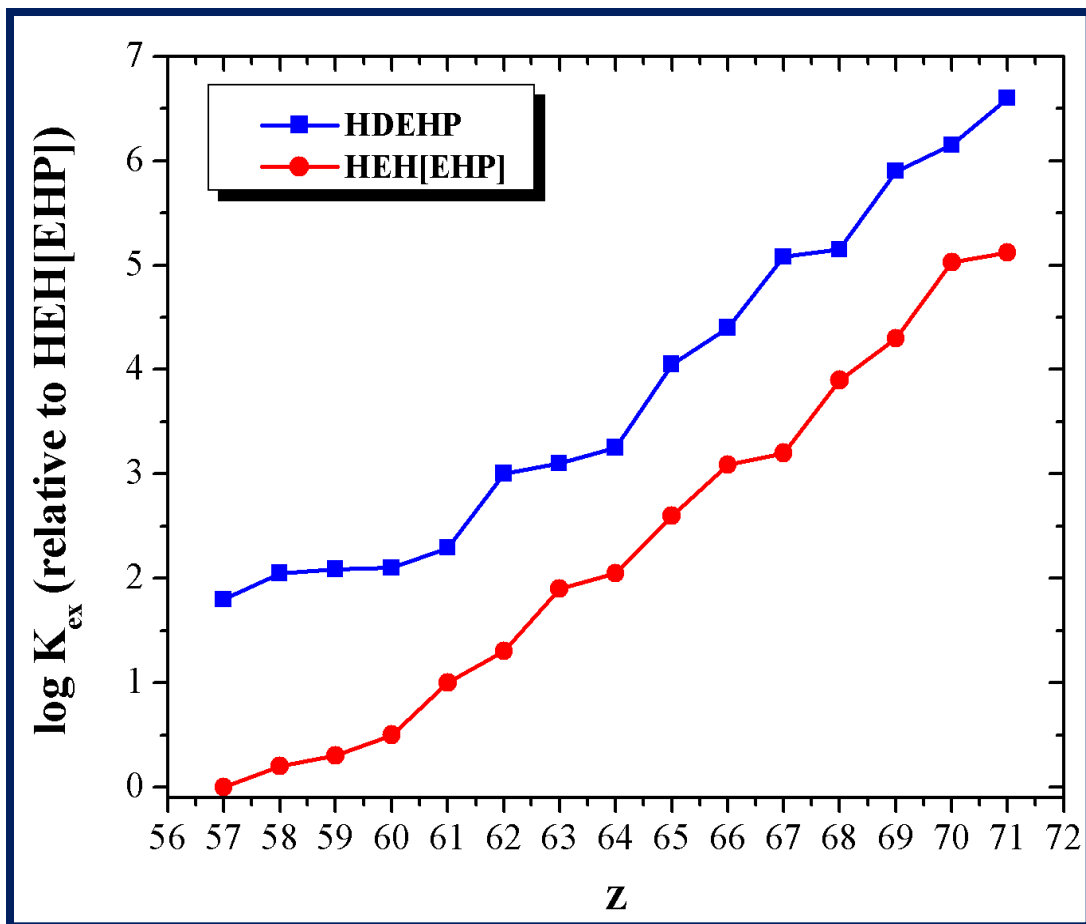

- Ternary complex formation
  - $M(Lac)(DTPA)^{-}$
  - $M(AHA)2(Lac)$
- Partitioning of HDEHP to aqueous phase
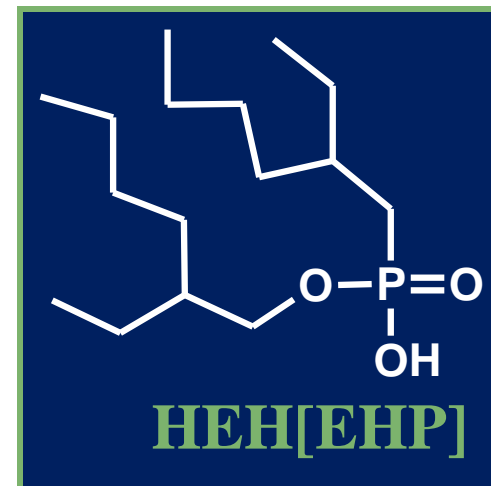- Sodium extraction
- Activity effects

Nash, K.L. Solvent Extr. Ion Exch. 2015, 33(1), 1-55.
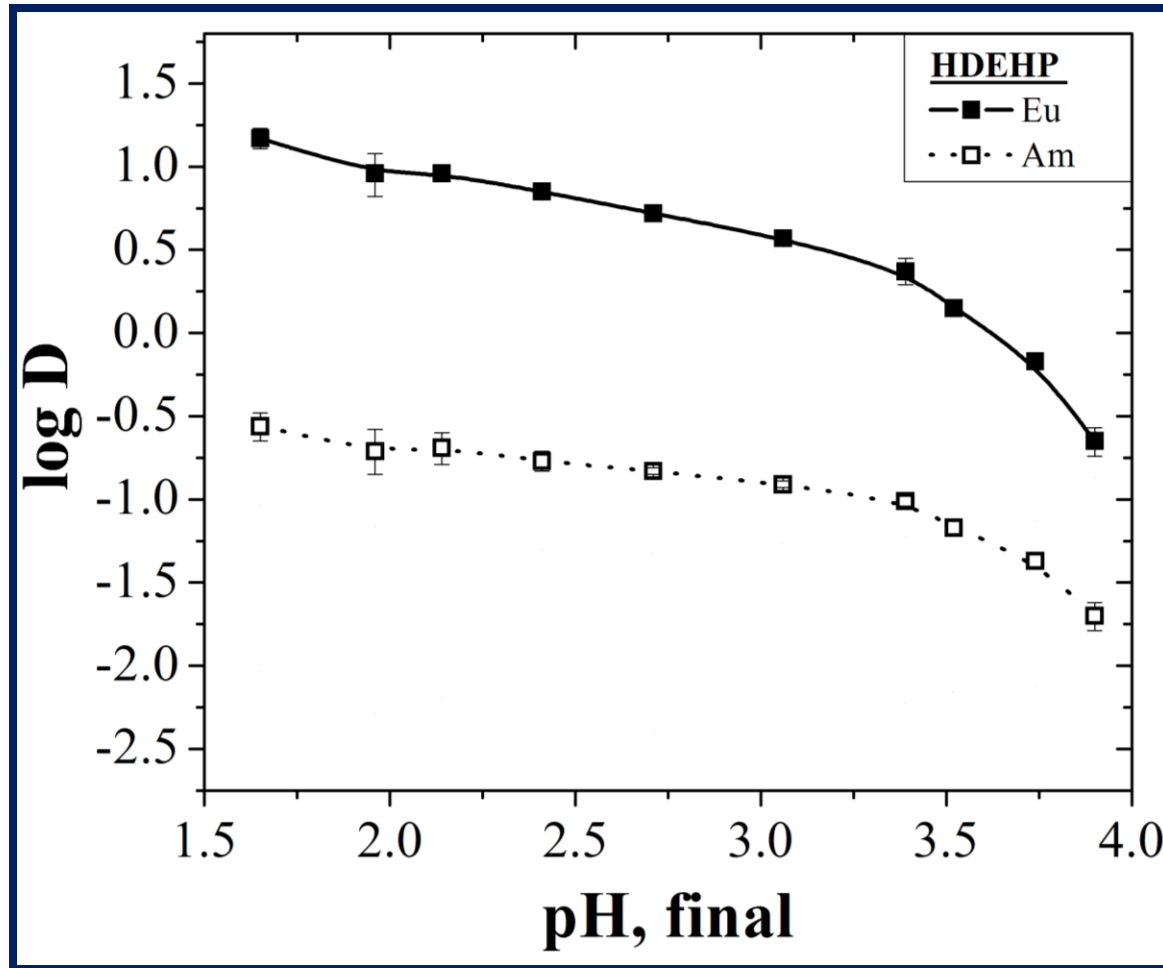Nilsson, M.; Nash, K.L. Solvent Extr. Ion Exch. 2007, 25, 665-710.

Insertion of HEH[EHP]

	HDEHP	HEH[EHP]
$\log pK_a$	3.47^1	4.51^1
$\log K_2$	3.68^2	3.37^2
$\log K_{ex}^{Na}$	-3.56^3	-5.46^4

HEH[EHP]

Braley, J.C.; Grimes, T.S.; Nash, K.L., Ind. Eng. Chem. Res., 2012, 51(2) 629−638.
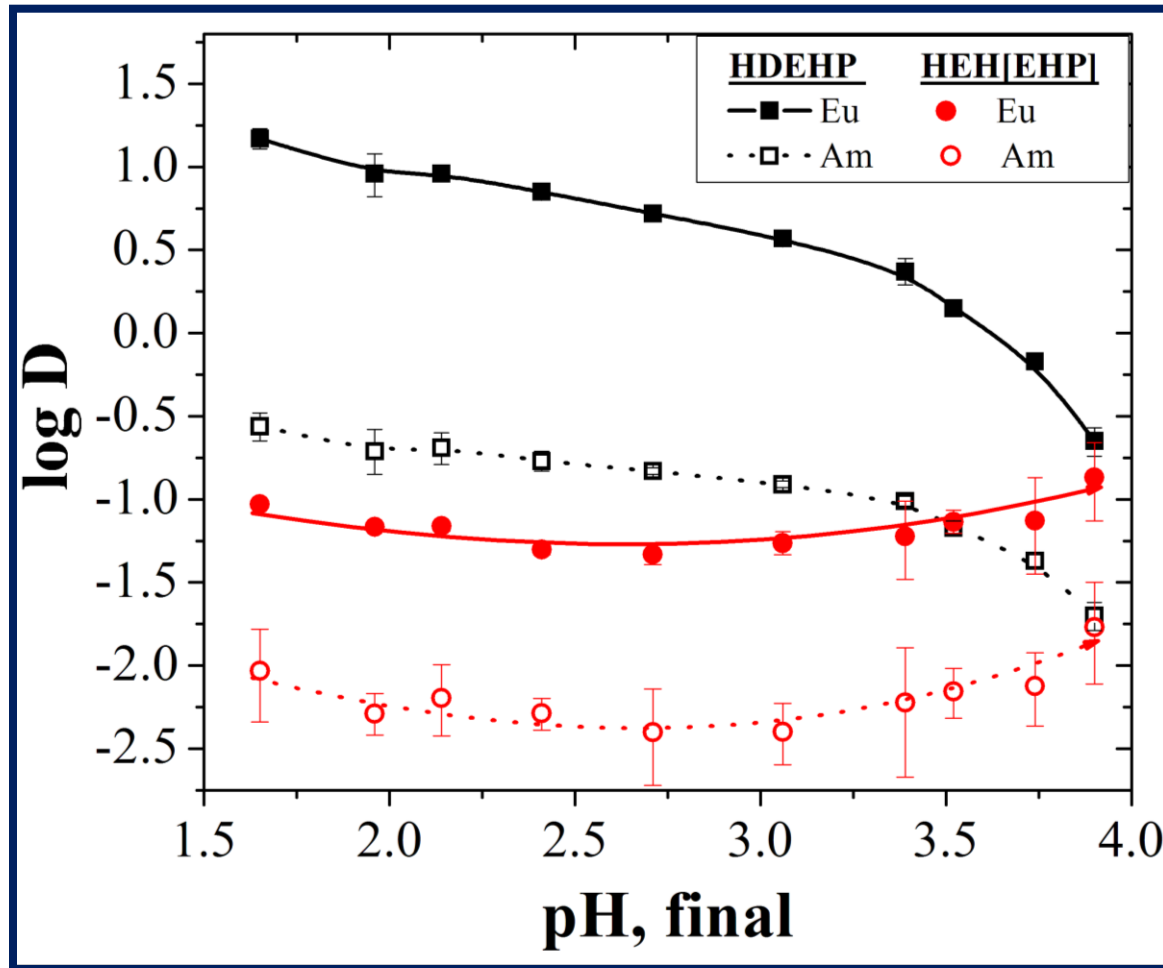
HDEHP vs HEH[EHP]

HEH[EHP]
- Consistent Am/Eu separation, regardless of pH
- Comparable behavior to predicted thermodynamic model

Organic: 0.10 M HDEHP or HEH[EHP]
Aqueous: 20 mM DTPA, 1.0 M NO_3^-, 1.0 M total lactic acid

HDEHP vs HEH[EHP]

HEH[EHP]
- Consistent Am/Eu separation, regardless of pH
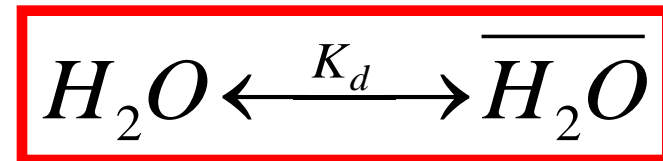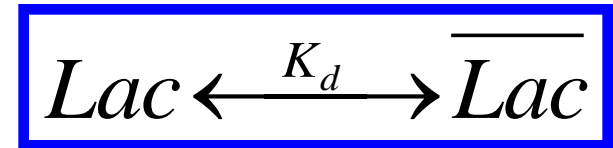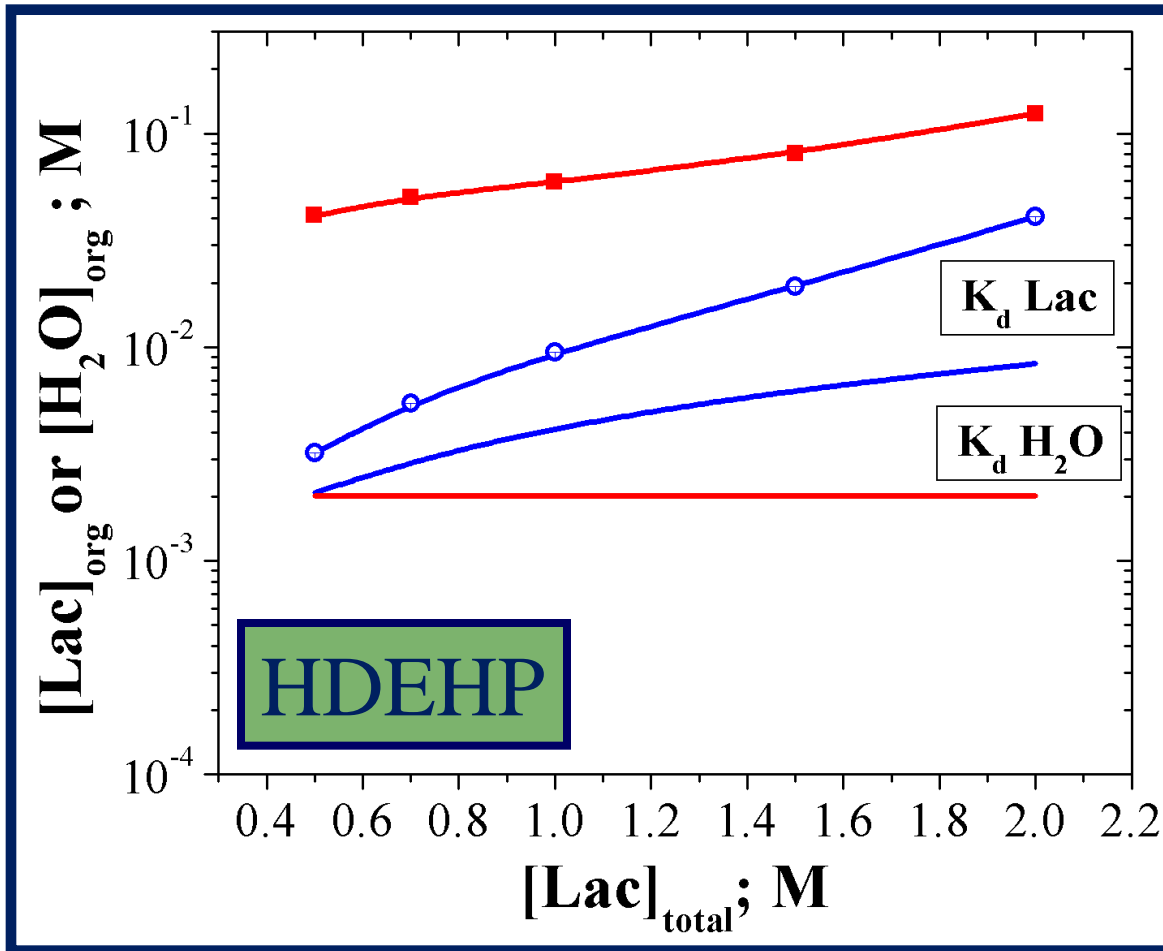- Comparable behavior to predicted thermodynamic model

Organic: 0.10 M HDEHP or HEH[EHP]
Aqueous: 20 mM DTPA, 1.0 M NO_3^-, 1.0 M total lactic acid

Lactate & Water Partitioning

$$Lac \xleftarrow{\quad K_d \quad} \overline{Lac}$$

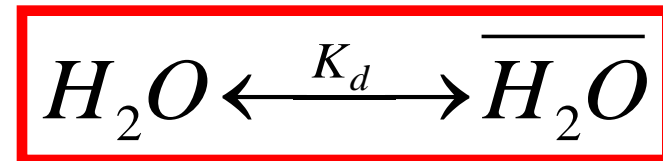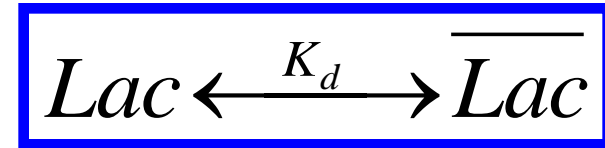$$H_2O \xleftarrow{\quad K_d \quad} \overline{H_2O}$$
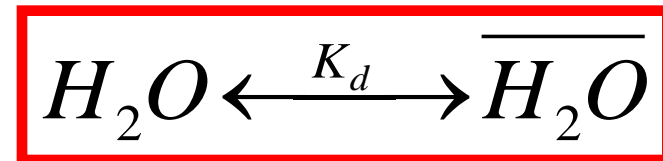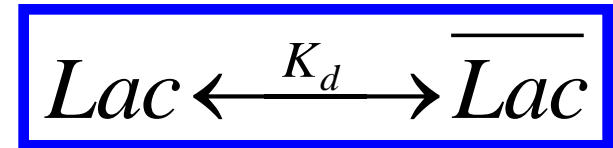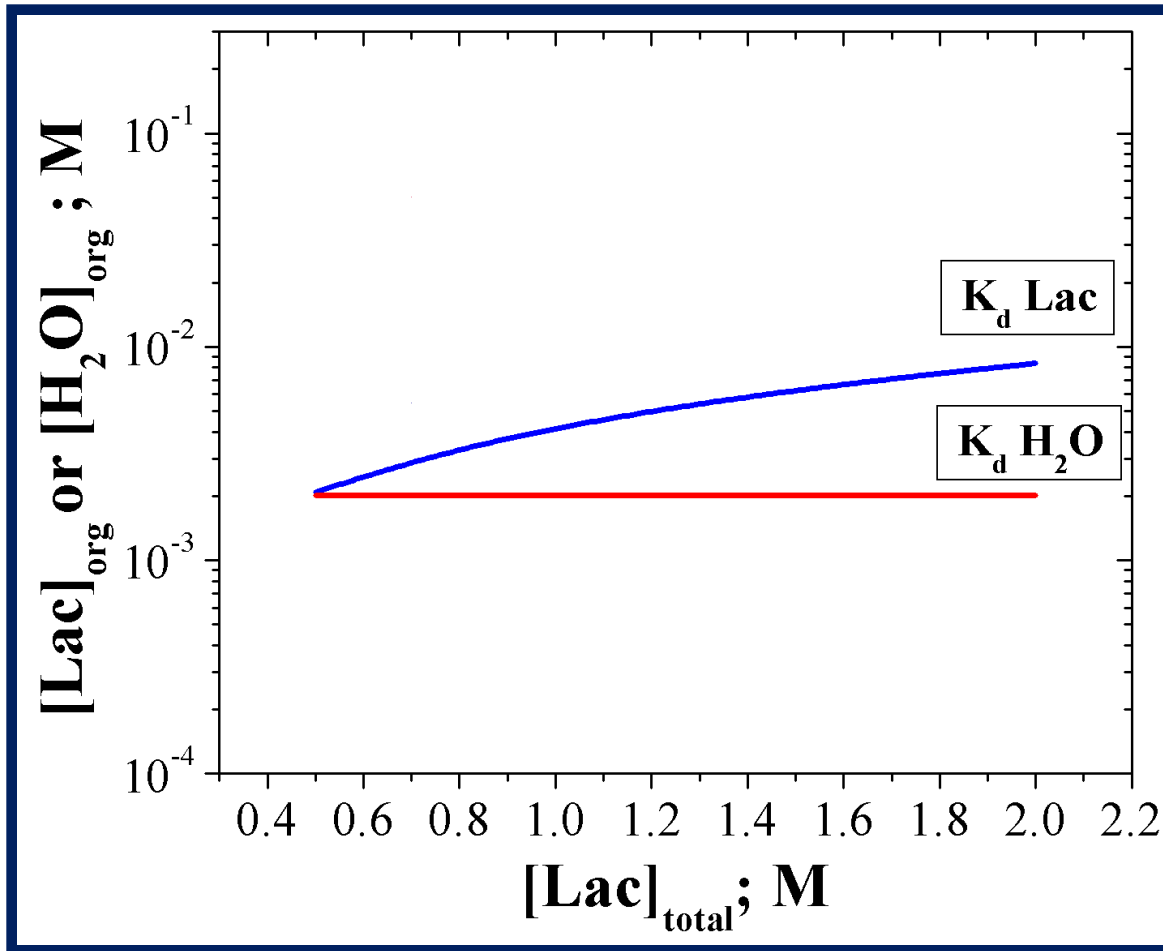
Lactate & Water Partitioning
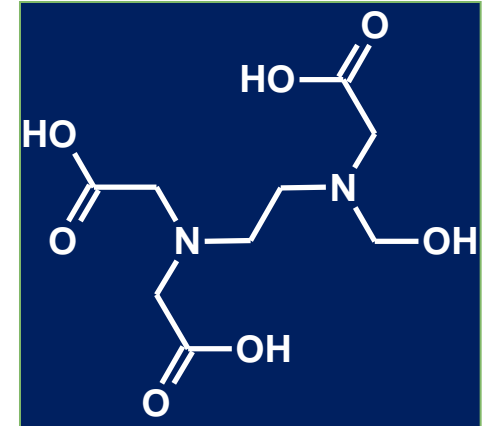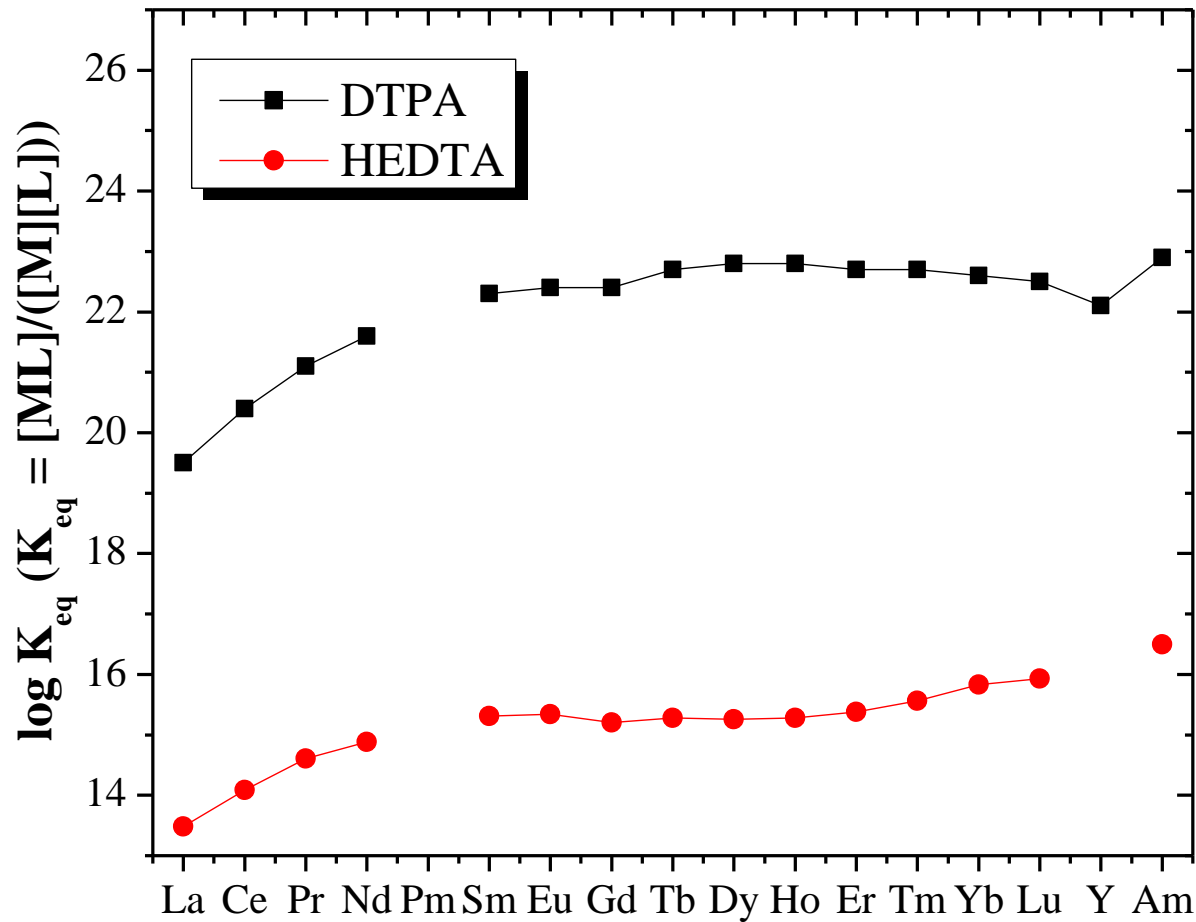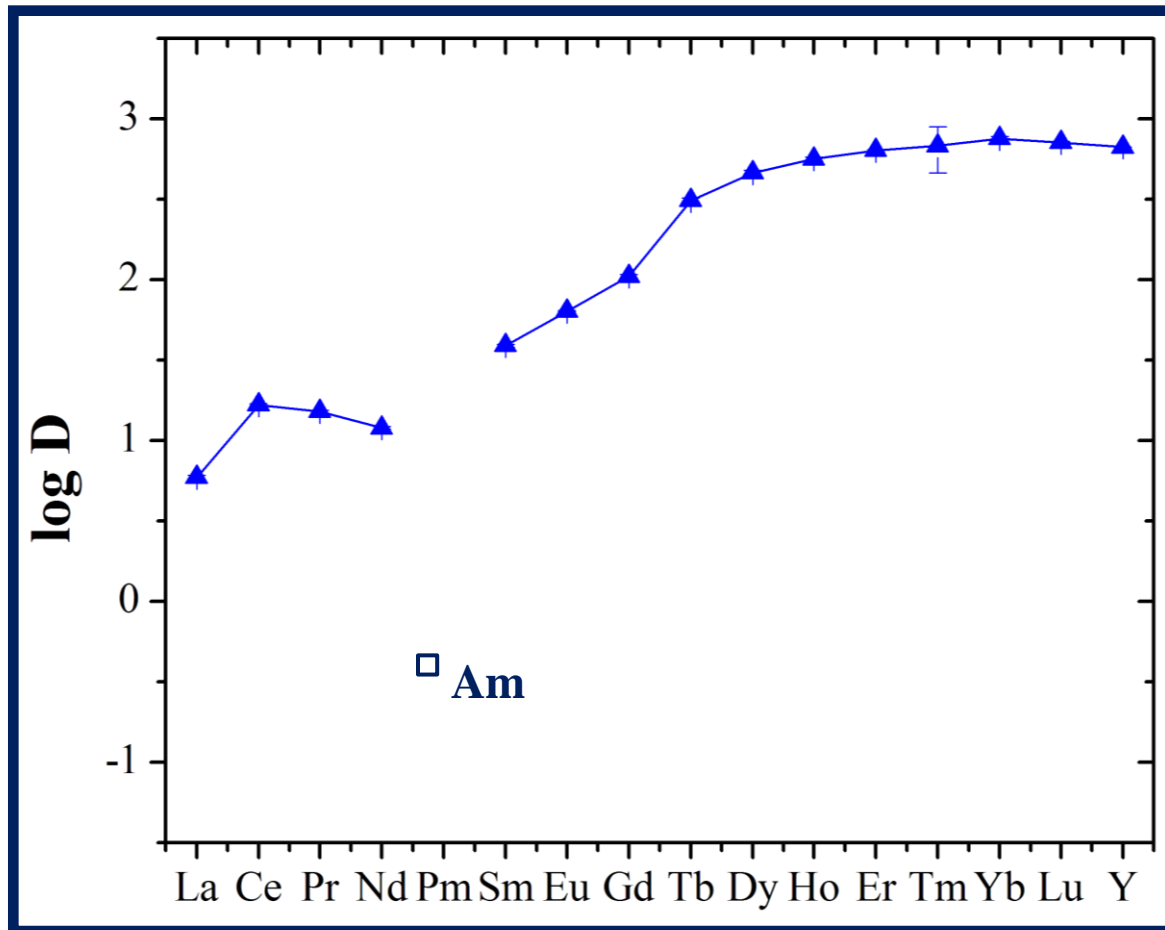
Lactate & Water Partitioning
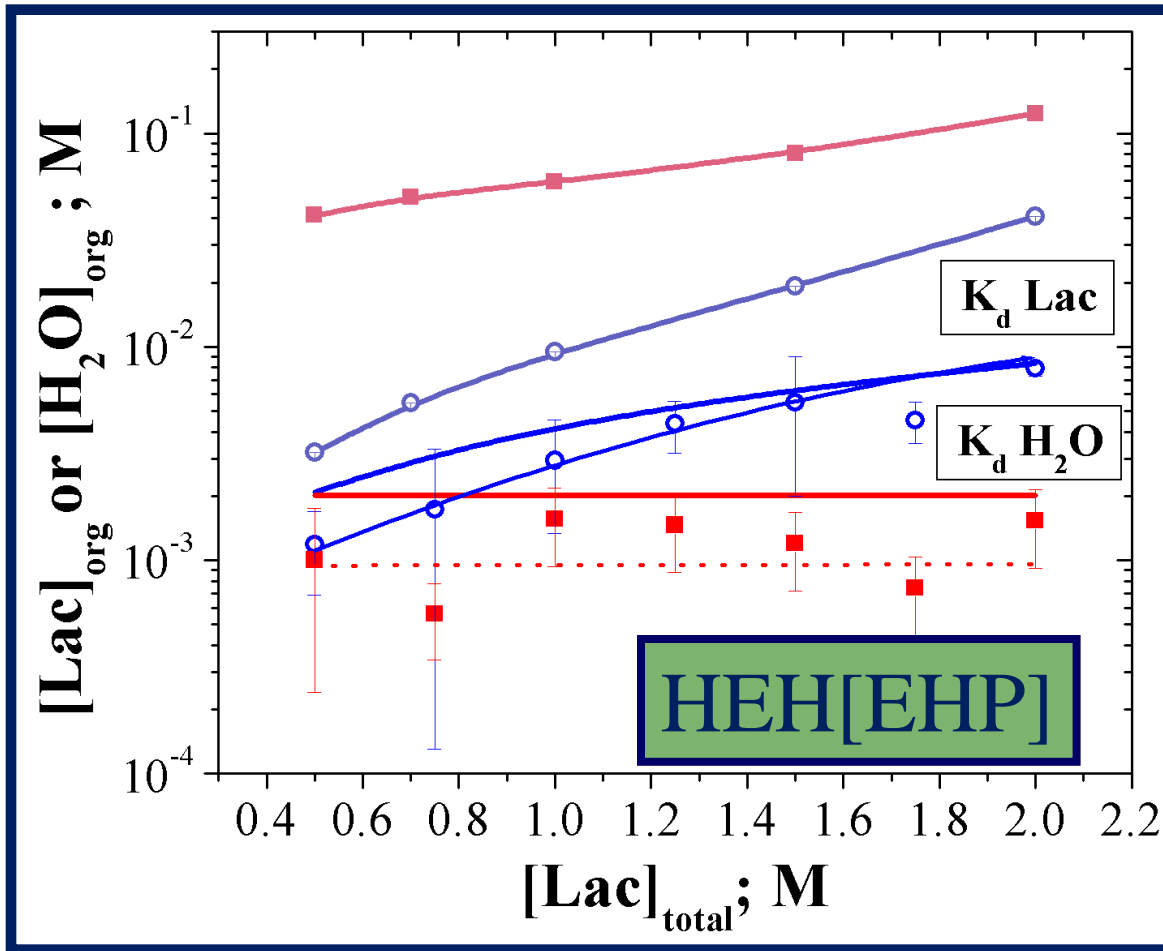
HEH[EHP]/HEDTA-based TALSPEAK

- HEDTA may be more compatible with HEH[EHP] for TALSPEAK purposes
- Changing composition of aqueous phase may improve kinetic issues

trans-Lanthanide Partitioning

- Reasonable separation between light lanthanides and Am

- Lanthanum behavior slightly unanticipated
  - $SF_{La/Am} \sim 40$

Advanced TALSPEAK

TRUSPEAK

CMPO   HDEHP   DTPA
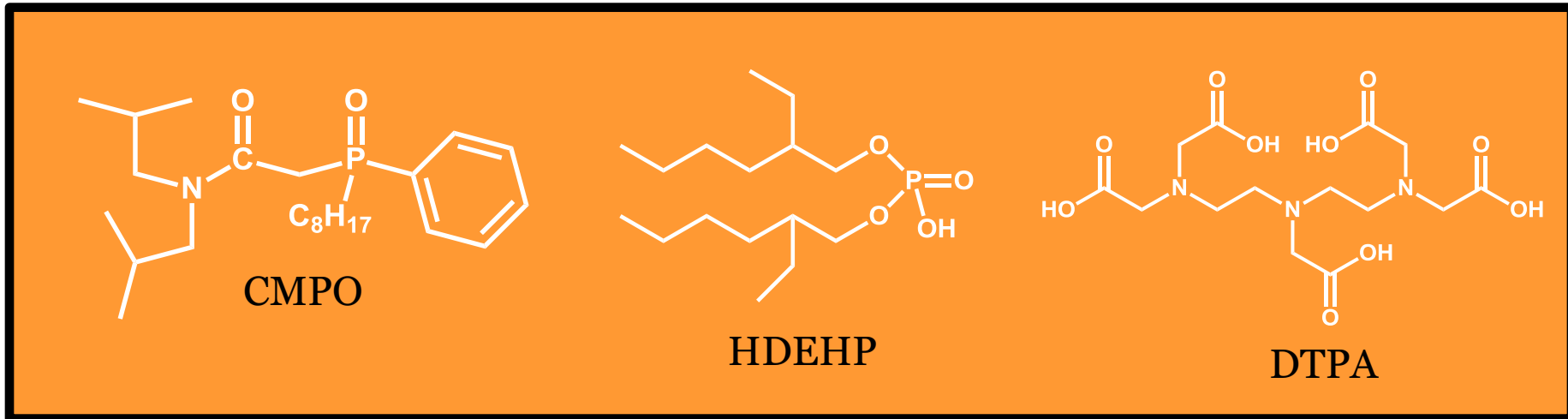
- Combines trivalent f-element recovery properties of TRUEX with the actinide selectivity of TALSPEAK

- Extraction of f-elements occurs under high acid conditions (ph < 0), selective recovery of actinides occurs at higher (pH ~ 3) conditions

Lumetta, G.J.; Gelis, A.V.; Vandegrift, G.F., Solvent Extr. Ion Exch. 2010, 28(3), 287-312
Lumetta, G.J.; Gelis, A.V.; Braley, J.C.; Carter, J.C.; Pittman, J.W.; Warner, M.G.; Vandegrift, G.F., Solvent Ext. Ion Exch. 2013, 31(3), 223-236.

TRUSPEAK - Synergism

Chart parameters:
[CMPO] + [(HDEHP)$_2$] = 0.2 M
0.05 M DTPA/1.5 M lactate/pH 3.6
Am at tracer level
Initial [Nd] = 0.005 M
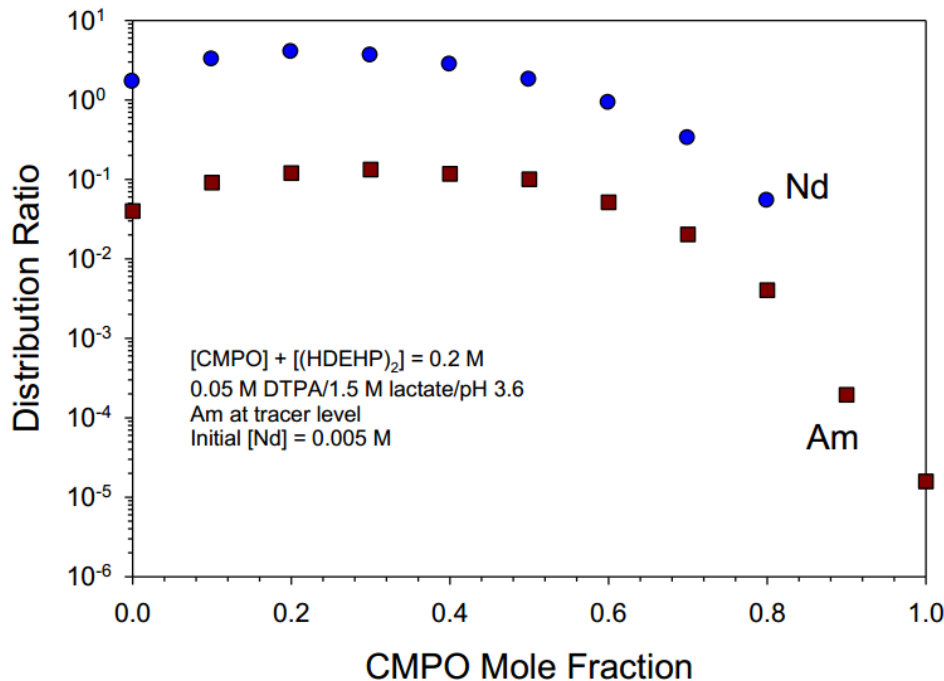
Three mechanisms generally encourage synergism

1. Opening of chelate ring and insertion of solvating extractant
2. Removal of residual water from the unsaturated extracted complex
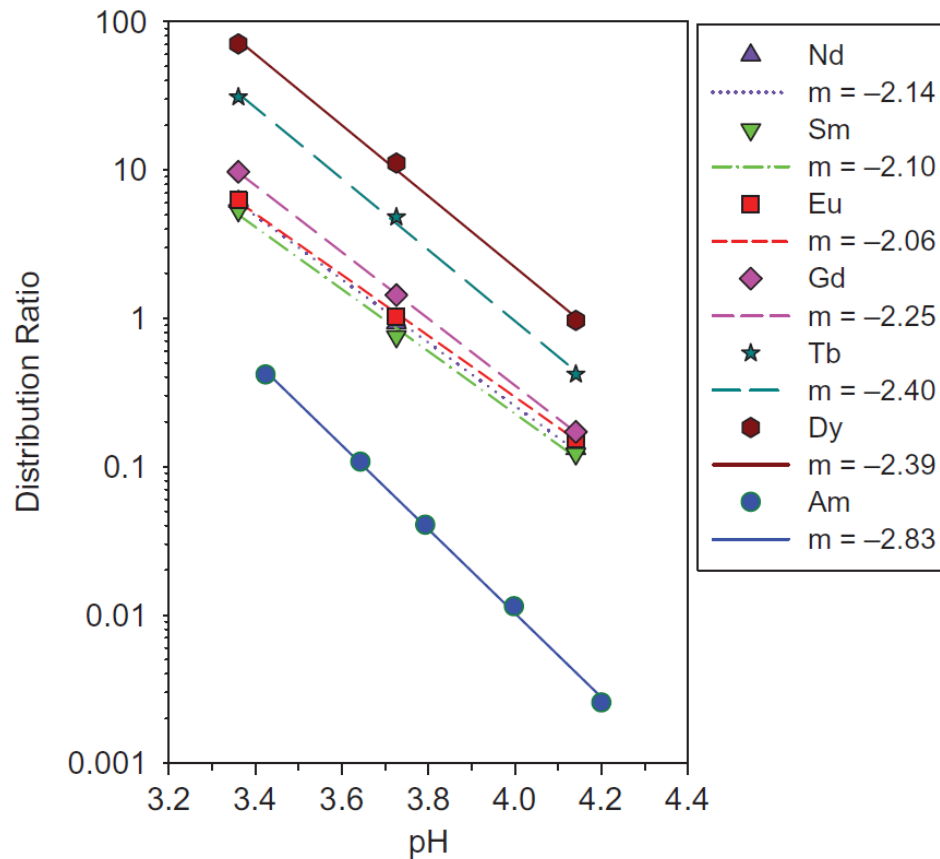3. Expansion of the coordination sphere

Synergism frequently occurs when a cation exchange and solvating extractant are present in the same organic phase

TRUSPEAK – Other aspects

Stage	Zr Distribution
Extraction	3.1×10^4
Scrub	2.2×10^4
Am Strip 1	223
Am Strip 2	245
Am Strip 3	172
Ln/Zr Strip 1	98.9
Ln/Zr Strip 2	0.67
Ln/Zr Strip 3	0.41

- Steep pH dependence (analogous to TALSPEAK)
- TEDGA (N,N,N'N'-tetraethyldiglocolymide) recovers Zr

Advanced TRUSPEAK
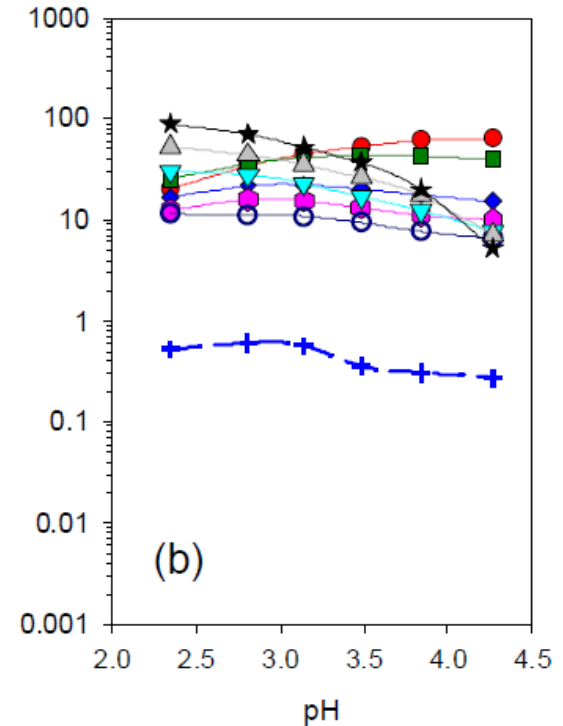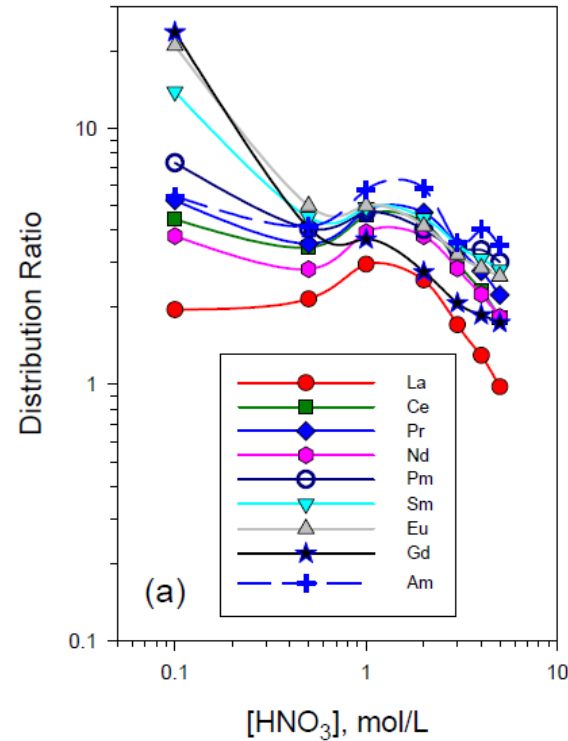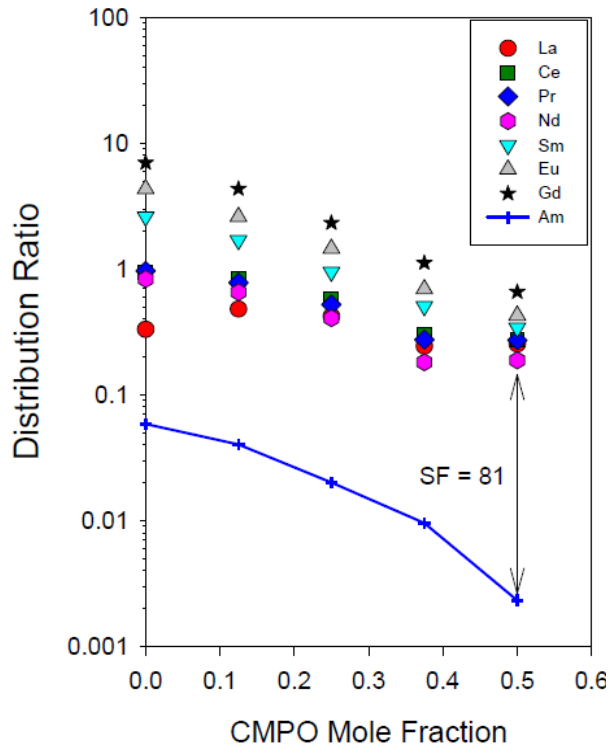
- Decreased synergism attributes
  - Less factors dictating distribution behavior
- More 'CMPO-like' extraction behavior at high acid conditions
- Fairly flat pH profile during higher pH (Am/Ln) separation step

Lumetta, G.J.; Braley, J.C.; Carter, J.C.; Solvent Ext. Ion Exch. 2013, 31(6), 567-577.

ALSEP process (Actinide/Lanthanide Separation)

- Jointly developed between ANL and PNNL with support from WSU and other universities
- Combines TRUEX/TALSPEAK functionality into a single process
- Testing at batch contact stage, conceptual flowsheet developed and flowsheet testing with spiked simulants planned for 2015-2016
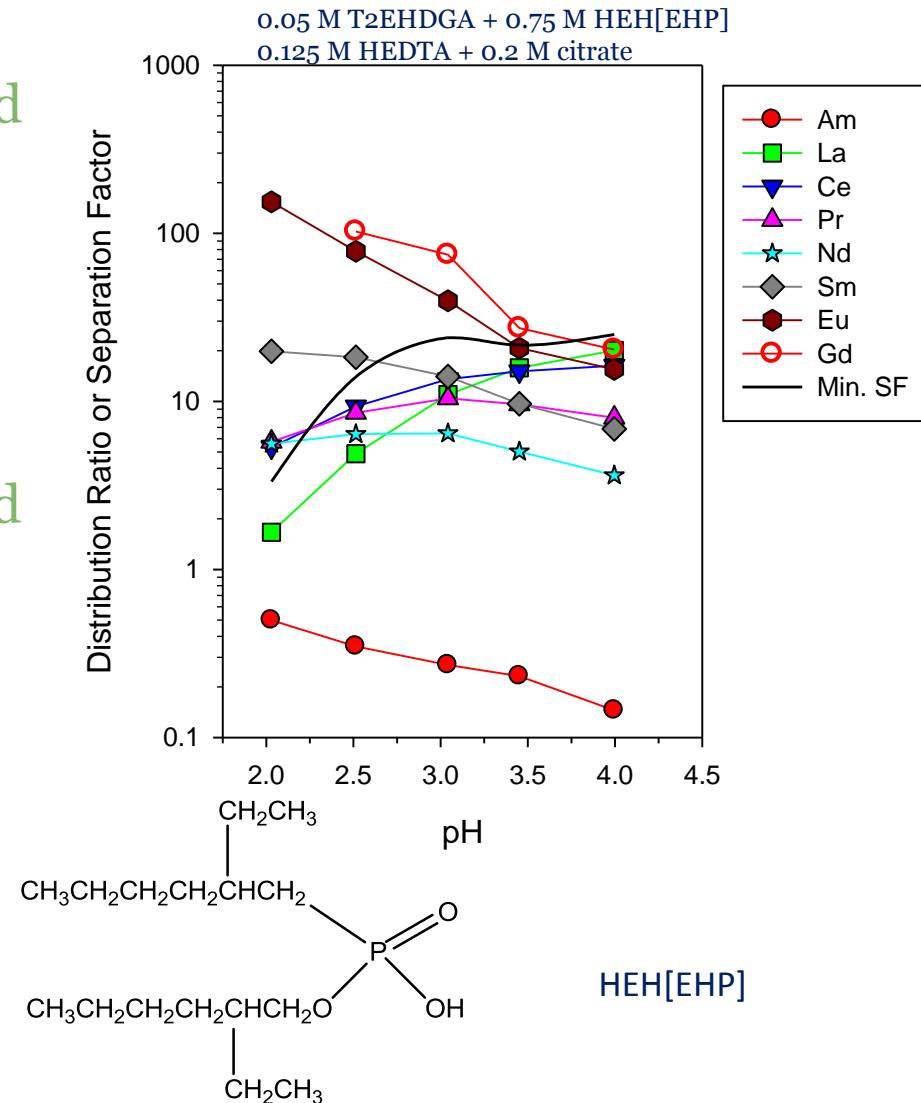 - Still resolving some kinetics issues and scrubbing of Zr, Mo, Ru

0.05 M T2EHDGA + 0.75 M HEH[EHP]
0.125 M HEDTA + 0.2 M citrate

T2EHDGA; R = $CH_2CH(CH_2CH_3)CH_2CH_2CH_2CH_3$
TODGA; R = $n-(CH_2)_7CH_3$

CH_2CH_3
$CH_3CH_2CH_2CH_2CHCH_2$
$CH_3CH_2CH_2CH_2CHCH_2O$
CH_2CH_3

HEH[EHP]
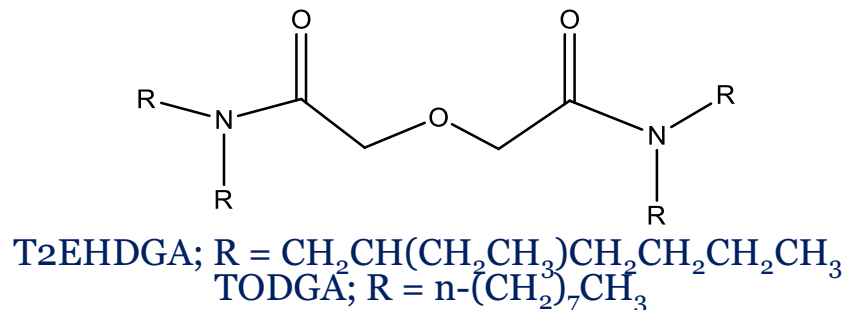
ALSEP – Attractive Features
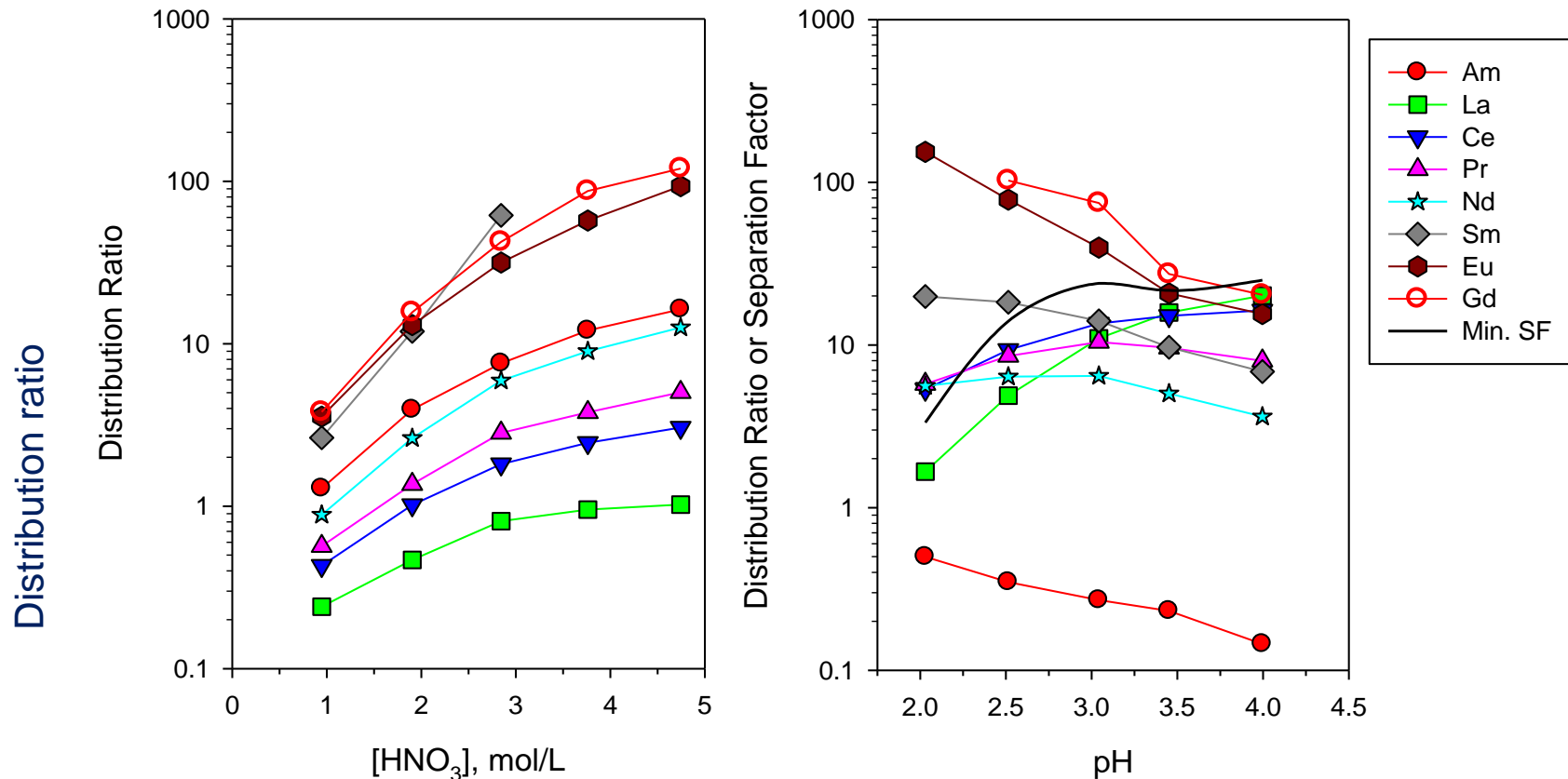
- Very straightforward extraction profile under acidic conditions
- Better separations factors at lower pH than TRUSPEAK

Lumetta, G.J.; Gelis, A.V.; Carter, J.C.; Niver, C. M.; Smoot, M. R., Solvent Ext. Ion Exch. 2014, 32(4), 333-347.
Gelis, A.V.; Lumetta, G.J.; Ind. Eng. Chem. Res., 2014, 53(4), 1624–1631.

ALSEP- Suppression of Zr extraction [51]

- Oxalic acid was investigated for suppressing the extraction of Zr from HNO_3
  - D_{Zr} values were reduced
  - But Am D values were also reduced
  - Potential for precipitation of Ln oxalates
- *trans*-1,2-Diaminocyclohexane-N,N,N',N'-tetraacetic acid (CDTA)
  - Effectively reduces Zr extraction
    - 0.1 M CDTA in 3 M HNO_3
      - D_{Zr} = 0.02 for 0.05 M T2EHDGA/0.75 M HEH[EHP]
  - Does not adversely impact Am extraction
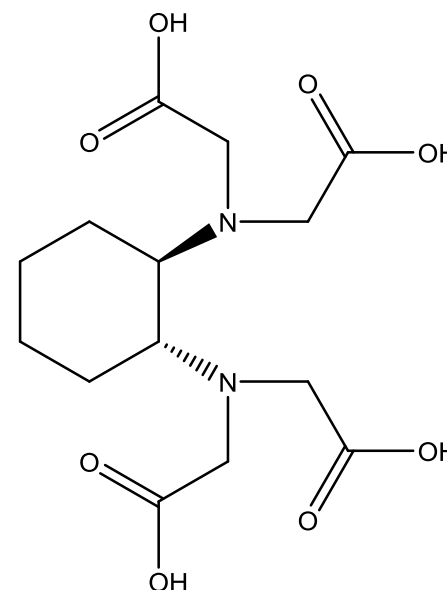
ALSEP: Optimization of Mo scrubbing
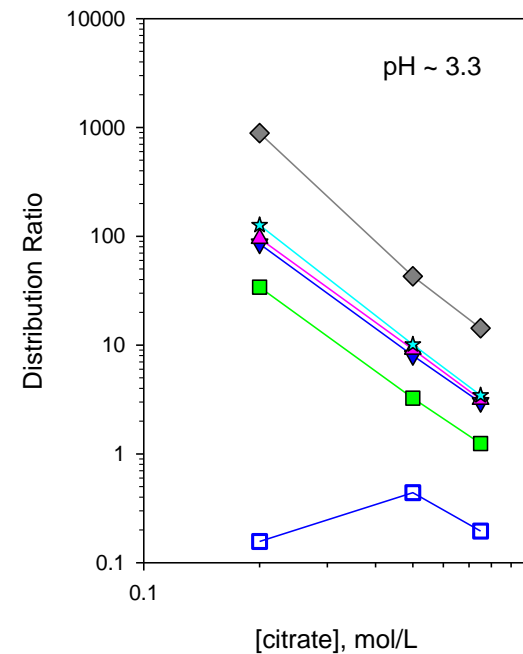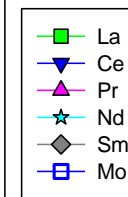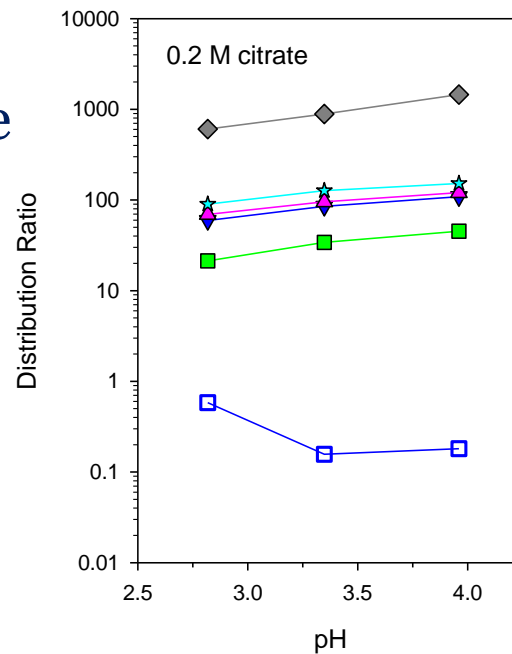
- Mo(VI) strongly extracted from HNO$_3$
- Mo must be removed from the solvent before MA stripping so that the MA product is not contaminated with Mo
- Scrubbing with a citrate buffer is the preferred method
  - Removes Mo
  - Conditions solvent for subsequent MA stripping step
- Residual acid in the solvent is removed

0.05 M T2EHDGA + 0.75 M HEH[EHP]

- **0.2 M citrate is adequate—no advantage to higher concentrations**
- **pH independent at pH > 3.3**

Next Stages

UREX → U
→ Tc

TRUSPEAK → Non-RE FPs
→ Ln/An(III)
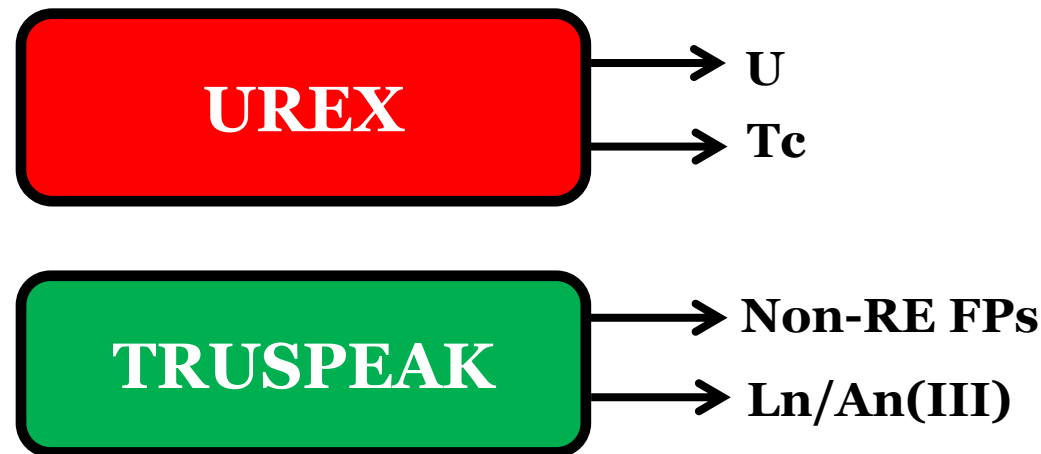
Even with TALSQueak or TRUSQueak, advanced partitioning probably has too many steps

SAnHex – Separations of Actinides using Hexavalent state

- Oxidation of Am (III) to V or VI

$$Am(III) \xrightarrow{\text{1.68V}} Am(VI)$$

$$Am(III) \xrightarrow{\text{1.73V}} Am(V)$$

$$Am(III) \xrightarrow{\text{2.62V}} Am(IV)$$
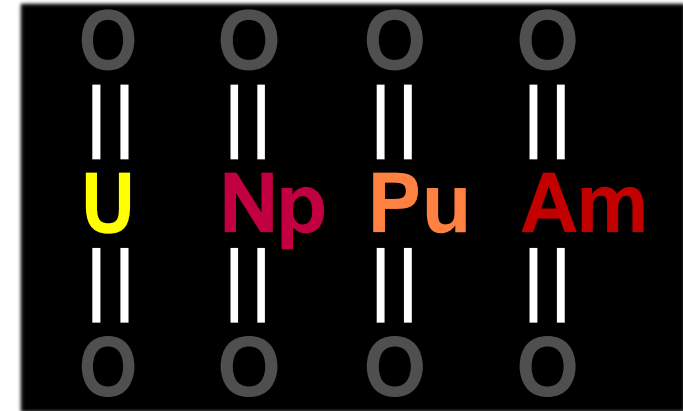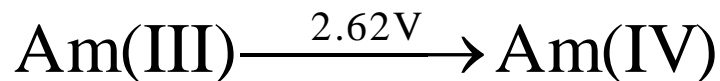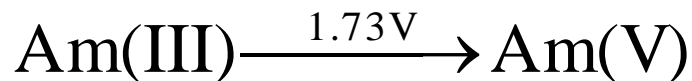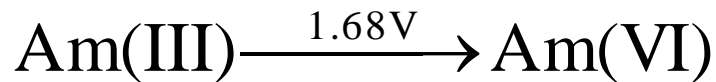
- Done with Ozone, Ag^{2+} or $NaBiO_3$
- $NaBiO_3$ brute oxidant and consumed
  - Clogs contactors
- Silver-catalyzed peroxydisulfate, fluoroxysulfate or ozone have also been examined
- Aqueous soluable Cu(III) and Ag(III) periodates are under consideration

SAnHex – Sodium Bismuthate (NaBiO$_3$)

- NaBiO$_3$ oxidizes Am(VI) sufficiently to allow recovery by DAAP

- Insoluble nature of NaBiO$_3$ will probably be an engineering challenge

- Hard to detect impurities in the system can significantly alter separations results

- Very preliminary results suggest that systems containing monoamide extractants minimize reproducibility issues
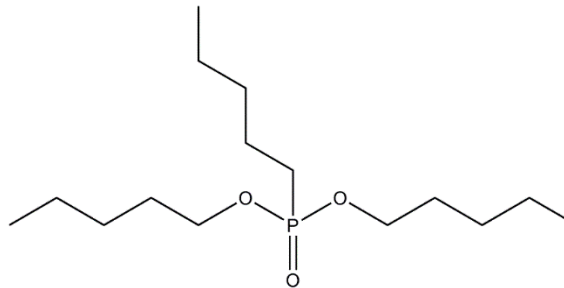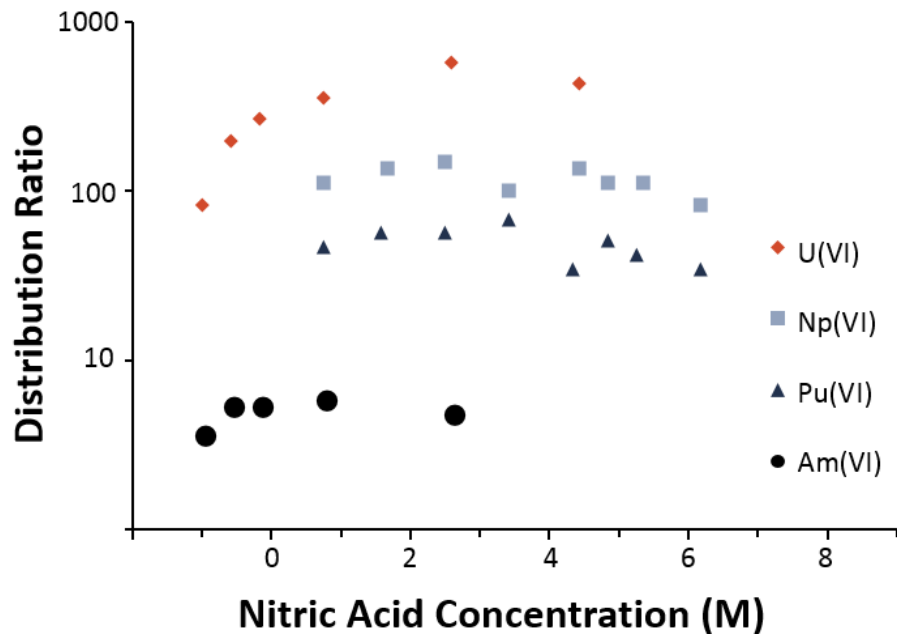
Diamyl amyl phosphonate (DAAP)

Mincher, B.J.; Schmidt, N.C.; Tillotson, R.D.; Elias, G.; White, B.M., Law, J.D., Solvent Extr. Ion Exch. 32(2), 2014, 153-166.
Mincher, B.J.; Martin, L.R.; Schmitt, N.C. Solvent Extr. Ion Exch. 30(5), 2012, 445-456.

SAnHex – Cu(III)/Ag(III) Periodates
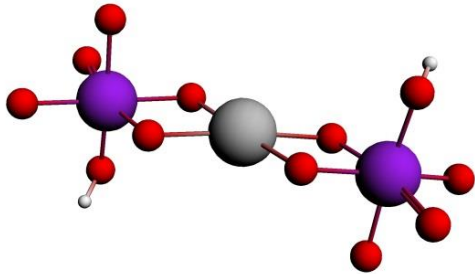
Silver (III) periodate

Copper (III) periodate

- Periodate was reported to oxidize Am(III) to one of the unidentifed higher oxidation states (tracer study), but not quantitatively

- It is known that the periodate ion forms numerous complexes with transition metals and stabilizes some of them in unusually high oxidation states, e.g. nickel as Ni^{IV}, copper as Cu^{III}, silver as Ag^{III}

- Cu(III) periodate exhibits a high oxidation potential of the Cu^{III}/Cu^{II} redox couple in alkaline medium. This fact was employed in the 1970s to achieve oxidation of Np(VI) and Pu(VI) to their heptavalent state in KOH solution

- Oxidation behavior of copper(III) periodate in acidic medium with respect to transuranium elements, as far as we know, was never examined

Summary courtesy Drs. Lumetta and Sinkov, PNNL

SAnHex - Am(III) oxidation by Cu(III) periodate in HNO_3

Part 1: Constant oxidant to Am(III) ratio with acidity variation
(10:1 molar excess = 3.33:1 stoichiometric excess)

C_{HNO_3}, M	0.25	0.5	0.75	1	2	3	3.5
Am(VI), %	99.6	99.8	99.6	99.9[1] (99.8)[2]	99.0	98.0	76.8[3] (83.2)[4]

- Cu(III) periodate is easy to prepare and sustains its ability to oxidize Am(III) for at least two months without any signs of degradation.

- Cu(III) periodate dissolves in nitric acid much more easily, compared with the Bi(V) compounds, with Cu(III) lifetime from several minutes to several hours depending on acidity.

- Oxidation efficiency of Am(III) with Cu(III) periodate is ~ 4 times higher than that of $NaBiO_3$ for the same acidity (3.5 M HNO_3) and stoichiometric excess of oxidation agent (3.33 to 1).

Summary courtesy Drs. Lumetta and Sinkov, PNNL

Am Oxidation Alternative Approach

Ozone oxidation of Am(III) effective when catalyzed
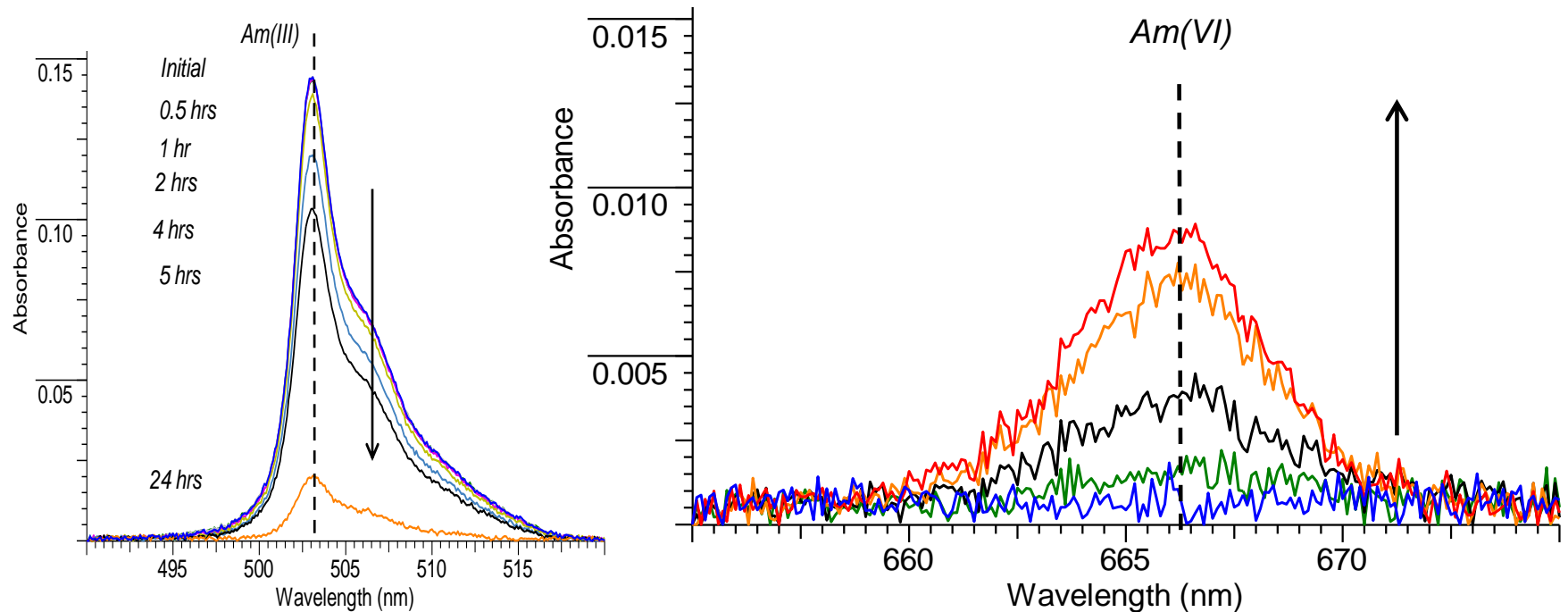
Oxidation of Am(III) to Am(VI) by ozone in 1 M HNO$_3$ in the presence of Ag

Slide courtesy of George Goff, Gordon Jarvinen and Wolfgang Runde, Los Alamos National Laboratory

Conclusions

- The U.S. is examining several advanced partitioning methods to achieve advanced fuel cycle goals
  - Aqueous Complexing Technology
    - TALSPEAK, Advanced TALSPEAK, TRUSPEAK, Advanced TRUSPEAK, ALSEP
  - Combined Extractant Systems
    - TRUSPEAK, Advanced TRUSPEAK, ALSEP
  - SAnHex Separations
    - NaBiO3, Copper Periodate, Ag-Catalyzed Ozone

The fundamental chemistry of these proposed systems is under examination to better inform future US, and maybe worldwide, decisions related to an advanced nuclear fuel cycle.

Acknowledgments

- Slides and information provided by
  - Professor Mikael Nilsson, UC-Irvine
  - Dr. Bruce Moyer, ORNL
  - Dr. Gregg Lumetta, PNNL
  - Dr. Sergey Sinkov, PNNL
  - Dr. Patricia Paviet, DOE

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- Lumetta, G.J.; Gelis, A.V.; Vandegrift, G.F., Solvent Extr. Ion Exch. 2010, 28(3), 287-312
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- Mincher, B.J.; Martin, L.R.; Schmitt, N.C. Solvent Extr. Ion Exch. 30(5), 2012, 445-456.

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