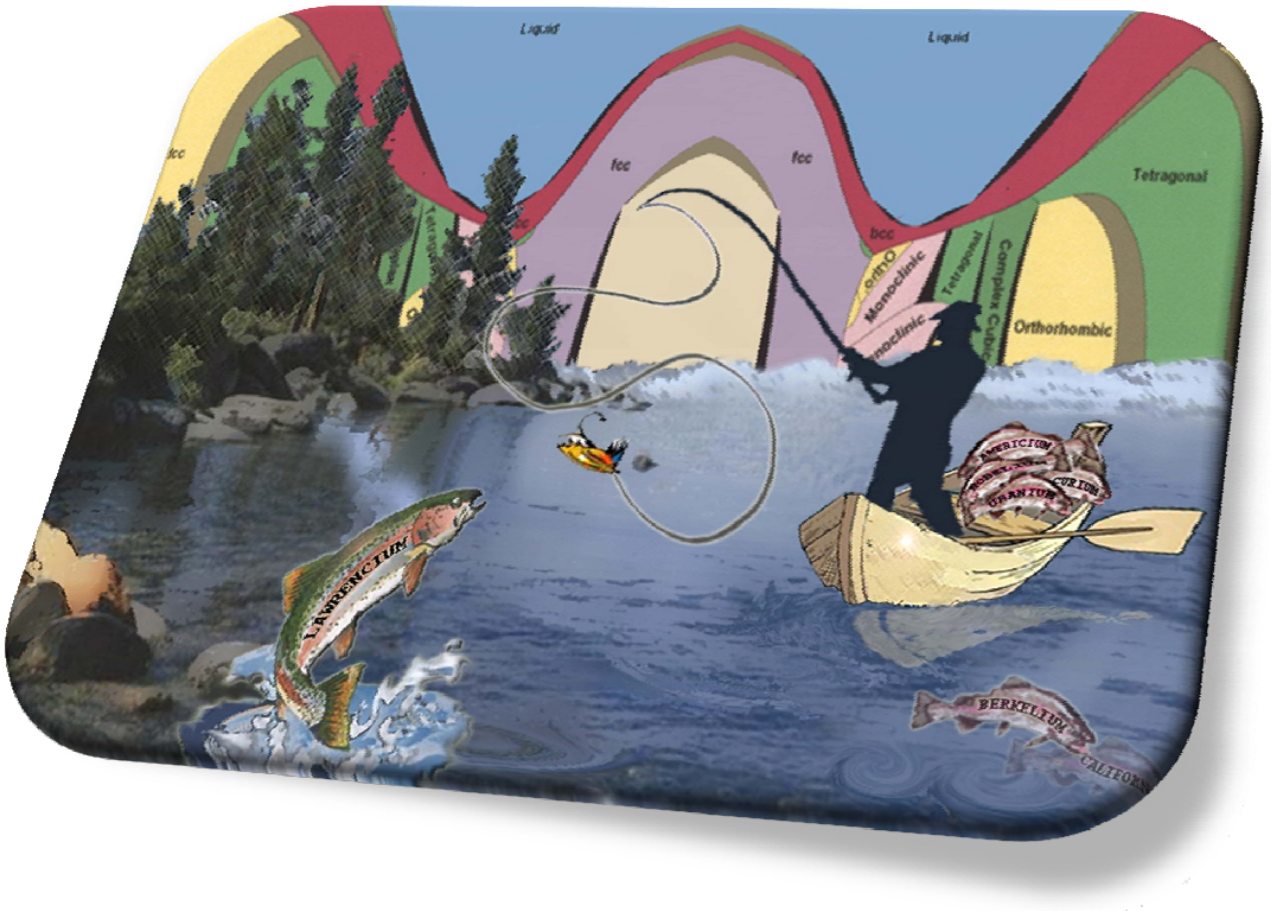
33rd Actinide Separations Conference

May 18-21, 2009 Tahoe City, CA



Hosted by Lawrence Livermore National Laboratory

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Logo Design by Andrea Goins, LLNL
Authors - L. M. McDonald and P. A. Wilk

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Notes:

Welcome to the 33rd Actinide Separations Conference

Welcome to the 33rd Actinide Separations Conference hosted this year by the Lawrence Livermore National Laboratory. This annual conference is centered on the idea of networking and communication with scientists from throughout the United States, Britain, France and Japan who have expertise in nuclear material processing. This conference forum provides an excellent opportunity for bringing together experts in the fields of chemistry, nuclear and chemical engineering, and actinide processing to present and discuss experiences, research results, testing and application of actinide separation processes. The exchange of information that will take place between you, and other subject matter experts from around the nation and across the international boundaries, is a critical tool to assist in solving both national and international problems associated with the processing of nuclear materials used for both defense and energy purposes, as well as for the safe disposition of excess nuclear material.

Granlibakken is a dedicated conference facility and training campus that is set up to provide the venue that supports communication between scientists and engineers attending the 33rd Actinide Separations Conference. We believe that you will find that Granlibakken and the Lake Tahoe views provide an atmosphere that is stimulating for fruitful discussions between participants from both government and private industry.

We thank the Lawrence Livermore National Laboratory and the United States Department of Energy for their support of this conference. We especially thank you, the participants and subject matter experts, for your involvement in the 33rd Actinide Separations Conference.

Conference Chair
Karen Dodson

Conference Liaison
Lisa McDonald

Conference Technical Program Chair
Philip Wilk

Conference Organizing Committee
James McNeese
Brandon Chung
David Riley

Notes:

Actinide Separations Conference Advisory Board

Elizabeth A. Bluhm
Los Alamos National Laboratory

Calvin H. Delegard
Pacific Northwest National Laboratory

Karen Dodson
Lawrence Livermore National Laboratory

L. Kevin Felker
Oak Ridge National Laboratory

Mark P. Jensen
Argonne National Laboratory

Alice M. Murray
Savannah River National Laboratory

Kenneth L. Nash
Washington State University

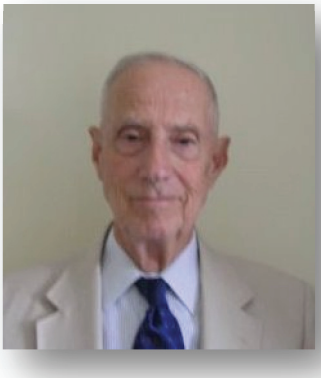
Robert T. Paine
University of New Mexico

Terry A. Todd
Idaho National Laboratory

Notes:

2009 Glenn T. Seaborg Award Winner

Raymond G. Wymer



Dr. Raymond G. Wymer received his B.S. degree from Memphis State University, and his M.S. and Ph.D. degrees in chemistry and physics from Vanderbilt University.

Dr. Wymer was employed by Oak Ridge National Laboratory in the Chemical Technology Division from 1953 until his retirement in 1991. During his employment at ORNL he was involved in research and development in all aspects of the nuclear fuel cycle. He became Director of the Chemical Technology Division, a chemical engineering division employing about 300 chemical engineers, chemists, technicians and support staff.

Dr. Wymer has consulted extensively since his retirement in 1991 in the areas of radioactive waste management, fuel cycle process chemistry, and site remediation for DOE and its contractors. He has had extensive consulting experience at Hanford with the Tank Waste Remediation Systems program. He assists DOE and its contractors in program reviews. Dr. Wymer has served on numerous committees and workshops of the National Academies (formerly the National Academy of Science) that deal with DOE's waste management and site remediation activities and closure activities. He is an Associate Member of the National Academies and a member of the Academies Nuclear and Radiation Studies Board.

Dr. Wymer's other activities include consulting with DOE, the U.S. Department of State and the International Atomic Energy Agency on matters of nuclear non-proliferation in the areas of nuclear fuel reprocessing and uranium enrichment by chemical exchange processes. He served on a United Nations UNSCOM team to Iraq in the mid-1990s evaluating Iraq's uranium enrichment capability by chemical exchange. He is currently a consultant for Oak Ridge National Laboratory and serves on the Global Nuclear Energy Partnership Independent Review Committee for DOE.

Dr. Wymer is co-author of a book "Chemistry in Nuclear Technology" and co-edited a book on "Light Water Reactor Fuel Reprocessing." He was an editor of the journal Radiochimica Acta for more than ten years until his retirement. He has written numerous reports and open literature publications on all aspects of the nuclear fuel cycle and has contributed technical articles for incorporation in encyclopedias. Dr. Wymer is an Adjunct Professor in the Department of Civil and Environmental Engineering of Vanderbilt University.

Dr. Wymer has received recognition for his contributions in the nuclear area, including the Robert E. Wilson Award in Nuclear Chemical Engineering from the American Institute of Chemical Engineers. He is a Fellow of the American Nuclear Society. Dr. Wymer and his wife Kenalene reside in Oak Ridge, Tennessee.

Notes:

Past Seaborg Award Recipients

1984	Glenn T. Seaborg	University of California at Berkeley
1985	Don E. Ferguson	Oak Ridge National Laboratory
1986	Larned B. Asprey	Los Alamos National Laboratory
1987	Wallace W. Schulz	Westinghouse Hanford Company
1988	Lawrence J. Mullins	Los Alamos National Laboratory
1989	Gregory R. Choppin	Florida State University
1990	Donald R. Orth	Westinghouse Savannah River Company
1991	David O. Campbell	Oak Ridge National Laboratory
1992	E. Philip Horwitz	Argonne National Laboratory
1993	Earl J. Wheelwright	Pacific Northwest National Laboratory
1994	Leslie Burris	Argonne National Laboratory
1995	Robert R. Penneman	Los Alamos National Laboratory
1996	David G. Karraker	Westinghouse Savannah River Company
1997	Major C. Thompson	Westinghouse Savannah River Company
1998	Walter D. Bond	Oak Ridge National Laboratory
1999	Jack L. Ryan	Pacific Northwest National Laboratory
2000	John L. Swanson	Pacific Northwest National Laboratory
2001	George F. Vandegrift	Argonne National Laboratory
2002	Leonard W. Gray	Lawrence Livermore National Laboratory
2003	Kenneth L. Nash	Argonne National Laboratory
2004	Emory D. Collins	Oak Ridge National Laboratory
2005	Terry A. Todd	Idaho National Laboratory
2006	Renato Chiarizia	Argonne National Laboratory
2007	Leland L. Burger	Pacific Northwest National Laboratory
2008	Gordon D. Jarvinen	Los Alamos National Laboratory

Notes:

Conference Agenda

Monday, May 18, 2009		
6:00 pm to 9:00 pm	Registration	Granhall
6:30 pm to 7:30 pm	Granlibakken Hosted Social Hour	Granhall
7:00 pm to 8:00 pm	Dinner	Granhall

Tuesday, May 19, 2009		
7:30 am to 8:30 am	Breakfast	Granhall
8:30 am to 8:35 am	Welcome and Introductions	Mountain Room
8:35 am to 10:05 am	Plenary Speakers	Mountain Room
10:05 am to 10:25 am	A.M. Break	Mountain Room
10:25 am to 12:00 pm	Plenary Speakers cont'd.	Mountain Room
12:00 pm to 1:00 pm	Lunch	Granhall
1:00 pm to 2:40 pm	Technical Session IA: Lanthanide & Actinide Separations and Advanced Characterization	Mountain Room
2:40 pm to 3:00 pm	P.M. Break	Mountain Room
3:00 pm to 4:40 pm	Technical Session IB: Reactor Fuel Technology	Mountain Room
6:00 pm to 7:00 pm	Dinner	Granhall
7:00 pm to 9:00 pm	Poster Session Granlibakken Hosted Social Hour	Lake Room

Notes:

Wednesday, May 20, 2009		
7:30 am to 8:30 am	Breakfast	Granhall
8:30 am to 10:10 am	Technical Session II: Actinide Solution Chemistry	Mountain Room
10:10 am to 10:30 am	A.M. Break	Mountain Room
10:30 am to 12:10 pm	Technical Session II: Actinide Solution Chemistry cont'd.	Mountain Room
12:10 pm to 1:00 pm	Lunch	Granhall
1:00 pm to 5:00 pm	Afternoon is free	
12:10 pm to 2:00 pm	Advisory Board Meeting	Alumni Room
6:00 pm to 7:00 pm	Dinner	Granhall

Thursday, May 21, 2009		
7:30 am to 8:30 am	Breakfast	Granhall
8:30 am to 10:10 am	Technical Session III: Aqueous and Pyrochemical Processing	Mountain Room
10:10 am to 10:30 am	A.M. Break	Mountain Room
10:30 am to 12:10 pm	Technical Session III: Aqueous and Pyrochemical Processing cont'd.	Mountain Room
12:10 pm to 1:15 pm	Lunch	Granhall
1:15 pm to 2:55 pm	Technical Session IV: Waste Treatment, Stabilization, and Disposition	Mountain Room
2:55 pm to 3:15 pm	P.M. Break	Mountain Room
3:15 pm to 4:30 pm	Technical Session IV: Waste Treatment, Stabilization, and Disposition cont'd.	Mountain Room
5:30 pm to 6:30 pm	Granlibakken hosted Social Hour	Granhall
6:30 pm to 9:00 pm	Banquet Dinner/Seaborg Award Presentation/Keynote Address	Granhall

Notes:

Tuesday, May 19, 2009		
Plenary Session **Chair: Karen Dodson, LLNL**		
8:30 am	Welcome	Dodson
8:35 am	Actinide Separations in the Advanced Fuel Cycle Initiative	Todd
9:20 am	Nuclear Technology in China and the U.S. for the 21st Century	Gray
10:05 am	**Break**	
10:25 am	Radiochemical Milking as a Technique for Small Actinide Signatures	Moody
11:10 am	Sustainability and Nuclear Energy: Perspectives on Alternate Futures	Nash
12:00 pm	**Lunch**	

Technical Session IA: Lanthanide & Actinide Separations and Advanced Characterization **Chair: Brandon Chung, LLNL**		
1:00 pm	Results on the Production of Neptunium Oxide from Impure Solutions	Kyser
1:25 pm	Recovery of Ac-225 from Aged Light Water Breeder Reactor Fuel Sources	Meikrantz
1:50 pm	Group Separation of Actinides and Fission Products with Diamides of Dipicolinic Acid	Paulenova
2:15 pm	Effects of Chemical Composition on Metallurgical Properties of Plutonium Alloys	Chung
2:40 pm	**Break**	

Technical Session IB: Reactor Fuel Technology **Chair: Kevin Felker, ORNL**		
3:00 pm	Overview and Recent Advances in Uranium Ore Processing	Cooper
3:25 pm	Capture of Tritium During Voloxidation of Zircaloy Fuel Hulls	Crowder
3:50 pm	Preliminary Results of Voloxidation Processing of Kilogram Quantities of Used Nuclear Fuel	Spencer
4:15 pm	Recycle of Fuel-Fabrication Scrap and Recycle of Spent Fuel from High-Performance Research Reactors	Vandegrift
4:40 pm	**Adjourn**	

Notes:

7:00 pm – 9:00 pm	Poster Session	Chair: Philip Wilk, LLNL
Recycling of Metallic-Based Plutonium Residues: Conversion into PuO_2 for Aqueous Polishing		Faure
Qualitative and Quantitative Study of the Radiolytic Products of Bis-DGA UAM-069 Used for Actinides(III) and Lathanides(III) Co-Extraction Processes		Galán
Combining TRUEX and TALSPEAK in One Process: Solvent Development		Guelis
Alternative Red-Ox Reagents in NPEX and TRUEX Processes of UREX+		Guelis
Separation of Water Soluble Impurities from Plutonium Oxide		Harland
Hijacking Proteins with Plutonium		Jensen
Electrochemical Reduction of Plutonium Oxide		Jones
Aspen/OLI Model of Waste Evaporation and Neutralization Process for MOX Waste Solidification Building		Laurinat
Reduction of Tetravalent Plutonium in the Presence of Acetohydroxamic Acid		Paulenova
Complexation and Extraction of Tc[II] with Novel Methylresorcin [4]Arene Derivatives		Paviet-Hartmann
Removal of Solids from Highly Enriched Uranium Solutions Using the H-Canyon Centrifuge		Rudisill
LLNL's Materials Process Laboratory DOE Standard 3013 Packaging of Stabilized Materials		Silveira
Uranium Characterization in K Basin Sludge		Sinkov
NMR Analysis of the TALSPEAK Process Solvent After Alpha-Irradiation with Plutonium-238		Snow
Application of Formohydroxamic Acid in Nuclear Processing: Synthesis and Complexation with Technetium-99		Wright

Notes:

Wednesday, May 20, 2009		
Technical Session II: Actinide Solution Chemistry **Chair: Ken Nash, Washington State University** **Co-Chair: Tracy Rudisill, SRNL**		
8:30 am	Potentiometric Determination of Uranyl-Nitrate Stability Constants by Competition Method	Smith
8:55 am	Spectrometric Measurements of Metal-DTPA Complexes in Lactic Acid Media under TALSPEAK-Like Conditions	Nilsson
9:20 am	Preorganization and Rigidity: New Ligands for Trivalent Actinides/Lanthanides Separation	Delmau
9:45 am	Ternary Complexes for Am/Cm Separation in a TALSPEAK Framework	Jensen
10:10 am	**Break**	
10:30 am	Enhancing Understanding of TALSPEAK Chemistry: Sigma Team Research at WSU	Nash
10:55 am	TALSPEAK Chemistry in the Absence of DTPA: Nd(III) Speciation in the Organic Phase Helps to Identify One More Previously Unaccounted for Player in This Process	Sinkov
11:20 am	Development of a Sequential Separation Method for up to Five Actinides from Nitric Acid Media Using Chromatographic Extraction Resins	Farawila
11:45 am	Selective Separate Actinide Ions Using N,N-di-2-ethyl-hexyl-3-oxa-glutaramic Acid	Tian
12:10 pm	**Lunch** **Afternoon Free (Advisory Board Meeting)**	

Notes:

Thursday, May 21, 2009		
Technical Session III: Aqueous and Pyrochemical Processing **Chair: James McNeese, LLNL** **Co-Chair: Sharon Slade, AWE**		
8:30 am	Processing of Residues for Offsite Shipment at LLNL	McNeese
8:55 am	Tantalum Corrosion and Ceramic Adherence with Plutonium Titanium Alloys	Slade
9:20 am	Americium-Beryllium Neutron Source Dissolution	Fonnesbeck
9:45 am	Actinide Fuel Dissolution Methods	Berg
10:10 am	**Break**	
10:30 am	A Path Forward for Recovery of Purified ^{241}Am from Hydrochloric Acid Plutonium Processing Operations	Schulte
10:55 am	Fabrication of Minor Actinide Bearing Nuclear Fuel	Willson
11:20 am	The Chromatographic Separation of Trace Actinides from Large Quantities of Uranium	McAlister
11:45 am	H_2 Attenuation in K Basin Sludge from the Uranium Metal – Water Reaction	Delegard
12:10 pm	**Lunch**	

Technical Session IV: Waste Treatment, Stabilization and Disposition **Chair: David Riley, LLNL**		
1:15 pm	Long Term Storage of Used Fuel followed by Recycle and Reuse of Most Components as a Sustainable Path for Abundant Nuclear Energy	DelCul
1:40 pm	The Status of De-Inventory of Security Category I/II Materials from Lawrence Livermore National Laboratory	Riley
2:05 pm	Technetium Separations and Waste Form Development	Jarvinen
2:30 pm	3013 Surveillance and Monitoring Program – Characterization of Pu-Bearing Solids	Kessinger
2:55 pm	**Break**	
3:15 pm	Moisture Analysis of Chloride-Bearing Plutonium Oxide Surrogates for Small-Scale Corrosion Tests	Duffey
3:40 pm	Analysis of the Causal Factors that Lead to Hydrogen Generation within 3013 Storage Containers	Almond
4:05 pm	Gas Generation in Small-Scale Corrosion Tests with Chloride-Bearing Plutonium Oxide Surrogates	Duffey
4:30 pm	**Adjourn**	

6:30 pm	**Seaborg Award Banquet**	

Notes:

Tuesday, May 19

Plenary Session

Chair: Karen Dodson, LLNL

Notes:

Actinide Separations in the Advanced Fuel Cycle Initiative

Terry A. Todd*[1], L. Kevin Felker[2]

[1]Idaho National Laboratory

[2]Oak Ridge National Laboratory

The Department of Nuclear Energy's Advanced Fuel Cycle Initiative (AFCI) is developing actinide separation technologies in support of future sustainable fuel cycles. One of the driving forces behind separating actinides from used nuclear fuel is to reduce the time that used fuel or waste products will require isolation from people. Currently, the radiotoxicity of used fuel is higher than that of natural uranium ore for hundreds of thousands of years. By separation and transmutation of actinides (Np, Pu, Am) the radiotoxicity of used fuel can be reduced to that of uranium ore in several hundred years. This means that the majority of the used fuel can be managed on engineering time scales, rather than geologic time scales. The small remainder of long-lived fission products, primarily ^{129}I, ^{99}Tc, and ^{135}Cs, have very long half lives, but have relatively low radiotoxicity. These will require new technologies to transmute them or isolation for very long (millions of years). The AFCI program is researching new methods to separate actinide elements, including modifications to TBP-based extraction processes, new extraction processes, and electrochemical separation in molten salt. An update of recent research activities will be presented as well as a discussion of new program direction to include more science-based research into the program.

Notes:

Nuclear Technology in China and the U. S. for the 21st Century

Leonard W. Gray

Lawrence Livermore National Laboratory
Livermore, California

Under the auspices of the People-to-People Ambassador Program, the ANS invited a select group of ANS members to participate in an exchange with Chinese Nuclear Scientists and Engineers. Meetings with several Chinese Nuclear Institutes took place in Beijing and Shanghia, including visits to several of the Chinese Institutes and several reactor sites with tours of reactor training simulators, control rooms, construction sites and R&D Laboratories. The Chinese nuclear program to train personnel and construct reactors is extremely aggressive – 11 units in operation and 16 units under construction. Their goal is to increase the nuclear portion of electrical power generation from <1% in 2007 to 10% by 2035 and 30% by 2050. This report will review the trip and compare the Chinese Nuclear Program to that of the United States.

Radiochemical Milking as a Technique for Small Actinide Signatures

K. J. Moody

Lawrence Livermore National Laboratory
Livermore, California

The minor isotopic signatures in samples of uranium and plutonium provide information that is important for inferring the origin of an interdicted sample. Radiochemical milking is a method by which the concentrations of these nuclides can be quantified in the presence of overwhelming quantities of other isotopes of the same element. The basic method will be described, and information extracted from real samples will be presented.

Notes:

Sustainability and Nuclear Energy: Perspectives on Alternate Futures

Ken Nash

Chemistry Department
Washington State University, P.O. Box 644630, Pullman, WA 99164-4630

About 85% of the energy consumed at present on Earth is derived from fossil carbon. Though the Industrial Revolution that produced today's standard of living was fueled primarily by these resources, increasing evidence that atmospheric CO_2 is causing (or at minimum contributing to) perceived changes in the global climate reduces the desirability of continuing to exploit carbon as a primary energy source. Furthermore, all indications are that exploitable oil supplies are in decline. Worst case scenarios place the end of oil only a few decades in the future. Oil might in fact become too valuable as a chemical feedstock to be used for combustion to produce heat. Though there has been resistance in some quarters to the idea that a significant response to this combined energy-environmental "crisis" is needed today, political and scientific leaders around the world are increasingly accepting of the need for change, particularly for new energy supplies. In this presentation, an overview of the current status of energy supplies and usage will be offered focusing primarily on energy as the primary driver of continuing advancement of society. The role of fission-based nuclear energy (with and without recycle of useful components) in defining this future will be emphasized.

Notes:

Tuesday, May 19

Technical Session IA:
Lanthanide & Actinide Separations and Advanced Characterization

Chair: Brandon Chung, LLNL

Notes:

Results on the Production of Neptunium Oxide from Impure Solutions

E. A. Kyser*
Savannah River National Laboratory

J. L. Bodkin, B. L. Sims Jr.
HB-Line Engineering

The Savannah River Site has historically recovered ^{237}Np from reactor fuel. Over the last five years accumulated solution inventories were processed into neptunium oxide for future production of ^{238}Pu. This stabilization of neptunium solutions generated additional recycle solutions that contained a different composition and concentrations of impurities. Solvent extraction and anion exchange processes were modified to remove these additional impurities (in the "Np Part II campaign"). This processing approach allowed the use of the existing bases documents for safe shipment of the oxide product. This presentation discusses the details of purification for the major impurities and the results from the Np Part II campaign.

SRNL-STI-2009-00211

Notes:

Recovery of Ac225 from Aged Light Water Breeder Reactor Fuel Sources

David Meikrantz*, Troy Tranter, Richard Tillotson
Idaho National Laboratory, Idaho Falls, ID, USA

Philip Horwitz, Daniel McAlister
PG *Research Foundation, Darien IL, USA*

James Harvey
Northstar Nuclear Medicine, Madison, WI, USA

Experiments have been conducted to investigate the potential of recovering Ac225 from forty year old light water breeder reactor fuel stored at the INL. The material is composed of about 97% natural thorium and 3% U233 in ceramic oxide fuel pellets. Decay of U233 to Th229 provides a source of daughter Ac225, a medical isotope that has shown efficacy in the treatment of leukemia type cancers. Dissolution of this ceramic oxide fuel in a mixture of nitric and hydrofluoric acid is described. Recovery of the daughter Ac225 from the Th229 is complicated by dilution in massive amounts of natural thorium. Therefore a unique separation scheme was devised and tested that includes solvent extraction of the uranium and thorium into Diamyl, amylphosphonate and subsequent scrubbing to remove Ra225 and Ac225 daughters. Recovery and purification of the Ac225 by extraction chromatography techniques provides a high degree of separation from both metallic and radioisotope impurities and concentrates the product into a small volume for use.

Notes:

Group Separation of Actinides and Fission Products with Diamides of Dipicolinic Acid

A. Paulenova[1], J.L.Lapka[1], M.Yu. Alyapyshev[2], V.A. Babain[2], J.D. Law[3], R.S. Herbst[3]
[1]Oregon State University, Corvallis, Oregon, USA
[2]Khlopin Radium Institue, St-Petersburg, Russia
[3]Idaho National Laboratory,Idaho Falls, USA
(Email: alena.paulenova@oregonstate.edu)

In recent years, the effort toward a closed nuclear fuel cycle has grown in importance and interest because chemically similar radionuclides (e.g., Cs and Sr) can be vitrified in a stable matrix for long term storage, while lanthanides and minor actinides can be further processed for recycling in reactors. The Universal Extraction (UNEX) is a process that has been developed for the simultaneous extraction of cesium, strontium, and actinides from acidic solutions, followed by selective stripping of radionuclides by changing aqueous phase conditions.[1]

The UNEX extraction mixture, based on CCD (cobalt dicarbollide), PEG and CMPO in polar solvent FS-13 (phenyltrifluoromethyl sulfone), provides simultaneous extraction of Cs, Sr, actinides and lanthanides from aqueous nitric acid solutions up to ~2 M HNO_3 and ~4 M total nitrates. However, the current usage of the UNEX process is limited due to the low solubility of CMPO in the organic phase, and as a result, new extractants are being investigated as possible substitutes for a modified UNEX group separation process.

A great synergistic effect with chlorinated cobalt dicarbollide (CCD) on the extraction of Am and Ln with diamides of dipicolinic acid was observed. [2] Five different diamides of different structure were synthesized and their extraction performance toward selected actinides (An) and lanthanides (Ln) with the studied diamides and the mixture of diamides with CCD were investigated as a function of the ligand structure and nitric acid concentration. As a result of numerous experiments with various complexants, the experimental conditions which provide common separation of Cs, Sr, actinides and lanthanides were found. With an appropriate stripping protocol, the modified diamide-UNEX process can be used for the extraction of Cs, Sr, An and Ln followed by selective separation.

References:

[1] V. N. Romanovskiy, I. V. Smirnov, V. A. Babain et al., Solv. Extr. Ion Exch. 19 (2001) 1
[2] A. Paulenova, M. Yu. Alyapyshev, V. A. Babain, et al; Sep. Sci. Techn., 43, 2606 2008

Notes:

Effects of Chemical Composition on Metallurgical Properties of Plutonium Alloys

Brandon W. Chung*, Kenneth E. Lema, Jeff A. Stanford, and David S. Hiromoto
Lawrence Livermore National Laboratory, 7000 East Avenue, Livermore, CA 94551 USA

Plutonium exhibits notoriously complicated metallurgical behaviors, depending sensitively on phase as well as on chemical content, microstructure, and age. Of the six allotropic phases, alpha is the hardest and least ductile, and delta is the softest and most ductile. To design alloys, process controls are therefore required to control phase and chemical content, and to insure phase and microstructure stability. One of the key issues in understanding the metallurgical behavior is the gallium microsegregation that occurs during cooling from the melt. This "coring" process results in partitioned microstructures that consist of Ga-rich cores with Ga-poor edges. Core grains can be homogenized at moderately high (i.e. >400°C) temperatures. Other common impurities, such as Fe and Ni, also have significant effect on metallurgical properties. Another issue is the radioactive decay of plutonium that incessantly creates lattice damage and in-growth of radiogenic helium which drives metallurgical evolution. In this paper, we apply several analytical techniques to characterize chemical segregations in plutonium alloys as functions of homogenization time and temperature. We will present how chemical segregation and in-grown helium bubbles in plutonium alloys affect metallurgical properties such as microstructure and static mechanical properties.

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

Notes:

Tuesday, May 19

Technical Session IB:
Reactor Fuel Technology

Chair: Kevin Felker, ORNL

Notes:

Overview and Recent Advances in Uranium Ore Processing

Brian Cooper*, Dennis Gertenbach, and James D. Navratil
Hazen Research Inc., Golden, CO, USA

The recent increased demand for uranium fuel has caused a boom in uranium exploration, mine development, plant operations and reprocessing. The extraction of uranium values from low- to mid-grade ores that were previously not economical is now a standard activity in industry, as is the attempt to recover values from waste streams. The production of yellow cake from uranium ore may involve crushing and grinding, acid or alkaline leaching, purification by SX or IX and precipitation-calcination steps. There have been some recent improvements to these production steps, especially in separation technologies. This paper will present an overview of yellow cake production from various uranium ores and highlight current practices as well as recent developments in uranium separation and purification methods.

Capture of Tritium during Voloxidation of Zircaloy Fuel Hulls

Mark L. Crowder* and James E. Laurinat
Savannah River National Laboratory, Aiken, SC, USA

Zircaloy fuel hulls comprise about 25% by mass of the spent nuclear fuel in the United States. Leading treatment options for recycle of the hulls and waste alloy processing require heating, which causes release of tritium and other radioisotopes. Accurate information is needed on the volatile components of the large and growing amount of spent fuel and its zircaloy cladding. This study demonstrated a simple method to evaluate the tritium content of hulls via oxidation of the hull and capture of the volatilized tritium in liquids. Past experimental data were used to develop an Arrhenius model of zircaloy oxidation, which includes an initial, nonlinear rate and a faster, linear rate after "breakaway" of the oxide film. The zircaloy hulls used in this study were previously separated from fuel via dissolution in nitric acid at elevated temperature and pressure. Hull portions were heated in air inside a thermogravimetric analyzer (TGA). The TGA was rapidly heated to 1000 °C to promote the oxidation and easy capture of tritium. To capture tritium, the TGA off-gas was bubbled through a series of liquid traps. The study demonstrated that complete oxidation of the zircaloy samples occurred within the two hours predicted. The concentrations of tritium in bubbler solutions indicate that tritium was captured nearly quantitatively.

Notes:

Preliminary Results of Voloxidation Processing of Kilogram Quantities of Used Nuclear Fuel

B. B. Spencer*, G. D. Del Cul, R. T. Jubin, R. S. Owens, D. W. Ramey, and E. D. Collins
Oak Ridge National Laboratory, Oak Ridge, TN, USA

Advanced nuclear fuel processing methodologies are being studied as part of the Advanced Fuel Cycle Initiative (AFCI) program at ORNL. To support this initiative, processes and equipment were deployed at ORNL to perform all steps in the recycle process on actual used nuclear fuels, ranging from used fuel receipt to production of products and waste forms at the kilogram-scale.

Initially, approximately 4 kg of saw-segmented Dresden reactor used fuel was processed using lab-scale equipment in multiple batches. The second processing campaign used a new single pin shear and a new bench-scale voloxidizer to perform the dry head-end treatment of two different fuels, Surry and North Anna, for an additional total amount of approximately 5 kg of used fuel. The overall goal was to evaluate the oxidation of various used fuel (low and high burn-up, short- and long-cooled fuel) under a range of processing conditions.

The material used for each batch and general processing conditions are summarized in Table 1. Progress of the oxidation reaction was monitored continuously by two primary measurements; the concentration of oxygen in the effluent stream which was depressed as the oxygen was consumed, and the concentration of krypton-85 in the effluent stream as measured by a gamma counter on the off-gas pipeline.

Table 1. Voloxidation test conditions for second campaign.

Batch	Fuel Source	Burnup (GWd/MT)	Batch size (kg*)/(kg**)	Segment Length (in)	Oxidation Gas	Operation Temperature (°C)
1	Surry-2	36	1.223/1.704	1.0	Air	500
2	North Anna	63–70	2.071/2.885	0.88	Air	600
3	North Anna	63–70	2.012/2.803	0.88	Oxygen	600

* Heavy metal basis.

** Total fuel (oxide + cladding) basis.

Fission product gases evolved from the fuel during the oxidation process were trapped for subsequent chemical and radiochemical analysis. The series of traps included a bed of molecular sieves to recover tritium (as HTO), silver-substituted zeolite to capture iodine (e.g. as AgI), a caustic scrubber to collect carbon dioxide (including $^{14}CO_2$), a hydrogen-substituted mordenite to capture krypton (e.g. ^{85}Kr) by cryogenic temperature swing adsorption, and a silver-substituted mordenite to capture xenon by cryogenic temperature swing absorption. The quantities of these volatile gases collected were compared to ORIGEN calculations to estimate the effectiveness of the voloxidation process to separate the volatiles from the used fuel.

This paper will describe the voloxidation system and present preliminary results from the second processing campaign.

Notes:

Recycle of Fuel-Fabrication Scrap and Recycle of Spent Fuel from High-Performance Research Reactors

George F. Vandegrift*, Dominique C. Stepinski, Javier Figueroa, Mark A. Williamson
Laura E. Maggos, John Swanson, James Jerden, Jeff Fortner, and Allen Bakel
Argonne National Laboratory, Argonne, IL, USA

The NNSA Global Threat Reduction--Conversion program is currently engaged in development of nuclear fuel that would enable conversion of US high-performance research reactors (HPRRs) from high-enriched uranium (HEU) to low enriched uranium (LEU) fuel. The fuel design is based on a monolithic uranium-molybdenum fuel alloy, containing 10% (w/w) Mo (U-10Mo), enclosed in Al-6061 cladding, with a diffusion/bonding interlayer composed of zirconium or aluminum-silicon alloy material. Annually, several metric tons of LEU are required for production of U-10Mo fuel elements for HPRRs. Argonne National Laboratory is developing means to recycle scrap from the fabrication of these fuels and to reprocess HPRR spent fuel. Both aqueous and pyrochemical flowsheets have been developed to do so. In aqueous processing of the spent fuel and fuel scrap, the Zr-bonding layer complicates the dissolution process due to the necessity of adding fluoride to the dissolver solution to negate a potential explosive reaction of the U-Zr compounds formed at the uranium/zirconium interface.

In this presentation, we will discuss the requirements for use of this fuel, both aqueous and pyrochemical processing schemes, and concerns associated with aqueous dissolution of the fuel and fuel scrap. The results of our study show that pyrochemical processing is the clear path forward for both scrap recovery and reprocessing of US HPRR spent fuel.

Notes:

Tuesday, May 19

Poster Session

Chair: Philip Wilk, LLNL

Notes:

Recycling of Metallic-Based Plutonium Residues: Conversion into PuO_2 for Aqueous Polishing

Sébastien Faure*, Stéphanie Saintignon, Danièle Cardona
CEA, DAM, VALDUC, F-21120 Is sur Tille, France

Plutonium purification, elaboration and casting processes produce residues. The recovery of residual plutonium in these by-products justifies the development of new recycling facilities.

The goal is to obtain easily disposable waste after plutonium separation.

This study focuses on three types of plutonium residues:

- anode heel residues from electrorefining process (massive metallic pieces, containing high amount of impurities),
- tantalum vessel (stirrers, funnels…),
- pieces of magnesia crucibles from electrorefining process.

The anode heel residues are recycled in a two steps process. Firstly, an oxidation step at 450°C under controlled O2 and H_2O atmosphere allows the conversion of metallic residues into impure PuO_2. Secondly, impure PuO_2 is dissolved in nitrofluorhydric and purified on anion exchange resins. Current development work aims at improving the high temperature oxidative step, in particular by increasing the amount of feed materiel together with a significant improvement of the kinetics.

Meanwhile, efforts are made to extend this method to other plutonium metal-based residues. Preliminary oxidation tests of plutonium metal deposited on tantalum demonstrated the efficiency of the process: more than 95 % of plutonium is easily separated leaving Ta vessel almost clean.

In the case of magnesia crucibles, plutonium that diffused into the MgO matrix during the electrorefining (on roughly 1mm) is difficult to recover by the oxidation process. However, recent trials show a good decontamination factor of Pu from MgO. The recovery of more than 75 % of plutonium content in these residues is possible.

Notes:

Qualitative and Quantitative Study of the Radiolytic Products of Bis-DGA UAM-069 used for Actinides (III) and Lanthanides (III) Co-Extraction Processes

H. Galán[1], A.G. Espartero[1], P. Prados[2], A. Núñez[1], M.T. Murillo[2], R. Sedano[2], J. de Mendoza[3]

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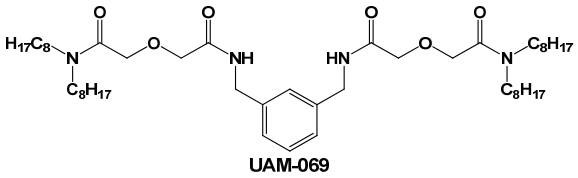
2/ Dpto. Química Orgánica, Facultad de Ciencias, Universidad Autónoma de Madrid. Cantoblanco-28049-Madrid SPAIN
3/ Institut Catalá d'Investigació Química (ICIQ) Avgda. Països Catalans, 16, 43007-Tarragona SPAIN

For nuclear hydrometallurgical separation process development, it is necessary to demonstrate the stability of the extracting systems, since it is well known that their radiolytic and hydrolytic degradation leads to undesirable effects (such as a decrease of selectivity, poorer phase separation, third phase formation, etc), in order to minimize the regeneration of the used solvent, the volume of secondary waste generated, and process costs. Recently, our laboratories have developed a new family of bis-diglycolamide molecules [1, 2] and have studied their stability against radiolysis and hydrolysis. One of the molecules studied, **UAM-069**, still showed high distribution coefficients after its irradiation at 1000 kGy [3]. Then, a comprehensive study was carried out to identify and quantify the sub-products formed during the irradiation process as well as the quantity of the remaining **UAM-069**.



UAM-069

This paper describes the qualitative and quantitative analyses of the irradiated **UAM-069** at integrated doses up to 1000 kGy (dose rate 4 kGy/h) using external ^{60}Co sources at NAYADE facility (CIEMAT site). A HPLC-MS qualitative analysis indicated the presence of seventeen degradation compounds. All the fragments were identified and synthesized. Their assigned structure was proven by comparing their retention times and mass spectra with those of the irradiated UAM-69 system. To complete this study, the An(III) and Ln(III) extraction properties of these fragments were assessed under the same experimental conditions as those used to evaluate the An(III) and Ln(III) extraction by irradiated **UAM-069**. This work was developed in the framework of the European Project ACSEPT (FP7) and ENRESA.

[1] Prados, P.; Murillo, M. T.; Espartero, A. G.; Sánchez-Quesada, J.; Almaraz, M.; Modolo, G.; de Mendoza, J. *European Patent 1923473 (21st May, 2008)*.
[2] Murillo, M. T.; Espartero, A. G.; Sánchez-Quesada, J.; de Mendoza, J.; Prados, P.; Solvent Extraction and Ion Exchange; 27, 110-134, 2009.
[3] A.G. Espartero, P. Prados, M.T. Murillo, J. Sánchez-Quesada, J. C. Iglesias-Sánchez, A. Núñez, J. de Mendoza; 10th International Exchange Meeting (10IEMP&T), OCDE/NEA; Mito (Japón) Octubre 2008.

Notes:

Combining TRUEX and TALSPEAK in One Process: Solvent Development

Artem V. Guelis [*] [(1)] **, George F. Vandegrift** [(1)] **and Gregg J. Lumetta** [(2)]

[(1)] *Argonne National Laboratory*
[(2)] *Pacific Northwest National Laboratory*

Separation of the minor actinides (Am, Cm) from the lanthanides at an industrial scale remains a significant technical challenge for closing a nuclear cycle. We describe here a chemical system in which a neutral extractant—octyl(phenyl)-N,N-diisobutyl-carbamoylmethylphosphine oxide or CMPO—is combined with an acidic extractant—bis-(2-ethylhexyl)phosphoric acid or HDEHP—to form a single process solvent for separating Am and Cm from the other components of irradiated nuclear fuel following the removal of U, Pu and Np. In this system, the extraction chemistry of CMPO dominate under conditions of high acidity (> 1 M HNO_3), resulting in co-extraction of the TRU and lanthanide elements into the organic phase. After suitable scrubbing steps, contacting the loaded solvent with a solution of DTPA/lactic acid at pH ~3 to 4 results in a condition in which the HDEHP chemistry dominates. We report here the influence of solvent formulation (e.g., CMPO and HDEHP concentrations, the effects of diluents and added modifiers) on the lanthanide/actinide separation factors. The extraction behavior of the actinides and some fission products will be presented also.

Notes:

Alternative Red-Ox Reagents in NPEX and TRUEX Processes of UREX+

Artem V. Guelis*, Delbert L. Bowers and George F. Vandegrift
Chemical Sciences and Engineering Division, Argonne National Laboratory
9700 South Cass Avenue, Argonne, IL 60439

In the frame of the Advanced Fuel Cycle Initiative (AFCI), we are developing alternative red-ox conditions to control the Np and Pu oxidation states in NPEX and TRUEX processes. The NPEX process includes co-extraction of Pu and Np with TBP. The TRUEX process separates lanthanides and actinides from the other components of spent nuclear fuel. The current technology utilizes sulfamic acid as a scavenger of nitrous acid in NPEX and iron (II) as reductant of Np in TRUEX. Both these reagents compromise the waste glass loading. The alternative red-ox conditions will be used for a design of the improved flowsheets of the NPEX and the TRUEX processes that will substantially decrease or eliminate the loading of the waste glass with undesirable sulfur and iron.

Separation of Waste Soluble Impurities from Plutonium Oxide

Michael Harland
Lawrence Livermore National Laboratory, Livermore, CA, USA

This oxide washing process along with the Loss-On-Ignition process is a major requirement in meeting the DOE 3013 Packaging standard. The Oxide Washer easily removes water soluble impurities from plutonium oxide that were generated from various processes such as, Direct Oxide Reduction (DOR), Molten Salt Extraction (MSE), Electrorefining (ER) and Salt Scrub. The washing process is performed by using distilled water (DI) to remove water soluble impurities such as calcium chloride from impure oxides. The impure oxide is loaded into the wash chamber and DI water is added and the drum/chamber is rotated by a motor to thoroughly mix the oxide and the water. This process is performed at various speeds and inclinations of the chamber. The rinse water with the impurities is removed via a centrifugal action similar to a clothes washing machine. The water is filtered with an ash-less filter paper to remove the fine particulates. The filter paper is calcined along with the washed oxide to remove the excess moisture. The chloride free oxide is further processed in the Material Processing Laboratory using the Loss-On-Ignition (LOI) process. The filtrate liquid is solidified and disposed of as Transuranic (TRU) waste.

Notes:

Hijacking Proteins with Plutonium

Mark P. Jensen*

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The exceedingly toxic element plutonium did not exist in biologically relevant amounts on earth until its isolation in 1941. As a consequence, organisms have had no opportunity to develop specific mechanisms to handle Pu poisoning, and in the body Pu adventitiously follows biochemical pathways dictated by its particular chemistry. Despite years of study, a mechanistic, molecular-level understanding of the pathways that allow transuranium elements such as plutonium to bio-accumulate was never achieved. In an effort to describe and possibly block the pathways of cellular plutonium trafficking, we have been examining the mechanisms of Pu uptake by mammalian cells and the nature of the important plutonium-protein complexes. We have identified a particular isomer of a stable Pu-protein complex that is recognized by cells and incorporated by endocytosis just like the naturally-occurring metalloprotein. With this specific target, strategies to mitigate Pu toxicity by pre-empting Pu incorporation by cells appear within reach.

Argonne National Laboratory's work was supported under U.S. Department of Energy contract DE-AC02-06CH11357.

Notes:

Electrochemical Reduction of Plutonium Oxide

Arfon Harris Jones (Arfon.Jones@awe.co.uk)
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Calciothermic reduction of plutonium oxide to plutonium is a high yielding process giving high quality product. However, the process is wasteful in that a large quantity of spent calcium chloride salt and waste hardware is produced. This waste arises from the limited solubility properties of calcium oxide in calcium chloride.

The Fray Farthing Chen (FFC) method is an electrochemical technique where an oxide is made the cathode in a molten salt cell. Oxygen is evolved directly at an anode whilst the cathode is reduced to metal. Potentially, the volume of waste generated by the FFC method is considerably less than the calciothermic method. This is because calcium oxide does not accumulate in the molten salt.

The FFC process has previously been reported on a number of materials including uranium oxide and mixed oxide (MOX) nuclear fuel. In these cases the oxide has been in the form of pressed and sintered pellets. Such pressing is an undesirable additional processing step.

This presentation reports upon studies of the FFC reduction of oxides, such as titanium oxide and cerium oxide, in powder form as a simulant to the reduction of plutonium oxide. These simulant studies were conducted using a variety of molten salt systems and cell configurations.

Plutonium oxide FFC reduction was also carried out at smaller laboratory scale. Comparisons with previously reported studies are drawn and the applicability of the FFC method to plutonium oxide discussed.

Notes:

Aspen/OLI Model of Waste Evaporation and Neutralization Process for MOX Waste Solidification Building

James E. Laurinat*
Savannah River National Laboratory, Aiken, SC, USA

The Mixed Oxide Fuel Fabrication Facility (MOX or MFFF) currently under construction at the Savannah River Site includes a Waste Solidification Building (WSB), which will house the process for converting aqueous wastes from the MFFF and the proposed Pit Disassembly and Conversion Facility (PDCF) into cements for long-term storage in either onsite repositories or at the Waste Isolation Pilot Plant (WIPP). The WSB process design required flowsheet modeling of the mixing tanks, batch evaporators, and neutralization tanks included in the WSB process. A steady state computer simulation was developed, using Aspen Plus® to model the mixing tanks and evaporators and OLI® to model the chemistry. In the Aspen Plus simulation, the batch evaporators are modeled as multistage flash evaporators to account for changes in the vapor overheads composition during each batch. The WSB process includes separate mixing tanks and evaporators for low activity and high activity wastes. Limits are placed on the uranium concentration in the condensate from the low activity waste evaporator and on the total salt concentration in the condensate from the high activity waste evaporator to ensure that solids do not precipitate in either the evaporators or the transfer lines from the evaporators to the neutralization tanks. In addition, the chloride concentration in the condensate from the low activity waste evaporator is limited to control corrosion. To eliminate the need for recycle loops, feed forward controls are used to apply these limits. The OLI chemistry was modified to add precipitation chemistry for sodium diuranate, which was absent from the standard OLI databank. The simulation was used to predict flow rates, compositions, and heating duties for feeds from the PDCF and alternate feedstocks. The simulation is configured so that both inputs and outputs appear on a Microsoft Excel® spreadsheet.

Notes:

Reduction of Tetravalent Plutonium in the Presence of Acetohydroxamic Acid

[1]Peter Tkac, [2]Martin Precek, [3]Alena Paulenova

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Acetohydroxamic acid (AHA), proposed as a salt-free reagent for preventing extraction of Pu and Np by tri-*n*-butyl phosphate in modified PUREX/UREX process, is a capable reductant of their hexavalent and a strong complexant for their tetravalent cations. Despite the complexation constants for Pu-AHA complexes are very high, the hydrolytic instability of hydroxamic group under acidic conditions cause a slow degradation of AHA followed by slow decomposition of Pu-acetohydroxamate complex and reduction of Pu(IV) to Pu(III). Therefore, the behavior and partitioning of Pu between the organic and aqueous phase in the proposed extraction systems containing acetohydroxamic acid can be difficult to predict due to the complexation and oxidation-reduction reactions, which can occur. Since the knowledge of speciation and distribution relationship of plutonium in the solvent extraction system is very important for a future successful design of separation flowsheets, characterization and understanding of all relevant chemical directions of plutonium is essential.

Reduction of Pu(IV) in the presence of AHA was monitored by Vis-NIR spectroscopy. All complexes between Pu(IV) and AHA have very intense absorption in the region of 400-700 nm; therefore, for easier evaluation of the reduction process, all experiments were performed under a low AHA:Pu(IV) ratio, where only the monoacetohydroxamate complex of Pu(IV) and Pu, non-complexed with AHA, are present in relevant concentrations. In order to characterize the reaction mechanism, the experimental absorption data were analyzed at several wavelengths. Time dependent concentrations of all absorbing species were resolved using the extinction coefficients for Pu(IV), Pu(III) and Pu(IV)-AHA complex. The extinction coefficients for Pu(IV)-acetohydroxamate complex were resolved from the experimental data obtained by absorption spectroscopy using the chemical equilibrium modeling software FITEQL 4.0. Based on the fitting of obtained experimental data it was proposed that reduction of tetravalent plutonium in the presence of AHA is a consecutive reaction, where decomposition of Pu-monoacetohydroxamate complex follows second order reaction mechanism. Reduction of Pu(IV) is faster at lower concentration of AHA, due to stabilization of Pu(IV) by formation of higher Pu(IV)-acetohydroxamate complexes. However, after some reaction period, reduced Pu(III) can be re-oxidized back to Pu(IV). The re-oxidation reaction occurs sooner for lower initial ratio of AHA:Pu(IV) and can be explained by oxidation of Pu(III) with nitrous acid.

Notes:

Complexation and Extraction of Tc(II) with Novel Methylresorcin[4]Arene Derivatives

Patricia Paviet-Hartmann*, Ana Nunez Gomez-Aleixandre, Joshua Pak*****
Amparo Glez Espartero, Frederic Poineau*, Amber Wright*, Edward Mausolf***
Ian Kihara*, and Jeff Hess*****

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**** Idaho State University, Department of Chemistry, Pocatello, ID 83209 USA*

The UREX+ process is currently being developed to complement or replace the PUREX process used worldwide to separate U(VI) and Pu(IV) from used nuclear fuel by tri-butyl phosphate (TBP) solvent extraction. In the UREX+ process, acetohydroxamic acid (AHA) will be added to the scrub stream to prevent the extraction of Pu(IV) and Np(IV) by TBP. Until recently, it was thought that AHA did not affect the technetium speciation but recent work performed in our laboratory has shown the formation of $[Tc(II)(NO)(AHA)_2H_2O]^+$ abbreviated as $(Tc(NO)AHA)^+$. The potential formation of $(Tc(NO)AHA)^+$ during reprocessing may strongly impact the fate of technetium in the nuclear fuel cycle. We have observed colloid formation when this technetium species was contacted with TBP, which will ultimately impede any extraction scheme. In addition, because of the small activities of technetium-99 relative to other fission products, like cesium-137 or strontium-90, and its long half-life ($t_{1/2}$ = 2.1•10^5 yrs), technetium-99 is one of the key isotopes that should be analyzed in the radioactive liquid waste streams from the reprocessing industry, where the largest concentrations are expected. The goal of our ongoing separation efforts is ultimately to develop a process to efficiently concentrate and recover technetium from the UREX aqueous product stream and convert the technetium to a metal or oxide form for incorporation in potential waste-forms. We are reporting the extraction of $[Tc(II)(NO)(AHA)_2H_2O]^+$ complex by new designed methylresorcin[4]arene derivatives as well as commercially available crown ethers from 18-crown-6 to 24-crown-8 in ring size and of varying derivatization. Several organic diluents with different dielectric constants are used to enhance the distribution coefficient of technetium(II). The experimental efforts are focused on determining the best extraction conditions by varying the macrocomponds nature and concentration, and the organic phase composition.

Notes:

Removal of Solids from Highly Enriched Uranium Solutions Using the H-Canyon Centrifuge

Tracy S. Rudisill*, Fernando F. Fondeur, and John H. Gray
Savannah River National Laboratory, Aiken, SC

The HB-Line facility at the Savannah River Site is currently used for the disposition of both highly enriched uranium (HEU) and plutonium-containing scrap and residues. These materials are dissolved in a slab tank dissolver and transferred to H-Canyon tanks for interim storage and subsequent disposition. Impure materials containing HEU were recently dissolved which contained impurities that did not dissolve during the dissolution flowsheet development. Following each dissolution cycle, the contents of the dissolver were transferred through a bag filter to a product hold tank and then to an H-Canyon storage tank. Subsequent analyses of samples from the canyon tanks showed the presence of U-containing solids indicating that the bag filter may have been penetrated when the solution was transferred to the product hold tank. In preparation for the dissolution of Pu-containing materials in HB-Line, the HEU solutions had to be transferred to provide storage space. The proposed plan was to centrifuge the solutions to remove solids which may present downstream criticality concerns or cause operational problems with the 1st Cycle of solvent exaction due to the formation of stable emulsions.

An evaluation of the efficiency of the H-Canyon centrifuge concluded that a sufficient amount (> 90%) of the solids in the solutions would be removed to prevent any problems. This conclusion was based on the particle size distribution of the solids isolated from samples of the solutions and the calculation of particle settling times in the centrifuge. The particle size distributions were calculated from images generated by scanning electron microscopy. The mean particle diameters for the distributions were 1-3 μm. A significant fraction (30-50%) of the particles had diameters which were < 1 μm; however, the mass of these solids was insignificant (< 1% of the total solids mass) when compared to particles with larger diameters. The settling times calculated for the H-Canyon centrifuge showed that particles with diameters less than 1 to 0.5 μm would not have sufficient time to settle. For this reason, the use of a gelatin strike was recommended to coagulate the submicron particles and facilitate their removal from the solution. Incomplete removal of particles with diameters < 1 μm would not have a detrimental effect on the HEU purification. Particles with diameters > 1 μm accounted for > 99% of the solid mass and would be efficiently removed by the centrifuge; therefore, the formation of emulsions during solvent extraction operations was not an issue.

Notes:

LLNL's Materials Process Laboratory
DOE Standard 3013 Packaging of Stabilized Materials

Kevin P Silveira*
Lawrence Livermore National Laboratory, Livermore, CA, USA

The Materials Process Lab (MPL) at LLNL performs a variety of critical missions that support research and development, as well as current programs. There is a range of capabilities including; dismantlement of former weapons parts and conversion of recovered materials, pyrochemistry, stabilization and packaging, as well as small sample preparation. Conversion of materials includes metal to hydrides, nitrides, oxides, and chlorine compounds. Pyrochemistry includes Direct Oxide Reduction, Molten Salt Extraction, Electro-Refining as well as other processes in both stationary and induction furnaces. The MPL also has a casting capability to support both pyrochemistry and small sample preparation. There is also development work with automated processing. The stabilization and packaging effort supports the NNSA's plan for "Complex Transformation". Materials are calcined at high temperatures to remove impurities of concern to the packaging requirements, and metals are consolidated. Material is packaged using British Nuclear Fuels Limited (BNFL) designed bagless transfer system, which consists of a laser welding system. Small samples are processed on a CNC mill and CNC lathe to support various sample forms including; gas gun, tensile, electron microscopy, density, dilatometer, and compression.

Notes:

Uranium Characterization in K Basin Sludge

Sergey Sinkov*, Cal Delegard, Andy Schmidt, Jeff Chenault
Pacific Northwest National Laboratory, Richland, WA, USA

Jim Sloughter, Ron Baker
CH2M Hill Plateau Remediation Company, Richland, WA, USA

Irradiated uranium metal fuel particles present in Hanford Site K West Basin sludge can create hazardous H_2 atmospheres during handling, immobilization, or subsequent transport and confined storage operations by its oxidation/corrosion in water. A thorough knowledge of the U metal concentration in sludge therefore is essential to successful sludge management and waste process design. Uranium metal oxidation products include uranium dioxide, UO_2 [U(IV)], formed via transitory uranium hydride, UH_3. With exposure to dissolved atmospheric oxygen, the UO_2 converts to hexavalent uranium as metaschoepite, $UO_3 \cdot 2H_2O$. The oxidation of UO_2 to $UO_3 \cdot 2H_2O$ potentially can proceed through ianthinite, $[U_2(UO_2)_4O_6(OH)_4(H_2O)_4](H_2O)_5$, in which 2/3 of the uranium is in the U(VI) oxidation state and 1/3 is in the U(IV) state. Like UH_3, ianthinite is a transitory phase and little is present in K Basin sludge. Further reactions of metaschoepite to form other U(VI) phases such as becquerelite [Ca-U(VI)] and soddyite [U(VI) silicate] occur with extended time. The UO_2 particle density is ~11.0 g/cm^3 while ianthinite, metaschoepite, and other U(VI) phase particle densities are about ~5.0 g/cm^3. Significant solids volume increase therefore occurs as the UO_2 oxidizes to form U(VI) phases. The alteration of sludge solids also can affect sludge particle agglomeration and cementation and thus sludge mobility and strength under extended storage conditions. Knowledge of uranium oxidation state distribution provides information on the extent of conversion of the dense and reactive U(IV) phase UO_2 to the less dense and relatively non-reactive U(VI) phases.

Methods to measure U metal concentration and oxidized uranium oxidation state distribution have been developed with simulated sludge and proven with genuine K Basin sludge. The uranium metal detection limit is ~0.004 wt% based on the testing performed with actual sludge, thus meeting the goal detection limit of 0.03 wt% in the presence of up to 1000-fold higher total uranium concentrations (i.e., up to 30 wt% and more uranium). Oxidized uranium oxidation state distribution is conveniently measured by spectrophotometry of the sludge leachates prepared in concentrated phosphoric acid in the initial steps of U metal concentration determination. Extended testing has shown that non-irradiated uranium oxidation state distribution in the H_3PO_4 is stable for months if kept in the dark but shows slow conversion from U(VI) to U(IV) under illumination. Radiolysis shows an opposite trend.

Notes:

NMR Analysis of the TALSPEAK Process Solvent after Alpha-Irradiation with Plutonium-238

Lanée A. Snow,* Bruce K. McNamara, Gregg J. Lumetta, and Susan A. Jones
Pacific Northwest National Laboratory, Richland, WA, USA

The TALSPEAK Process is being investigated in the U.S. for separating the lanthanide elements from the actinide elements (Np, Pu, Am, Cm), so that the latter can be transmuted into short-lived or stable isotopes. The TALSPEAK solvent consists of di-(2-ethylhexyl) phosphoric acid (HDEHP) dissolved in dodecane. Radiolytic degradation of this solvent is one aspect of the TALSPEAK chemistry that is little understood. An experiment was initiated to investigate the effects of alpha irradiation on the TALSPEAK solvent. To supply the alpha dose, plutonium-238 was extracted into the solvent phase. Sufficient Pu-238 was added to produce a dose rate of ~1.5 Gy/min. Samples of the degraded solvent were analyzed by multinuclear NMR techniques, the results of which will be summarized in this paper. Spectra of likely degradation products will be compared to the spectra of the alpha-degraded solvent.

Notes:

Application of Formohydroxamic Acid in Nuclear Processing: Synthesis and Complexation with Technetium 99

Amber Wright, Keri Campbell, Edward Mausolf, Frederic Poineau, Patricia Paviet-Hartmann

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Acetohydroxamic acid (AHA) is an organic ligand planned for use in the UREX process. It reduces neptunium and plutonium, and the resultant hydrophilic complexes are separated from uranium by extraction with tributylphosphate (TBP) in a hydrocarbon diluent. Hydroxamic acids undergo hydrolysis to hydroxylamine and the pertinent carboxylic acid. The reported reduction potentials of AHA and pertechnetate (TcO_4^-) indicate that it may be possible for AHA to reduce technetium, altering its fate in the fuel cycle. At UNLV, it has been demonstrated that TcO_4^- undergoes reductive nitrosylation by AHA under a variety of conditions. The resulting divalent technetium is complexed by AHA to form the pseudo-octahedral *trans*-aquonitrosyl (diacetohydroxamic)-technetium(II) complex ($[Tc^{II}(NO)(AHA)_2H_2O]^+$). During recent discussions of the UREX process, it has been proposed to replace AHA by formohydroxamic acid (FHA). Preliminary results on the synthesis of FHA and its complex formation with Tc are presented along with the characterization of FHA crystals achieved by NMR and IR spectroscopy. The complex formation between technetium and FHA has been studied by UV-visible spectrophotometry and compared to the complexation of AHA with Tc.

Notes:

Wednesday, May 20

Technical Session II: Actinide Solution Chemistry

Chair: Ken Nash, Washington State University
Co-Chair: Tracy Rudisill, SRNL

Notes:

Potentiometric Determination of Uranyl-Nitrate Stability Constants by Competition Method

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The basic chemistry of the uranyl ion in a nitrate rich media, such as in an aqueous reprocessing stream, has been explored for decades with only a handful of reliable data sets produced. Information on the mononitrato species is incomplete, while the stability constants for the higher nitrato species have yet to be firmly established. Based on the work of Ahrland, this study seeks to complement the existing data set by determining which uranyl nitrate species exist and their stability constants. This was done by competing the relatively weak nitrate complex against the stronger acetate complex at constant ionic strength and temperature. Initial results are presented.

Spectrometric Measurements of Metal-DTPA Complexes in Lactic Acid Media under TALSPEAK-like Conditions

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The combination of DTPA and lactic acid in the aqueous phase of the TALSPEAK solvent extraction system has proven to be successful for effectively separating trivalent actinides from lanthanides. The exact role of lactic acid in this system is not perfectly understood but there are many indications that lactic acid may have other parts than just to be an inert pH buffer. The existence of a mixed aqueous ternary complex between metal, DTPA and lactic acid has been suggested but no concrete evidence for this complex has been found. In this study we have investigated the complexes of Am, Ho and Nd with DTPA in lactic acid media. The spectrometric titrations of the binary systems metal-DTPA and metal-lactate were carried out for background purposes and results indicate that there are two DTPA-metal complexes with different spectrometric traces and up to 4 metal-lactate species, which is in accordance with previous investigations by other research teams. The metal-DTPA spectra taken in lactate media are distinctly different from those measured in nitrate media and reasons for the discrepancy will be discussed.

Notes:

Preorganization and Rigidity: New Ligands for Trivalent Actinides/Lanthanides Separation

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The separation of trivalent actinides from lanthanides is a challenge that's getting more visibility as interest in nuclear energy grows and fuel reprocessing becomes a real option. Actinide/lanthanide separation is crucial to closing the nuclear fuel cycle and yet is still not efficiently accomplished. Multiple ligands based on the hardness or the softness of the donor groups have been developed over the years to selectively extract the trivalent actinides (and in some cases the lanthanides), but typically, all show severe extraction limitations for pH lower than 1. When cation exchange is the mode of extraction, performance drops drastically (3 orders of magnitude for each pH unit) as the acidity of the system increases. Most efforts have been dedicated to change the acidity of the ligands, and while some improvements have been observed, there are no compounds that can achieve selective separations at pHs adequate for nuclear fuel reprocessing. The approach proposed here is to not modify the pH of the proton donor groups in the extractant but to preorganize them to increase the binding constants developed between the cation and the ligands. This increase is expected to lead to greater extraction, affording an improved performance at higher acidity. We conducted molecular modeling studies to determine the best preorganization of dithiophosninic acids and the corresponding scaffold. The best linkers between 2 dithiophosphinic acid functional groups were determined and results will be presented along with preliminary extraction results.

Research sponsored by the Advanced Fuel Cycle Initiative, Office of Nuclear Energy, U.S. Department of Energy, under contract No. DE-AC05-00OR22725 with Oak Ridge National Laboratory, managed and operated by UT-Battelle, LLC.

Notes:

Ternary Complexes for Am/Cm Separation in a TALSPEAK Framework

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Chemical separations of Am from Cm have typically relied either on chromatography to amplify the small chemical differences between Am(III) and Cm(III) through repeated separation, or on accessing the higher oxidation states of Am to affect a separation of Am from Cm(III). We are exploring a third way to separate Am from Cm. It exploits the slightly different ionic radii of these ions through formation of ternary actinide-DTPA-ligand complexes in a TALSPEAK-like aqueous phase that has been augmented with a small ternary ligand size-matched to the $Am(DTPA)^{2-}$ complex but too large to bind $Cm(DTPA)^{2-}$ effectively. Such complexes would allow direct Am/Cm separation by solvent extraction. Our investigations of DTPA-auxiliary ligand combinations suggest that they do not have the right match of rigidity and steric bulk to accomplish an Am/Cm separation. However, other aqueous aminopolycarboxylate-ligand combinations may be effective Am holdback agents.

Argonne National Laboratory's work was supported by the U.S. Department of Energy, Office of Nuclear Energy, under contract DE-AC02-06CH11357.

Notes:

Enhancing Understanding of TALSPEAK Chemistry (or Maybe Displacing TALSPEAK with Better Options?): Sigma Team Research at WSU

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Exercising the transmutation options for advanced nuclear fuel cycles will demand careful consideration of the group separation of trivalent actinides (Am and Cm, or maybe just Am) from fission product lanthanides. The two viable options for accomplishing this separation at an industrial scale (via aqueous processing options) involve 1) the application of ligand donor systems containing atoms softer than oxygen (N, S, Cl^-) or 2) adjusting the oxidation state of Am to one of its redox-unstable upper oxidation states. The former option offers flexibility of design, but reduced donor strength and questions about reagent stability. The latter option will of necessity demand rapid separations and quite probably unconventional methods and materials. In either case, there are aspects of the chemistry that have not been explored in depth; these gaps in understanding translate into opportunities for developing revolutionary new approaches to completing this difficult separation. Starting from the historical literature, actinide separation research at WSU began several years ago in research funded through the Nuclear Energy Research Initiative (NERI) and continues today. A primary focus of NERI studies at WSU has been on addressing the fundamental details of the TALSPEAK process (among other topics). Initial Sigma Team studies at WSU have built on this focus with studies of both water-soluble and organophilic soft donor complexants in the context of basic TALSPEAK chemistry. As early work has progressed slowly, new focus areas are receiving attention. In this presentation, a brief summation of the essential features of our TALSPEAK-focused investigations will be presented, complemented by a description of the concepts and motivations of our more exploratory next steps in soft donor and redox mediated separations.

Notes:

TALSPEAK Chemistry in the Absence of DTPA: Nd(III) Speciation in the Organic Phase Helps to Identify One More Previously Unaccounted-for Player in this Process

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Travis S. Grimes, Mikael Nillson, Kenneth L. Nash
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A recent review of the development and operational characteristics of the TALSPEAK process [1] pointed out that the likely existence of ternary complexes both in aqueous and in organic phases [$M(Lact)(DTPA)_{aq}$, and $M(Lact)(HDEHP)_{org}$] represents an unknown aspect of TALSPEAK. Understanding the thermodynamic features of these complexes is expected to explain some significant discrepancies between calculated and experimentally observed extraction profiles of Nd(III), Eu(III) and Am(III). In our work we've chosen to study Nd(III) speciation in the organic phase via extraction of this metal cation from the DTPA-free lactic acid/sodium lactate solution in search of the proposed $M(Lact)(HDEHP)_{org}$ complex. Elimination of DTPA from the extraction process allowed us to simplify calculation of expected distribution ratios for Nd(III) for the selected experimental conditions and their comparison with actually observed extraction profiles.

Key observations of our study can be summarized as follows:

- No spectral evidence for the lactate penetration into an inner coordination sphere of the $Nd(III)(DEHP{\cdot}HDEHP)_3$ complex) in the organic phase is found in a wide range of lactic acid to sodium lactate ratios in the aqueous phase.
- The presence of sodium lactate in the aqueous phase leads to partial conversion of HDEHP to NaDEHP in the organic phase and results in depletion of effective concentration of the extractant available for the Nd transfer to the organic phase.
- Sodium ion extraction by HDEHP from the lactic acid-sodium lactate-sodium perchlorate solution at C_{Na+} = 1 M (prior to introduction of Nd(III) into the extraction system) is more pronounced compared with the published data on Na^+ extraction from 1 M $NaClO_4$ [2]. Introduction of Nd(III) into this Na^+-preequilibrated system results in partial redistribution of the organic-bound sodium back to the aqueous phase and thus indicates a significant competition between Nd^{3+} and Na^+ for the extractant.
- Accounting for a partial transformation of HDEHP into its sodium form after contact with sodium lactate containing aqueous phase allows to achieve much better correspondence between experimentally observed and calculated extraction profiles of Nd(III) as a function of pH compared with simpler models not considering this process.

[1]. Nillson M., and K.L.Nash. *Solvent extraction and Ion Exchange*, **25**: 665-701, 2007.
[2]. Lundquist R., J.-F. Lu, and I.Svantesson. *Acta Chemica Scandinavica*, **A37**, 743-753, 1983.

Notes:

Development of a Sequential Separation Method for up to Five Actinides from Nitric Acid Media Using Chromatographic Extraction Resins

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Actinides exist globally at very low levels in the environment due to fallout from former nuclear tests, accidental releases, etc. There is therefore a need to determine low concentrations of actinides in the environment. This can be achieved using sensitive analytical methods such as alpha spectroscopy and mass spectrometry (e.g., Thermal Ionization Mass Spectrometry). However, these analytical techniques require highly robust elemental separations in order to eliminate the bulk sample matrix from the trace concentrations of transuranium elements in the sample. Our objective was to first extract and separate the actinides from the bulk sample matrix and then elute those actinides in groups or sequentially. The resulting matrix-free analyte fractions could then be further purified or separated, as required, prior to analysis.

Extractions and separations were performed using commercially available extraction chromatographic columns (N,N,N',N'-tetra-n-octyldiglycolamide (DGA) Resin and others from Eichrom Technologies, Inc.). Sequential actinide separation and group separations were performed. In both cases, we were able to separate uranium and thorium, which are predominant in environmental samples, from less abundant transuranium actinides.

The efficiency of both extraction and separation was optimized using different acid concentrations and a balance of oxidizing, reducing and complexing agents. Automated column separations and batch contact experiments were used to tune these variables. The result of these experiments will be further discussed and the extraction and separation results on actual dissolved soil matrix will be presented.

Notes:

Selective Separate Actinide Ions Using N,N-di-2-ethyl-hexyl-3-oxa-glutaramic Acid

Guoxin Tian* and **Linfeng Rao**
Lawrence Berkeley National Laboratory
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Phone: 1 510 486 5141, Fax: 1 510 486 5596, email: gtian@lbl.gov

Based on the results of a series of thermodynamic and structural studies, an acidic amide ligand, N,N-di-2-ethyl-hexyl-3-oxa-glutaramic acid has been developed and found to have the potential for separating actinides. Extraction experiments show that N, N-di-2-ethyl-hexyl-3-oxa-glutaramic acid has good extractability for actinides in all oxidation states (III, IV, V, and VI). It could form neutral complexes with actinides that have great solubility in conventional organic solvents such as kerosene, significantly reducing the problem of third phase formation that complicates many processes developed for fuel reprocessing. After being extracted into the organic phase, Pu and Np are not separated from U, which significantly improves the anti-proliferation ability of the process. Besides, because the extractant contains only C, H, O, and N elements, no secondary solid wastes will be generated from the extractant after combustion. The separation process using this extractant is more environmentally-benign. The structural information of the complexes has also been obtained by X-ray diffraction study on the crystals of actinides complexes with N,N-di-methyl-3-oxa-glutaramic acid.

This work was supported by the Director, Office of Science, Office of Basic Energy Sciences, Division of Chemical Sciences of US Department of Energy under Contract No. DE-AC02-05CH11231 at Lawrence Berkeley National Laboratory (LBNL).

Notes:

Thursday, May 21

Technical Session III:
Aqueous and Pyrochemical Processing

Chair: James McNeese, LLNL
Co-Chair: Sharon Slade, AWE

Notes:

Processing of Residues for Offsite Shipment at LLNL

James A. McNeese
Lawrence Livermore National Laboratory

The majority of the plutonium and uranium holdings at the plutonium facility at LLNL are being packaged for shipment offsite. This talk discusses the residues and the processing sequences that are required to meet shipping requirements as well as receiving site acceptance criteria. The facility has multiple pyrochemical furnaces, calcinations furnaces, and a small aqueous recovery capacity that are being employed to consolidate and stabilize materials. Also involved in the process is the attendant non destructive assay measurements and packaging quality assurance that ensure that the material is safe for shipment and properly measured.

Notes:

Tantalum Corrosion and Ceramic Adherence with Plutonium Titanium Alloys

Sharon Slade*, Tim Paget and Robert Watson
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During the pyrochemical processing of plutonium titanium alloys several issues have arisen including the corrosion of tantalum stirrer blades and the adherence of ceramic fragments to metal surfaces.

Studies to identify the mechanism of tantalum corrosion have been undertaken. Microscopy studies of a corroded stirrer have shown a plutonium/titanium/tantalum interlayer on the surface of the stirrer blade and whole grains appear to be removed from the tantalum surface. A series of trials using titanium coated tantalum have been performed to study the interaction of the two metals with heat cycling. Inter-diffusion of the two metals has been noted during progressive heat cycles. This layer of inter-diffused metal may, in the plutonium system, be creating titanium rich areas around the grain boundaries of the tantalum which are then easily dissolved by the plutonium leading to the severe corrosion noted.

Adherence of the ceramic to the metal has been found on several occasions when no salt has been present; this could be due to the titanium in the plutonium having a greater wetting effect on the crucibles than plutonium on its own. Re-casting of ceramic encrusted billets through a tantalum strainer into a tapered crucible has been successfully employed to recover the metal. Trials of casting plutonium titanium alloys into a small quantity of salt have also proved successful indicating that the salt is coating the ceramic and preventing wetting by the metal.

The problems encountered during the pyrochemical processing of plutonium titanium alloys have been investigated to determine their mechanisms and simple solutions put into place to reduce their effects

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Notes:

Americium-Beryllium Neutron Source Dissolution

Dr. Jacqueline E. Fonnesbeck*, Jeffrey F. Berg, Mary L. Adamic, Dr. Kevin P. Carney
Idaho National Laboratory, Idaho Falls, ID, USA

Idaho National Laboratory (INL) has been examining Americium-Beryllium (AmBe) neutron sources for their characteristic forensic signatures. These neutron sources are a mixture of americium oxide and beryllium metal. Small sample sizes (~2-10 mg) have been dissolved for chemical analysis using a sequential dissolution that dissolves americium preferentially from the beryllium in order to identity which phase (oxide or metal) potential signatures may be associated. INL's focus is to develop methods to sample, dissolve, and analyze these sources in order to distinguish source types between manufacturers. Samples were dissolved and analyzed for their elemental, isotopic and trace impurity composition. This presentation will discuss the effectiveness of these methods to selectively dissolve several AmBe neutron sources.

Actinide Fuel Dissolution Methods

Jeffrey F. Berg*
Idaho National Laboratory, Idaho Falls, ID, USA

Idaho National Laboratory (INL) is participating in a national program to develop fuels for use in transmuting long-lived transuranic actinide isotopes. This program is part of the Advanced Fuel Cycle Initiative (AFCI). INL's focus is to develop actinide metal and nitride fuels for transmutation. The development of fuels for minor actinide transmutation systems presents many technical challenges. Fabricated fuels must be dissolved and analyzed for composition and impurities. Post Irradiation Examination (PIE) of these fuels requires dissolution in a shielded facility in order to evaluate fuel performance. This presentation will discuss the methods employed for the dissolution of these actinide fuels.

Notes:

A Path Forward for Recovery of Purified ^{241}Am from Hydrochloric Acid Plutonium Processing Operations

Louis D. Schulte

Plutonium Materials Technology Division: Group PMT-2

Los Alamos National Laboratory

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Over the years from 1979 to 1984, 6.5 kg of ^{241}Am were purified (as AmO_2) at Los Alamos National Laboratory. This historical work utilized aged, reactor grade plutonium metal as feedstock, and also produced PuO_2 for the Fast Flux Test Facility in Hanford. External sales of this recovered ^{241}Am were handled through the Oak Ridge National Laboratory National Isotope Center, a business office run for the Department of Energy Isotope Program. The pool of available ^{241}Am in the USA has been depleted to nearly zero over the intervening years, and there is no new domestic supplier. An ongoing need exists for ^{241}Am to be used primarily in americium-beryllium neutron sources for oil and gas well logging. Additional common uses for ^{241}Am include smoke detectors, environmental studies, and demonstration of nuclear fuel burnup and recycling processes.

About 10 years ago, processes were developed at LANL to selectively remove Am and Pu from 5-7 M hydrochloric acid plutonium processing effluent streams. Due to changes in mission needs and priorities, the process was never fully implemented. The process utilized an extraction chromatography resin loaded with di-(4-t-butylphenyl)-N,N-diisobutylcarbamoylmethylphosphine oxide and diamyl amylphosphonate. High alpha activity HCl process effluents (over 1 Ci/L) were reduced in activity by 99 to 99.99% for more than 20 full scale process runs. The Am & Pu loaded on the resin could be effectively stripped using 0.1 M HCl and 0.1 M HCl/0.1 M oxalic acid eluent solutions. Modest alteration of the demonstrated technology could allow recovery of pure ^{241}Am from pyrochemistry residues processed in existing hydrochloric acid plutonium recovery operations. A large number of weapons plutonium molten salt extraction residue items that are rich in americium are available to provide an efficient feedstock for a campaign to recover ^{241}Am at large scale. Application of this technology would also have a significant impact on the volume of actinide residues that must be stored in a protected vault, and in the quantity of actinides discarded to the environment.

Notes:

Fabrication of Minor Actinide Bearing Nuclear Fuel

Stephen P. Willson*, Richard E. Mason, David A. Costa, John T. Dunwoody
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Nuclear fuel pellets containing minor actinide constituents have been fabricated at Los Alamos National Laboratory and irradiated in the Advanced Test Reactor at Idaho National Laboratory. The purpose of these experiments is to explore the fabrication constraints and irradiation performance of various potential nuclear fuel forms for use in nuclear fuel recycle. It is anticipated that further research will reveal the acceptable range of variation in starting feedstock parameters for production of suitable recycled nuclear fuel containing minor actinides. Determination of the range of inputs that are amenable to consistent fuel fabrication will assist in determination of desired outputs from an actinide separations campaign.

The Chromatographic Separation of Trace Actinides from Large quantities of Uranium

Daniel R. McAlister* and E. Philip Horwitz
PG Research Foundation, Inc., 1955 University Lane, Lisle, IL 60532 USA

This work describes the chromatographic separation of trace amounts of actinides (mg or less) from large quantities of Uranium (g or kg). The separation method employs an extraction chromatographic material containing the tetra(n-octyl)diglycolamide (TODGA) extractant to selectively retain Actinium, Thorium, Neptunium and Plutonium, while rejecting Uranium. The separation can be carried out at relatively low nitric acid concentrations (1-2 M HNO_3) with feeds containing 0.1-0.2 M Uranium. Applications of this separation include isolation of ^{234}Th from ^{238}U and ^{229}Th from ^{233}U. ^{234}Th could be a useful radiotracer, providing a continuous supply of radiothorium from natural or depleted uranium. ^{229}Th is the source material for ^{225}Ac and ^{213}Bi, radionuclides with promising applications in nuclear medicine.

Notes:

H_2 Attenuation in K Basin Sludge from the Uranium Metal – Water Reaction

Cal Delegard*, Sergey Sinkov, Andy Schmidt
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Jim Sloughter, Ron Baker
CH2M Hill Plateau Remediation Company, Richland, WA, USA

Uranium metal reacts with water in Hanford Site K Basin sludge to form transitory UH_3 then UO_2 and H_2. Because H_2 is flammable, its release into the gas phase above K Basin sludge during storage, processing, immobilization, shipment, and disposal is a nagging operational safety concern. In addition, sludge loading for shipment to the Waste Isolation Pilot Plant as remote-handled TRU waste is limited by the total H_2 generation rate dominated by the H_2 from the U-H_2O reaction with minor radiolytic H_2 contribution. Mitigating operational and drum loading constraints for sludge disposition to WIPP requires reducing H_2 rates by >100.

Four means to decrease the H_2 rate are to decrease temperature, isolate U metal from H_2O, use corrosion inhibitors, or use hydrogen scavengers. Although lower temperature is applicable to controlled systems, it is not applicable to WIPP, where shipped packages must show suitably low H_2 generation rates at ~60°C. Isolating reactants by grouting can decrease H_2 generation rates for simulated sludge by up to factor of ~3 but the mitigation effect is variable, and greater impacts are unlikely because grouts have appreciable water vapor pressures that allow U metal reaction with condensing H_2O films. Nochar N960, a commercial absorbent for nuclear waste applications, has been proposed as a desiccant based on its high aqueous solution sorption capacity while MgO or CaO can maintain 0.04% relative humidity. The most effective inorganic U metal aqueous corrosion inhibitors found in reported investigations were nitrite and phosphate. Dissolved O_2 inhibits the U metal corrosion rate in 60°C water by a factor of ~30 but maintenance of active aeration in dense heterogeneous sludge limits it applicability. Scavengers of the "nascent" hydrogen that appears in corrosion of U and other active metals in water include nitrate, nitrite, permanganate, and chromate. Of the inhibitors or scavengers, nitrate and nitrite are the most promising in Hanford experience and applicability/compatibility with the K Basin system.

Nochar N960 was tested as a desiccant and sodium nitrate, nitrite, and hydrogen phosphate were tested as corrosion inhibitors or hydrogen scavengers for U metal in water. While Nochar and phosphate had little or no beneficial effect, both nitrate and nitrite decreased H_2 generation rates with nitrite providing about 10 times more H_2 mitigation as nitrate on an equal molar basis. For example, H_2 generation was attenuated by a factor of 3.2 in 60°C 0.1 M $NaNO_3$ while 0.1 M $NaNO_2$ decreased H_2 generation by a factor of about 42 compared with parallel tests without added salt. At 1 M reagent, the H_2 attenuation factor was 660 for $NaNO_3$ and 5700 $NaNO_2$. The nitrate reduction products are predominantly nitrite and NH_3 while NH_3 is the nitrite reduction product.

Notes:

Thursday, May 21

Technical Session IV:
Waste Treatment, Stabilization and Disposition

Chair: David Riley, LLNL

Notes:

Long Term Storage of Used Fuel Followed by Recycle and Reuse of Most Components as a Sustainable Path for Abundant Nuclear Energy

G. D. Del Cul*, E. D. Collins, B. B. Spencer and R. T. Jubin
Oak Ridge National Laboratory, Oak Ridge, TN, USA

Nuclear energy represents a large and economical source of clean energy without carbon emissions. The present track record related to safety and reliability has been very good and the public perception is becoming favorable toward an expanded role of nuclear energy in the U.S. and the world. However, what to do with the 60,000 tonnes of used nuclear fuel currently being stored at the U.S. reactor sites and growing at about 2,300 tonnes per year is a major issue that needs to be fully resolved.

Recent research, development, and systems analysis studies have shown very significant potential benefits in terms of cost and safety to storing the used fuel assemblies for several decades prior to processing. These studies have shown that the recycling of most components in used fuel, including all actinides, cladding, hardware, and selected fission products, could be done at the same rate as used fuel is generated. This extended storage is in fact the present default strategy for used fuel management and the benefits have already largely accrued to the oldest fuels in the present inventory. During this time, the radioactivity and decay heat generation decrease substantially, such that the separations process can be simplified and made less costly, while minimizing emissions. This extended storage also enables recycle of long-lived actinides to be accomplished in the existing fleet of thermal reactors (LWR and CANDU). However, there is sufficient flexibility to include the recycling in future reactors as they become a reality. Overall, the safety, environmental, and cost benefits of treating the longer aged nuclear wastes are substantial and there appears to be an "optimum storage" of 30-70 years to maximize benefits in safety, environmental effects, and cost reduction while maintaining an acceptable radiation barrier to mitigate proliferation risks. Particular emphasis needs to be given to the drastic reduction of secondary wastes, losses and emissions during all processing steps and comprehensive in-plant recycling and reuse needs to be implemented as a primary design objective.

The near complete recycling, along with minimization of emissions and secondary wastes, should promote enhanced public acceptance and consensus building in the environmental community. Under this strategy, the bulk of the fuel material is not disposed and only the problematic residual wastes, which represent small fraction of the used fuel, must be accommodated.

Notes:

The Status of De-Inventory of Security Category I/II Materials from Lawrence Livermore National Laboratory

D. C. Riley, K. Dodson
Lawrence Livermore National Laboratory, Livermore, Ca, USA

The status of the de-inventorying of Lawrence Livermore National Laboratory (LLNL) Category I/II Special Nuclear Material (SNM) will be discussed. Lawrence Livermore is currently reducing the amount of SNM stored at LLNL as part of Department of Energy's Complex Transformation effort. This presentation will give an overview of the de-inventory activities. The kinds of materials that are being dispositioned will be described. An overview of what processing needs done will be shown. The presentation will show that this information was used to develop an overall schedule for the project. The status of the de-inventory effort will also be presented.

Technetium Separations and Waste Form Development

Gordon D. Jarvinen*, **David Kolman, Kristy M. Long, George S. Goff, Kurt Sickafus, James Valdez**
Los Alamos National Laboratory, Los Alamos, NM, 87545

Kenneth R. Czerwinski, Frederic Poineau, Thomas Hartmann, Edward Mausolf
Radiochemistry Program, University of Nevada, Las Vegas, Las Vegas, NV 89154-4003

The Uranium Extraction (UREX) process has been developed as part of a suite of separation processes to partition used nuclear fuel generically called UREX+. This group of solvent extraction operations has been tested at a kilogram scale with used fuel samples as part of the Advanced Fuel Cycle Initiative of the DOE Office of Nuclear Energy. The UREX process uses TBP to selectively extract uranyl nitrate and pertechnetate from the actinide and fission product mixture resulting from dissolving spent fuel in nitric acid and back extracts these species into dilute nitric acid. Technetium is a fission product of particular concern to the environment because of its long half-life and high solubility and mobility in groundwater as pertechnetate under oxidizing conditions. We are using anion exchange resins to remove the pertechnetate from the UREX product solution and investigating various methods to recover the technetium from the column. Technetium-containing alloys and ceramic oxides are under study as potential waste forms. Some of the recent work with waste form preparation and characterization will be reviewed.

Notes:

3013 Surveillance and Monitoring Program – Characterization of Pu-Bearing Solids

G. F. Kessinger,* M. G. Bronikowski, R. R. Livingston
Savannah River National Laboratory, Aiken, SC, USA

The Savannah River National Laboratory is responsible for plutonium oxide characterization associated with 3013 Destructive Examination (DE) Surveillance Program. The requirements for DE are defined by the Surveillance and Monitoring Program based on the Department of Energy's standard for long-term storage of plutonium (DOE-STD-3013). Characterization techniques for plutonium oxide samples include: density measurements, dissolution/leaching studies (to determine chemical composition), moisture content determination, and surface area determination.

These materials contain alkali halide contamination that varies from a few weight percent to about 50 weight percent, with smaller fractions of other alloys and oxides. During the past year, two dissolution flowsheets were tested for the digestion of PuO_2-bearing solids. Both flowsheets were tested at ~95°C. The first flowsheet utilizes 12 M HNO_3-0.2 M HF. The second flowsheet is also a 12 M HNO_3-0.2 M HF dissolution flowsheet; however, the dissolver product from the second dissolution process is complexed with 0.9 M H_3BO_3 at the dissolution temperature of ~95°C. The results of this investigation show that the complexed flowsheet is more effective for digesting PuO_2 and keeping Pu in solution than the un-complexed approach.

Notes:

Moisture Analysis of Chloride-Bearing Plutonium Oxide Surrogates for Small-Scale Corrosion Tests (U)

Jonathan M. Duffey*, Philip E. Zapp, Mark L. Crowder, Philip M. Almond, James E. Laurinat, Nicholas J. Bridges, Glen F. Kessinger, and Ronald R. Livingston
Savannah River National Laboratory, Aiken, SC, USA

Small-scale corrosion tests were conducted with mixtures of PuO_2 and NaCl, KCl, $MgCl_2$, and $CaCl_2$ salts placed in contact with stainless steel test coupons and exposed to a humidified He atmosphere. Test materials were packaged in He inside stainless steel test containers equipped with a pressure transducer and a micro-valve for gas sampling. Tests containers were maintained at room temperature for times ranging from five to 27 months while monitoring temperature and pressure. At the conclusion of each test, headspace gas samples were analyzed and post-exposure moisture contents of PuO_2/Cl^- salt mixtures were determined by thermogravimetric analysis coupled with mass spectrometric detection (TGA-MS). Water calibration curves were constructed using gypsum ($CaSO_4 \cdot 2H_2O$) as a standard. Initial moisture contents for the PuO_2/Cl^- compositions were calculated by correcting post-exposure moisture contents for water consumed by radiolysis or chemical reaction to generate H_2. Calculated initial moisture contents were compared to initial moisture contents as determined by gravimetric measurements during exposure to humidified He.
SRNL-STI-2009-00205-X

Analysis of the Causal Factors that Lead to Hydrogen Generation within 3013 Storage Containers

Philip M. Almond*, Mark L. Crowder, Nicholas J. Bridges, Jonathan M. Duffey, Ronald R. Livingston, Glen F. Kessinger, and Michael G. Bronikowski
Savannah River National Laboratory, Aiken, SC, USA

Matthew J. Arnold and Lance E. Traver
Savannah River Site, Aiken, SC, USA

The Savannah River National Laboratory (SRNL) is under contract to perform surveillance of 3013 containers utilized across the DOE complex to package and store plutonium-bearing material for up to 50 years. This is a requirement derived from the Surveillance and Monitoring Program based on the Department of Energy's Standard (DOE-STD-3013) for stabilization, packaging and storage. The Destructive Examination (DE) surveillance program at SRNL includes 3013 container metallography, headspace gas analysis, and plutonium oxide materials analysis. This presentation will focus on materials from containers that were analyzed and found to have appreciable hydrogen and/or oxygen in the headspace after storage. The dose rate, moisture content, and composition of plutonium-bearing material, and how these characteristics relate to the generation of headspace gases in these canisters will be discussed.
SRNL-STI-2009-00219

Notes:

Gas Generation in Small-Scale Corrosion Tests with Chloride-Bearing Plutonium Oxide Surrogates (U)

Jonathan M. Duffey*, Philip E. Zapp, Glen F. Kessinger, Philip M. Almond, Nicholas J. Bridges, and Ronald R. Livingston
Savannah River National Laboratory, Aiken, SC, USA

Gas generation measurements were performed for small-scale corrosion tests with mixtures of PuO_2 plus NaCl, KCl, $MgCl_2$, and $CaCl_2$ salts in contact with stainless steel test coupons. Test compositions were exposed to a humidified He atmosphere for several hours to attain a moisture content of nominally 0.5 wt%. Test materials were packaged in a He atmosphere in stainless steel test containers equipped with a pressure transducer and a micro-valve for gas sampling. The sealed containers were maintained at room temperature for times ranging from five to 27 months while monitoring temperature and container pressure. At the conclusion of each test, the container headspace was sampled and analyzed by mass spectrometry and/or gas chromatography to determine the major gas components. Hydrogen (H_2) generation was found to be primarily a function of water content and alpha dose. Oxygen (O_2) generation was more variable. For replicate sample compositions that were sampled at different times, average H_2 generation rates decreased with time as water was consumed by radiolysis or chemical reaction. Gas generation results were compared with sample compositions and corrosion observations to evaluate possible trends.

SRNL-STI-2009-00206-X

Notes:

Thursday, May 21

Seaborg Award Banquet

Keynote Speaker: David C. Antonucci

Notes:

Amazing Lake Tahoe Facts

Centered on the most frequently asked questions and mysteries about Lake Tahoe, this presentation covers the Lake's most fascinating facts. Viewers will learn about the natural forces that created Lake Tahoe, take a virtual dive into the underwater world and learn about the science of protecting Lake Tahoe.

Companion booklets are available for $5 each.

About David C. Antonucci

David is a 34-year plus resident of Lake Tahoe, holds two degrees in civil and environmental engineering from Cal Poly and has over 36 years experience in engineering, management and community leadership. He is a well-known local historian who advises local media and community groups on local history. He is a frequent presenter to conference groups on a wide range of topics. He has published the booklets *The Natural World of Lake Tahoe* and *Mark Twain at Lake Tahoe* and a number of professional papers.

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