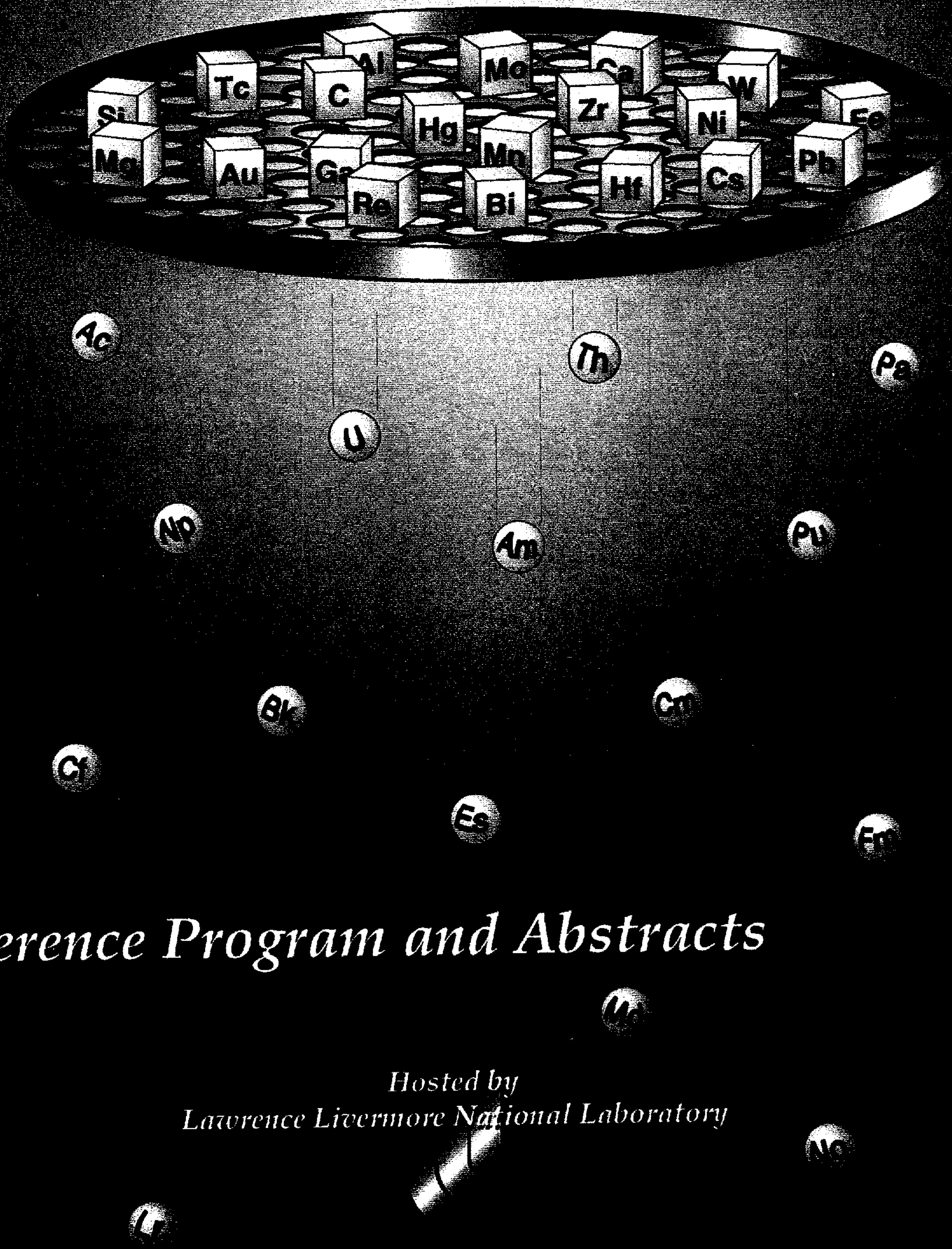
Nineteenth Annual

Actinide Separations Conference

June 12-15, 1995
Monterey, California



Conference Program and Abstracts

Hosted by
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1995	ROBERT A. PENNEMAN - LOS ALAMOS NATIONAL LABORATORY

RECIPIENT OF THE 1995
SEABORG ACTINIDE SEPARATIONS AWARD

DR. ROBERT A. PENNEMAN

Dr. Robert A. Penneman's broad contributions to actinide separations chemistry span five decades, starting with Manhattan Project work in July 1942 on radiation effects on radiolanthanum, bismuth phosphate, and ion exchange processes. After completing his Ph.D. Degree at the University of Illinois in 1947 (on separation of anhydrous hydrazine from its hydrate), Bob began a distinguished 37 year career in hands-on R/D and technical management at Los Alamos National Laboratory, continuing to the present time as a valued consultant in environmental restoration. Most of his work focused on R/D directly related to actinides separations as documented in more than 130 peer-reviewed publications, service on numerous national and international committees, many honors and awards, and a legacy of hiring many highly productive actinide scientists and engineers into the nation's nuclear complex.

Bob was responsible for building and maintaining one of Los Alamos' premier scientific groups, which ensured the maintenance of this institution's R/D base in actinide chemistry. The impact on actinide separations includes the following:

Americium

Conducted the first gram-scale separation of americium and discovery of Am(IV) and Am(VI), as reported in a series of classic papers. This work formed the basis for much separations chemistry based on higher oxidation states of americium. Bob also co-authored the americium and curium chapters in Seaborg's classic book on the actinide elements.

Curium

Discovery by Bob's group of Cm(IV), definition of its stability limits and authorship of much key technical literature on its chemistry. This work laid the groundwork for precipitation processes used subsequently at Savannah River for separating americium and curium.

Plutonium

Developed the fundamental chemistry of pentavalent uranium and plutonium, both as aqueous "yls" and complexed ions. In the 1980s, Bob's group also made a key discovery of low temperature agents for volatilizing plutonium as its hexafluoride, renewing interest in volatility based plutonium separations.

Lanthanide/actinide separations

Developed a pilot scale liquid ion exchange separations process using thiocyanate which was subsequently deployed at Rocky Flats.

The group managed by Bob also made significant contributions in separations science unrelated to actinides, including the development of large scale cryogenic column methods for separating stable isotopes of carbon, nitrogen, and oxygen.

CONFERENCE AGENDA

Monday, June 12, 1995

Registration	6:00 - 9:00 p.m.
Reception	7:00 -10:00 p.m.

Tuesday, June 13, 1995

Registration/Continental Breakfast	7:30 - 8:30 a.m.
Introduction - Leonard W. Gray	8:30 a.m.
Conference Business - Mark C. Bronson	8:45 a.m.
Invited Talk - Henry F. Dalton DOE-94-1 Implementation Plan Overview	9:00 a.m.
Invited Talk - Michael M. May Policy Issues with Plutonium Disposition	9:30 a.m.
Break	10:00 a.m.
Spent Fuel Treatment Session	10:30 a.m.
Waste Treatment Session	10:30 a.m.
Lunch Break	12:20 p.m.
Issues and Responses to DNFSB 94-1 Session	1:30 p.m.
Waste Treatment Session	1:30 p.m.
Poster Session/Reception	7:00 -10:00 p.m.

Wednesday, June 14, 1995

Registration/Continental Breakfast	7:30 - 8:30 a.m.
Issues and Responses to DNFSB 94-1 Session (cont.)	8:30 a.m.
Pyrochemical Technologies Session	8:30 a.m.
Unscheduled Afternoon	12:30 p.m.
Evening at the Monterey Aquarium	7:00 -11:00 p.m.

Thursday, June 15, 1995

Registration/Continental Breakfast	7:30 - 8:30 a.m.
Disposition Technologies Session	8:30 a.m.
Aqueous Separation Technologies Session	8:30 a.m.
Lunch Break	11:35 a.m.
Disposition Technologies (cont.)	1:00 p.m.
Aqueous Separation Technologies (cont.)	1:00 p.m.
Social Hour	6:00 p.m.
Banquet	7:00 p.m.

Session: Waste Treatment

Tuesday, June 13, 1995
10:30 a.m. - 4:10 p.m.

Session Chairs: Martyn Adamson, LLNL and Tom Mulcahey, ANL

Time	Title	Author
10:30	Session Business	
10:40	TRUEX Processing of Idaho Sodium - Bearing Wastes: Treatment of the Extraction Behaviors of Mercury and Zirconium in the Generic TRUEX Model	G.F. Vandegrift
11:05	Thermodynamic Modeling of the Solubility of $PuO_2 \bullet xH_2O(am)$ in Alkaline Hanford Waste Solutions	L. Rao
11:30	Plutonium Solubility in Savannah River Site High-Level Waste	D.G. Karraker
11:55	Lunch Break	
1:40	Waste Treatment at the Radiochemical Engineering and Development Center	W.D. Bond
2:05	Transuranic Residue Treatment: Regulatory Considerations	R.E. Wieneke
2:30	Transuranic Waste Management Program at the Idaho National Engineering Laboratory	J. Van Vliet
2:55	Break	
3:20	Computerized Waste Documentation at the TA-55 Plutonium Facility	R.E. Wieneke
3:45	Alkaline Leaching of Waste Tank Sludge	D.D. Ensor

Session: Spent Fuel Treatment

Tuesday, June 13, 1995
10:30 a.m. - 12:20 p.m.

Session Chairs: Walt Bond, ORNL and Lee Bendixsen, LITCO

Time	Title	Author
10:30	Session Business	
10:40	Separation of Uranium from Zircaloy Clad N-Reactor Fuel in a High Throughput Electrorefiner	E.C. Gay
11:05	High Temperature Treatment of Oxide Spent Fuels and Residues	G.K. Johnson
11:30	Engineering-Scale Demonstration of Oxide Spent Fuel Treatment Part 1 - Operations	T.P. Mulcahey
11:55	Engineering-Scale Demonstration of Oxide Spent Fuel Treatment Part II - Test Results	E.J. Karell

Session: Issues and Responses to DNFSB 94-1

Tuesday, June 13, 1995
1:30 p.m. - 4:35 p.m.

Session Chairs: Alice M. Murray, EG&G RFETS and
William G. Von Holle, DNFSB

Time	Title	Author
1:30	Session Business	
1:40	The Purpose of Recommendation 94-1	W.G. Von Holle
2:05	LLNL 94-1 Plutonium Stabilization Activities	B.H. Ives
2:30	Status of the Plutonium Vault Remediation Effort at Los Alamos	K.W. Fife
2:55	Break	
3:20	Solid Residue Stabilization Program at Rocky Flats Environmental Technology Site	A.M. Murray
3:45	Solid Residue Stabilization Strategic Plan	A.C. Muscatello
4:10	A Risk-Based Prioritization for the Stabilization and Repackaging of Nuclear Materials at the Los Alamos Plutonium Facility	L. Spanks

Session: Issues and Responses to DNFSB 94-1

Wednesday, June 14, 1995
8:30 a.m. - 12:00 p.m.

Session Chairs: Alice M. Murray, EG&G RFETS and
William G. Von Holle, DNFSB

Time	Title	Author
8:30	Session Business	
8:40	RFETS Solutions Stabilization Flowsheet Development	S.B. Schreiber
9:05	Stabilization of Americium/Curium Solutions by Vitrification	T.S. Rudisill
9:30	Unstable Ash Characterization and Stabilization	R.A. Van Konynenburg
9:55	Thermal Stabilization of Plutonium Bearing Scrap	L.T. Cunningham
10:20	Break	
10:45	A Proposed Standard for Safe Storage of Department of Energy (DOE) Plutonium Contaminated Residues	L.R. Crisler
11:10	A Technical Basis for Safety Surveillance of Stored Plutonium Metal and Oxide	R.L. Moment
11:35	Impacts of 94-1 Stabilization Activities on Pu Disposition Options	G.A. Armantrout

Session: Pyrochemical Technologies

Wednesday, June 14, 1995
8:30 a.m. - 12:25 p.m.

Session Chairs: Vic Freestone, AWE and Bill Averill, EG&G RFETS

Time	Title	Author
8:30	Session Business	
8:40	Electrochemical Processing using Cadmium Electrodes	Z. Tomczuk
9:05	Recovery of Plutonium Metal by a Hydride/ Dehydride/Casting (HYDEC) Process	C.A. Colmenares
9:30	Processing of C-Saturated Tantalum Hardware for Actinide Pyrochemical Processing	G.L. DePoorter
9:55	Uranium Separation and Transport in the Molten Salt Reactor Experiment	G.D. DelCul
10:20	Break	
10:45	Pyrochemical Techniques for the Separation of Actinides	D.C. Riley
11:10	Distillation Separation of Chloride Salts from Plutonium	E. Garcia
11:35	Vacuum Evaporation of KCl-NaCl Salts: Vaporization Rate Model and Experimental Results	L.L. Wang
12:00	TRUMP-S: Pyropartitioning of Actinides from PUREX Wastes	S.P. Fusselman

Session: Disposition Technologies

Thursday, June 15, 1995
8:30 a.m. - 3:55 p.m.

Session Chairs: Theresa Cremers, LANL and Tom Crawford, WHC

Time	Title	Author
8:30	Session Business	
8:40	AWE Plutonium Strategy	V. Freestone
9:05	Chipless Pit Bisector	R.D. Merrill
9:30	Preparation of PuO_2 from Weapons Grade Pu for use in Mixed Oxide Reactor Fuel Pellets	C.A. Colmenares
9:55	Break	
10:20	Electrolytic Decontamination of Oralloy - A Disposition Task	M.E. Huerta
10:45	Immobilization of Plutonium in Glass: Issues and Approaches	J.K. Bates
11:10	Surplus Pu to Pu-Glass Using Existing F-Area Facilities at SRS	E.P. Maddux
11:35	Lunch Break	
1:00	Immobilization of Actinides in Geomedia by Phosphate Mineralization	K.L. Nash
1:25	Excess Pu Disposition in Zirconolite-rich Synroc	E.R. Vance
1:50	Excess Pu Disposition in Zirconolite-rich Synroc containing Nepheline	V.M. Oversby
2:15	Break	
2:40	Use of Spent Fuel Treatment Technology for Plutonium Immobilization	J.J. Laidler
3:05	A Zirconium Metal Alloy for the Immobilization of Surplus Fissile Materials	S.M. McDeavitt
3:30	Dry-Blending Processes Applicable to the Denaturing and Disposition of Uranium Isotope Mixtures Incorporating ^{233}U	I.H. Zimmerman

Session: Aqueous Separation Technologies

Thursday, June 15, 1995
8:30 a.m. - 3:55 p.m.

Session Chairs: Fred Marsh, SNL and Frank Graham, SRS

Time	Title	Author
8:30	Session Business	
8:40	Full Scale Testing of Extraction Chromatography for Actinide Decontamination of Concentrated Hydrochloric Acid Waste Streams	L.D. Schulte
9:05	The Effects of Anion Exchange Functional-Group Variations on the Sorption of Pu(IV) from Nitric Acid	S.F. Marsh
9:30	Sorption of Americium from Hanford Tank 101-SY Simulant Solution	Z.V. Svitra
9:55	Break	
10:20	Sorption of Plutonium and Americium from Generic Hanford Complexant Concentrate (CC) Simulant Solution	S.F. Marsh
10:45	Reactivity of Tributyl Phosphate Degradation Products with Nitric Acid: Relevance to the Tomsk-7 Accident	G.S. Barney
11:10	Studies of Solvent Extraction of Actinides in TTA + Diamide Systems	Y. Sasaki
11:35	Lunch Break	
1:00	Zirconium Chemistry in the TRUEX Process Solvent	K.N. Brewer
1:25	TRUEX Flowsheet Development as Applied to Idaho Chemical processing Plant Liquid Acidic High-Activity Waste Using Centrifugal Contactors	J.D. Law
1:50	TRUEX Processing of Idaho Sodium-Bearing Wastes: Treatment of the Extraction Behaviors of Mercury and Zirconium in the Generic TRUEX Model	G.F. Vandegrift
2:15	Break	
2:40	A Comparison of TRUEX and CMP Solvent Extraction Process for Actinide Removal from ICPP Waste	R.S. Herbst
3:05	Recovery and Separation of Transplutonium Actinides from Irradiate Targets	L.K. Felker
3:30	A New Extraction Chromatographic Material for Actinide Cleanup Procedures	R. Chiarizia

Session: Poster

Tuesday, June 13, 1995
7:00 - 10:00 p.m.

Title	Author
An Overview of the Los Alamos Efforts in Tank History Characterization and Estimation of Chemical/Radionuclide Inventories for the Hanford Waste Tanks	S.F. Agnew
Design of a Furnace-Based Tritium Decontamination System for Plutonium	W.H. Atkins
Design of an Alpha Spectrometer to Determine Pu/Am Fixed Contamination on Uranium	W.H. Atkins
Radionuclides Transport and Colloid Formation During Sludge Washing	W.D. Bond
Criticality Safety when using Neutronic Poisons with Plutonium	R.H. Condit
Actinide Neutron Source Recovery	S.M. Dinehart
Actinide Recoverability from Synroc Ceramics	B.B. Ebbinghaus
ALPHA Vitrification Facility	P. Guiberteau
Plutonium Immobilization Hot Press Development	J. M. Lawson
Declassification of Non-Plutonium Weapons Components	D.P. McAvoy
Metal Conversion and Oxide Stabilization Systems	S.T. Mills
ALPHA Swipe Spectroscopy for Oralloy Disposition	A.N. Morgan
Magnetic Swing Adsorption: A New Actinide Separation Process	J.D. Navratil
Nuclear Applications for Magnetic Separations	D.A. Romero
Oxalate Precipitation of Pu(III) from very Dilute HCl Solutions	R.R. Salazar

Invited Talk

DOE 94-1 Implementation Plan Overview

Henry F. Dalton
Nuclear Material Stabilization Task Group
U.S. Department of Energy

On May 26, 1994, the Defense Nuclear Facility Safety Board issued to the U.S. Department of Energy its Recommendation 94-1. In this Recommendation, the Board expressed concern about certain liquids and solids containing fissile materials and other radioactive substances located in temporary storage configurations at several department facilities. The Department concurred with and accepted the Recommendation and issued its Implementation plan on February 29, 1995. Accordingly, the Department established the Nuclear Materials Stabilization Task Group to integrate the Department program to carry out the Implementation Plan. The Task Group focus is on near-term storage. As recommended by the Board, a systems engineering approach is being implemented to ensure that facilities and capabilities at the various Department sites are being utilized effectively and efficiently. The structure and approach of the Task Group will be discussed, with particular attention given to the partitioning of effort according to material type. Particular areas of activity will be discussed along with currently identified needs for technology development.

Invited Talk

Policy Issues with Plutonium Disposition

Michael M. May
Director Emeritus
Lawrence Livermore National Laboratory

With the end of the Cold War, the United States and the nations of the former Soviet Union are engaged in arms reduction on an unprecedented scale. What to do with the materials from the tens of thousands of nuclear weapons to be dismantled has become a pressing problem for international security. "Management and Disposition of Excess Weapons Plutonium," a study by the National Academy of Sciences' Committee on International Security and Arms Control (CISAC)resulted from a request by General Brent Scowcroft, then National Security Adviser to President Bush. This Study has principal recommendations in four general areas:

1. A New Weapons and Fissile Materials Regime
2. Safeguarded Storage
3. Long-term Plutonium Disposition, and
4. All Fissile Materials

In the area of long-term plutonium disposition, the NAS noted the two most promising alternatives to be:

- fabrication and use as fuel, without reprocessing, in existing or modified nuclear reactors; or
- vitrification in combination with high-level radioactive waste.

A third option, burial of the excess plutonium in deep boreholes, has until now been less thoroughly studied, but could turn out to be comparably attractive.

This paper will review the NAS Study.

Session: Waste Treatment

TRUEX Processing of Idaho Sodium-Bearing Wastes: Treatment of the Extraction Behaviors of Mercury and Zirconium in the Generic TRUEX Model

George F. Vandegrift, Carol Mertz and Monica Regalbuto
Argonne National Laboratory

The extraction behavior of mercury and zirconium in the TRUEX solvent is of interest for the processing of Idaho sodium-bearing and dissolved-calcine wastes. The TRUEX process is a solvent extraction process developed to extract transuranic elements from acidic nitrate media. The Generic TRUEX Model (GTM) has been developed as a tool for (1) designing flowsheets for treating the wide variability of a high-level and transuranic waste streams at DOE sites and (2) guiding experimental programs to test the applicability of the TRUEX process for those wastes. Much of the development of the GTM has been directed to treating Hanford wastes; therefore, the extraction behavior of mercury has not been included previously in this model, and zirconium extraction has been given only limited attention. For the same reason, the effects of chloride ion on extraction behaviors have also been ignored. These components are extremely important in designing flowsheets for treating wastes stored at Idaho National Engineering Laboratory and are being added to the GTM.

To this end, batch distribution ratio measurements are being performed to determine the extraction mechanisms for Zr(IV) and Hg(II) in the TRUEX process. The thermodynamic and mechanistically correct models derived from these data will allow the GTM to predict the aqueous-phase solution chemistry and extraction behavior of these species. Enhancement will also include expansion of the current speciation models to include Hg(II) and chloride ions using literature complexation-stability and acid-dissociation equilibrium constants. (Including chloride ion speciation will affect the extraction behavior, to varying degrees, of all metal ions.) In this paper we will discuss our results and how they affect prediction by the GTM.

Session: Waste Treatment

Thermodynamic Modeling of the Solubility of $PuO_2 \cdot xH_2O(am)$ in Alkaline Hanford Waste Solutions

Linfeng Rao, Andrew R. Felmy and Dhanpat Rai
Pacific Northwest Laboratory

Calvin H. Delegard
Westinghouse Hanford Company

Knowledge of the thermodynamics of actinide elements in alkaline solutions is of interest in the storage of high-level nuclear waste and the development of remediation techniques for the Hanford tank waste solutions. In the present work, experimental data on the concentrations of plutonium in NaOH, $NaOH/NaNO_3$, $NaOH/NaNO_2$, $NaOH/NaNO_3/NaNO_2$, $NaOH/NaAl(OH)_4$, and $NaOH/Na_2CO_3$ solutions were analyzed with the specific ion-interaction approach of Pitzer et al. Based on these solubility data and available information on both solid (amorphous $Pu0_2 \cdot xH_20$, identified by XRD) and aqueous plutonium species (Pu(V), observed by absorption spectroscopic measurement in concentrated NaOH), a thermodynamic model was proposed. The predictions of plutonium concentrations by this model are in good agreement with the observed results in the NaOH, $NaOH/NaNO_3/NaNO_2$, and $NaOH/NaAl(OH)_4$ systems, but less satisfactory for the $NaOH/NaNO_3$ and $NaOH/NaNO_2$ systems. Modeling of the $NaOH/Na_2CO_3$ system is still in process. Limitations in the model development and the need for more experimental data are discussed. As an effort to provide further information on actinide speciation in alkaline solutions and improve the current model, an analogous study using neptunium(V) is underway.

This work is supported by the Tank Waste Remediation System Program (TWRS) through Westinghouse Hanford Company. Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

Plutonium Solubility in Savannah River Site High-Level Waste

D. G. Karraker
Savannah River Technology Center

Waste tanks at Savannah River and Hanford contain a layer of base-insoluble solids ("sludges") and a liquid supernate, the latter a basic solution of nitrates, nitrites, aluminates, sulfate, etc. The solubility of plutonium in the supernate is low, but significant to supernate processing. Processing the supernate involves evaporation to conserve storage space, dissolution of crystallized salts for ^{137}Cs precipitation and ^{90}Sr adsorption, storage of the decontaminated supernate in a grout ("Saltstone").

The solubility of Pu(IV) in a synthetic supernate was found to increase with NaOH concentration from ca. 1 to 10-15 ppm; a further increase was found after irradiation in a ^{60}Co source to simulate the 4 x 10^4 R/hr radiation field in high-level waste tanks. The effects of the anions in the supernate has been investigated; in general, the solubility correlates only with NaOH concentration (activity).

Pu(VI) solubility was found to be 50-100 mg/L in NaOH solutions, and the Pu(VI) species was stable over a several week period in synthetic waste supernate solution. Pu(VI) stability increases as the concentration of NaOH increases; in 2M NaOH, radiation products reduce Pu(VI) to a species of lower solubility, probably Pu(IV). In 6-10M NaOH, radiation does not affect the solubility of Pu in Pu(VI) spiked synthetic waste solutions.

Session: Waste Treatment

Waste Treatment at the Radiochemical Engineering and Development Center

W. D. Bond, R. R Brunson, D. E. Benker, F. R. Chattin and E. D. Collins
Oak Ridge National Laboratory[1]

At the Radiochemical Engineering and Development Center (REDC) irradiated targets are processed for the recovery of valuable radioisotopes, principally transuranic nuclides. We are in the process of installing a system for treating the various liquid wastes as they are generated at the REDC. Radionuclides to be removed from the wastes include actinides, fission products, and activation products. The radionuclides removed are stored as solids and thus the future discharge of radionuclides to low-level liquid waste tank storage is greatly reduced. The treatment system is of modular design and will be installed in a hot cell (Cubicle 7) in Building 7920 at the REDC facility. The module incorporates the following: (1) a resorcinol-formaldehyde resin column, (2) a cross flow filtration unit and (3) a waste solidification unit. Process flowsheets for operation of the module and the supporting data for the flowsheets are discussed.

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[1] Managed by Martin Marietta Energy Systems, Inc., for the U. S. Department of Energy under Contract No. DE-AC05-840R21400.

Transuranic Residue Treatment: Regulatory Considerations

Ronald E. Wieneke
Los Alamos National Laboratory

The Federal Facilities Compliance Agreements between the respective sites who process plutonium bearing residues and their regulatory agencies require compliance with the regulations of the Resource Conservation and Recovery Act (RCRA) and waive sovereign immunity to environmental enforcement actions. In many cases the status of residues is uncertain and may make them subject to regulation under "reclamation" activities and their consequent classification as "hazardous waste" under RCRA. The treatment processes themselves as well as waste products would require proper licensing and treatment in accordance with these regulations. Careful forethought and advanced planning is needed in order to obtain the required licensing and to provide adequate process descriptions and coverage while maintaining the flexibility of operations. Treatment Demonstration Permits are somewhat more easily obtained, but severely restrict the quantities of waste and the duration of the treatment process within their purview. Key in this evaluation is the status of existing residues and the Department of Energy's commitment to define a programmatic mission for the recovered nuclear materials. In many cases, maintaining compliance with applicable environmental regulations can be as challenging as establishing the treatment processes themselves, and certainly carries a significant legal liability for the contractor.

Session: Waste Treatment

Transuranic Waste Management Program at the Idaho National Engineering Laboratory

Jim Van Vliet
Lockheed-Martin Idaho Technologies

The Idaho National Engineering Laboratory (INEL) currently stores about sixty-percent of the nation's transuranic waste (TRU). This waste is planned for permanent disposal at the Waste Isolation Pilot Plant (WIPP), near Carlesbad, New Mexico. A number of programs and projects totaling in excess of one billion dollars are in progress at INEL to prepare the stored TRU for shipment to WIPP. These programs are focused on waste reconfiguration, waste characterization, waste treatment, waste repackaging and waste transportation. The programs are structured and sequenced to enable INEL to ship TRU by the WIPP opening date of 1998.

Waste Reconfiguration. The INEL TRU storage system does not currently conform to the requirements of RCRA. DOE and the State of Idaho have negotiated an agreement and schedule for moving the TRU into RCRA compliant storage. To meet these requirements, Lockheed Idaho is constructing nine RCRA compliant storage facilities, a retrieval enclosure (large enough to house four football fields or twenty space shuttles), and has contracted for a waste recovery vehicle that will allow robotic recovery. At present, readily retrievable waste is being moved into RCRA compliant storage, and the retrieval enclosure is under construction.

Waste Characterization. The INEL waste characterization facility will perform those waste inspections needed to verify conformance to the WIPP waste acceptance criteria. These include a broad set of requirements, including container integrity, RCRA characteristics, particle size, free liquid content, and gas generation rates to be specified through the no migration petition submittal to EPA. At the present, the waste characterization facility is waiting construction authorization from DOE. Lockheed Idaho is also performing gas generation data collection for the WIPP no migration petition.

Waste Repackaging and Treatment. Some TRU will need to be repackaged and/or treated prior to transportation to, or disposal at, WIPP. Program planning efforts are in progress to define the requirements for an appropriate facility or approach to satisfy this need. Technical studies are also in progress on preferred treatment processes.

Waste Transportation. The current TRUPAC II transportation cask is not suitable for transport of boxed TRU. Boxed TRU represents about sixty percent of the total INEL TRU inventory. Assessments are being performed to identify the best approach to this problem; which generally ranges from development of a new cask to repackaging.

Session: Waste Treatment

Computerized Waste Documentation at the TA-55 Plutonium Facility

Ronald E. Wieneke
Los Alamos National Laboratory

Los Alamos National Laboratory's TA-55 Plutonium Facility is preparing to initiate testing, prior to implementation, of a new computer based Waste Management Program. The program will initially address transuranic wastes from plutonium processing activities within the plant, but will be expanded to encompass low-level, hazardous and mixed wastes as well. The system will reside on the TA-55 local area network and will be accessed by means of notebook computers and network ports placed strategically within the processing areas. All information on waste items will be kept on the system beginning with generation through treatment, packaging, interim storage and eventual shipment out of the Facility. This will accomplish several goals; establish a paperless system reducing suspect low-level paper waste generation, provide ready access to waste item information by all involved personnel, present a user friendly interface, eliminate transcription and computational errors, automate error checking and provide rapid electronic transfer of waste information to organizations outside of TA-55. This system and its integral database will allow ready access to waste generation information, automated generation of waste management and minimization performance indicators and the automatic generation of reports for both internal and external use. The next step will be the integration of bar code labeling for item identification and tracking.

References

1. Agnew, S. F., "Hanford Defind Wastes: Chemical and Radionuclide Compositions", Report: LA-UR-94-2657, August 1994.

Session: Waste Treatment

Alkaline Leaching of Waste Tank Sludge

D. D. Ensor
Tennessee Technological University

B. Z. Egan, J. L. Collins and E. C. Beahm
*Oak Ridge National Laboratory**

The large volume of radioactive waste currently stored in underground tanks at various U. S. Department of Energy sites was generated by uranium and plutonium recovery processes. Although the composition of the waste varies, it is generally in the form of metal oxides or precipitated metal hydroxides and the bulk of the material is nonradioactive. Removal of the nonradioactive components through enhanced sludge washing has created interest in taking advantage of the amphoteric character of aluminum and other metals present in the waste. Dissolution studies of surrogate waste tank sludges and an actual sludge sample from Melton Valley Storage Tank W-25 have been done in NaOH solutions with concentrations up to 6M. In both the surrogate sludges and the W-25 sludge the most leachable elements were aluminum, chromium, and zinc. However, the percent removal of these elements from the W-25 sludge was much less. The usefulness of alkaline leaching as a pretreatment method as well as efforts to model the leaching behavior of these waste forms will be discussed.

*Managed by Martin Marietta Energy Systems, Inc. for the Department of Energy under contract DE-AC05-840R21400.

Session: Spent Fuel Treatment

Separation of Uranium from Zircaloy Clad N-Reactor Fuel in a High Throughput Electrorefiner

E. C. Gay, W. E. Miller and J. J. Laidler
Argonne National Laboratory

A compact, high throughput electrorefiner (HTER) was used to separate uranium from unirradiated Zircaloy clad N-Reactor fuel. This method is proposed as a means of separation of fission products, transuranic elements, and Zircaloy cladding from uranium in spent N-Reactor fuel. The Zircaloy cladding is separated as metal and can be incorporated in a metal alloy waste form that can be readily qualified for repository disposal.

In the HTER, uranium is electrotransported from anodic dissolution baskets, which are rotated in channels formed by concentric cylinder cathodes, to the solid cathode in a molten salt electrolyte ($LiCl-KCl-UCl_3$) at 500°C. The uranium is scraped off of the cathodes by the rotating baskets and collected in a vessel that is attached to the bottom of the outer cathode tube. Tests to date indicate that the granular uranium that is removed from the electrorefiner will contain between 1 to 5 wt % salt. This salt will contain fission products and transuranic elements. The uranium will be removed from the electrorefiner and washed in a vessel with molten salt in contact with cadmium and lithium. The transuranic elements and most of the fission products will be removed from the salt in the uranium product. This can then be displaced with cadmium prior to retorting which gives consolidated, decontaminated uranium.

The throughput rate goal for plant-scale electrorefining of spent N-Reactor fuel is 1000 kg uranium per day. Electrorefining tests of unirradiated N-Reactor fuel segments in an 8-in. diameter HTER assembly indicated that increased electrode area and shorter interelectrode distance in the HTER resulted in a throughput rate four times that measured with conventional electrodes. Based on these results, design of a 24-in. diameter HTER assembly was initiated. This HTER assembly will be a prototype of a module that is expected to electrorefine 200 kg uranium per day. Five of these modules in a single vessel containing electrolyte would be capable of electrorefining 1000 kg uranium per day.

Session: Spent Fuel Treatment

High Temperature Treatment of Oxide Spent Fuels and Residues

G. K. Johnson, R. D. Pierce, C. C. McPheeters and K. Gourishankar
Argonne National Laboratory

Several electrometallurgical processes have been developed by the Chemical Technology Division of Argonne National Laboratory for the treatment of spent oxide nuclear fuel. The lithium process was selected as the reference process from among the options. In this process, the spent oxide fuel is reduced by lithium at 650°C in the presence of molten LiCl which dissolves the product Li_20. The spent salt and lithium are recycled after the Li_20 is electrochemically reduced into lithium and oxygen using an inert anode. The reduced metal components of the fuel are separated from the LiCl salt phase and introduced into an electrorefiner that separates a uranium product stream. The highly purified uranium product may be enriched for recycle into fuel, stored for future use, or converted to a suitable form for disposal as waste. The TRU product, which is contaminated with uranium and fission products, can be alloyed with constituents of the cladding to produce a stable metal waste form, or absorbed on zeolite to make a mineral waste form.

The lithium process offers several potential advantages over other calcium-based processes that were studied. These advantages include lower temperature, a less corrosive environment, and a waste salt that can be treated by established processes. Laboratory work on the lithium system has been focused on small-scale experiments to demonstrate the process steps and to select conditions for engineering-scale tests. The laboratory experiments have led to a better understanding of the interactions that occur between the TRU elements and Li_20 and between Li_20 and rare-earth oxides in the reduction salt. Electrowinning and electrorefining steps have been successfully demonstrated on the laboratory scale. Electrowinning, using a Pt-10 % Rh anode, is the current flowsheet process although other inert anodes are being examined.

Although the lithium process was initially developed for the treatment of LWR fuel, it is directly applicable to other oxide fuels or residues. It is also expected to be usable with other fuels (e.g., carbide, hydride, etc.) with an appropriate head-end treatment.

Session: Spent Fuel Treatment

Engineering-Scale Demonstration of Oxide Spent Fuel Treatment Part I - Operations

T. P. Mulcahey, E. J. Karell, G. K. Johnson, C. C. McPheeters,
R. E. Everhart, R. G. Palm, R. D. Pierce, J. L. Smith,
M. A. Vest and D. W. Warren
Argonne National Laboratory

Argonne National Laboratory Chemical Technology Division is engaged in a program to develop an electrometallurgical process to treat DOE spent fuel. Engineering-Scale tests are being conducted to demonstrate, on a 20 kg of fuel batch size, that the laboratory-scale processes are amenable to process scale up for handling the amounts and kinds of spent fuel in need of processing. The lithium reduction process is key to converting the non-metallic fuels into a metal form suitable for continued electrometallurgical treatment. A successful experimental demonstration of the lithium reduction was performed using 10 kg of simulated oxide spent fuel for feed. Success of the experiment was gauged on the basis of success in the reduction process, the suitability of the equipment designed for the process, and effectiveness of the training and operations performed by the operators. This presentation will describe the equipment and operations involved in the experiment.

Session: Spent Fuel Treatment

Engineering-Scale Demonstration of Oxide Spent Fuel Treatment Part II - Test Results

E. J. Karell, T. P. Mulcahey, G. K. Johnson, C. C. McPheeters,
R. E. Everhart, R. G. Palm, R. D. Pierce, J. L. Smith, M. A. Vest
and D. W. Warren
Argonne National Laboratory

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Session: Issues and Responses to DNFSB 94-1

The Purpose of Recommendation 94-1

William G. Von Holle and Davis Hurt
Defense Nuclear Facilities Safety Board

The production of fissile nuclear weapon components continued at the many Department of Energy facilities until the late 1980's, when such production was ended suddenly. Large amounts of nuclear materials in the pipeline became surplus at Rocky Flats, Hanford, Savannah River and Los Alamos, which have large inventories of unencapsulated plutonium. In addition to plutonium metal and oxide, there are large numbers of containers of plutonium solutions, solid scrap and plutonium compounds. Some of these materials are unstable, some are gas-generating, and there is a growing risk of container breaches and leaks as a result of their packaging, which in many cases was hasty and not intended for long storage periods.

The problems associated with DOE's plutonium inventory caused the DNFSB to issue Recommendation 94-1. The situation has been the subject of many reviews and discussions with representatives of DOE at all levels and with contractor personnel. The hazards of continued plutonium storage at all DOE facilities without stabilization or repackaging, were summarized in a recently issued technical report[1]. The DOE subsequently issued a plutonium vulnerability report,[2] which agreed with the DNFSB report concerning the hazards of continued improper storage, and the deterioration of the situation with time.

The DNFSB issued Recommendation 94-1 in order to expedite the treatment and stabilization of "specific liquids and solids containing fissile materials and other radioactive substances in. reprocessing canyons, processing lines, and various buildings once used for processing and weapons manufacture.[3] Even though plans for remediation of the situation at some sites have been formulated and operations started in some cases, the Board was concerned about the slow pace of activities and concluded that "imminent hazards could arise within two to three years unless certain problems are corrected." The recommendation also calls for repackaging all metal and oxide in accordance with the DOE storage standard by the year 2002. The DOE has submitted an Implementation Plans, which is currently being reviewed by the Board.

Examples of the hazards of improperly stored plutonium metal, oxides, solutions, and solid residues will be briefly discussed, including, how specific actions called for in the recommendation will eliminate the hazards.

1. "Plutonium Storage at Major Department of Energy Facilities," Defense Nuclear Facilities Safety Board Technical Report DNFSB/TECH-1, April 1994.
2. "Plutonium Working Group Report on Environmental, Safety and Health Vulnerabilities Associated with the Department's Plutonium Storage," U. S. Department of Energy, DOE/EH-0415, October, 1994.
3. "Defense Nuclear Facilities Safety Board Recommendation 94-1 to the Secretary of Energy" dated May 26, 1994.
4. Defense Nuclear Facilities Safety Board Recommendation 94-1 Implementation Plan, DOE, February 28, 1995.

Session: Issues and Responses to DNFSB 94-1

LLNL 94-1 Plutonium Stabilization Activities

Brent H. Ives
Lawrence Livermore National Laboratory

The Lawrence Livermore National Laboratory (LLNL) is preparing to stabilize its inventory of plutonium according to the 94-1 recommendation. LLNL has plutonium holdings including metal, oxide and residue materials, all of which need stabilization of some type. Metals are being reviewed for direct contact with plastics, oxides will be stabilized per the DOE-STD-3013-94, and residues are being examined for proper stabilization technique. LLNL is also involved in several thrust areas of the Nuclear Materials Stabilization Task Group including the Integration Working Group, the Research Committee, the Residue Standard development team, and the Small Sites facilities plan. Activities and findings in several of these areas will be reported.

Session: Issues and Responses to DNFSB 94-1

Status of the Plutonium Vault Remediation Effort at Los Alamos

Keith W. Fife
Los Alamos National Laboratory

In 1994, the ES&H Plutonium Vulnerability Assessment and the DNFSB Recommendation 94-1 brought visibility to the complex-wide problems associated with legacy residue inventories from previous weapons manufacturing activities. In response to this problem, Los Alamos is processing residues and separating the plutonium to meet the long-term storage criteria for oxide and metal (>50 wt. % plutonium). This presentation will describe the processing philosophy in operation at Los Alamos, and will cover the current status of our plutonium vault remediation effort.

Session: Issues and Responses to DNFSB 94-1

Solid Residue Stabilization Program at Rocky Flats Environmental Technology Site

Alice M. Murray
EG&G Rocky Flats

The Solid Residue Stabilization Program (SRSP) has been established at the Rocky Flats Environmental Technology Site to remedy the concerns outlined in the Defense Nuclear Facilities Safety Board's (DNFSB) Recommendation 94-1. The solid residues have been grouped into five categories: pyrochemical salts, combustibles, incinerator ash, inorganics, and miscellaneous. The risks associated with each solid residue category have been assessed and ranked. The SRSP schedule is extremely aggressive to meet the DNFSB 94-1 deadlines. The highest risk residues must be treated by the end of 1997 with the exception of combustibles which have been given a one-year extension. The remainder of the solid residues must be treated by May 2002.

This presentation will describe the SRSP's risk assessment, treatment strategies and facility selection for each solid residue category.

Session: Issues and Responses to DNFSB 94-1

Solid Residue Stabilization Strategic Plan

Anthony C. Muscatello
Los Alamos National Laboratory
Transition Technologies Office at Rocky Flats

On January 18-19, 1995, a Solid Residue Stabilization Strategic Plan Workshop was held in Boulder, CO to develop a strategic plan to stabilize residues at the Rocky Flats Environmental Technology Site (RFETS). Both the Defense Nuclear Facilities Safety Board (DNFSB) and the Department of Energy's (DOE) Plutonium ES&H Vulnerability Study have identified some of these residues as "possibly unstable" or as a vulnerability to worker safety and health. This presentation provides the key points covered by the workshop.

Hosted by the Los Alamos National Laboratory's Transition Technologies Rocky Flats Program Office (LAPO), the workshop was designed to define a strategy to allow work to move forward at RFETS to stabilize the residues even with a great deal of uncertainty as to the final disposition of these materials and the plutonium that they contain. For example, the guidance given by the DOEs Office of Environmental Remediation and Waste Management (EM), the RFETS Landlord, is to prepare the residues for discard to the Waste Isolation Pilot Plant (WIPP) in New Mexico. However, WIPP is not yet open and some doubt remains as to its final Waste Acceptance Criteria (WIPP/WAC). The largest uncertainty concerns the acceptability of Land Disposal Restricted (LDR) components in some of the residues because the Non-Migration petition to allow their disposal has not been granted by the EPA.

Furthermore, the discard of the approximately 3 metric tons of plutonium in the residues will result in the generation of a large number of waste drums (about 25,000) because the current WIPP/WAC has severe limitations on the amount of plutonium allowed per drum (200 grams) for criticality safety, and other limitations because of radiation exposure and gas generation which push the limit even lower. There are also indications that the existing volume of Transuranic Waste (TRU Waste) in the DOE Complex already exceeds the capacity of WIPP, without adding the residues and their much higher plutonium load.

For these and other similar reasons, Los Alamos proposed conducting the workshop to develop a strategy which could be agreed upon even by those who disagree about the ultimate disposition of the residues.

Session: Issues and Responses to DNFSB 94-1

A Risk-Based Prioritization for the Stabilization and Repackaging of Nuclear Materials at the Los Alamos Plutonium Facility

Lisa Spanks, Dean Sanzo, Keith Fife, M. K. Sasser and Tresa Yarbro
Contributors: Timothy Hayes, Michael Palmer, Wayne Punjak, Roland Hagan,
Victoria Longmire, Cindy Mills, Nelson Demuth and Charles C. Thomas, Jr.
Los Alamos National Laboratory

In response to the Defense Nuclear Facility Safety Board (DNFSB) 94-1 recommendations, Los Alamos National Laboratory initiated an effort to stabilize and repackage the over 5,000 items in its plutonium facility (PF-4) storage vault over a period of 8-15 years. Before beginning this extensive project, the personnel at Los Alamos determined that it was necessary to understand the storage vault inventory by collecting certain data on a small, but "statistically significant", sampling. The approach included the following steps:

- Breakdown the vault inventory into groups based on risk factors (i.e., age of packaging, type of material).
- Select a sampling of vault items for inspection.
- Perform container inspections and collect the data from each of these inspections.
- Use these results to assess the risk and prioritize items for repackaging.

The risk-based prioritization analysis, using the standard definition of risk (probability times consequence), begins with the development of accident scenarios and uses the data collected during the container inspections to determine the necessary probabilities. Consequences are defined in the areas of worker safety and health, laboratory and DOE missions and programs, and cost of the clean-up of an inadvertent release. Using risk analysis software developed at Los Alamos, the repackaging prioritization of items in the PF-4 storage vault is determined. This talk will present more on the background of this project, results of the container inspections, a more detailed explanation of the risk analysis technique, results of the prioritization, and information on the continuing work being done at Los Alamos to address the packaging concerns of the DNFSB.

Session: Issues and Responses to DNFSB 94-1

RFETS Solutions Stabilization Flowsheet Development

Stephen B. Schreiber, Elaine M. Ortiz, Benjie T. Martinez, Charles W. Kranker,
Robert L. Schempf, Thomas T. Butler and Richard L. Ames
Los Alamos National Laboratory

This presentation describes the precipitation process to be used at the Rocky Flats Environmental Technology Site (RFETS) to treat nitrate solutions containing high levels of plutonium (greater than 6 g/L). The precipitation process was demonstrated by the Los Alamos National Laboratory - Nuclear Materials Technology in the plutonium facility (TA-55). The process demonstrated consists of plutonium (III) oxalate precipitation followed by plutonium (IV) hydroxide precipitation of the filtrate, using full scale RFETS process simulation laboratory (PSL) equipment. The demonstration included the assistance of EG&G Rocky Flats Experimental Operators and resulted in a demonstrated procedure to be incorporated into RFETS operations.

Session: Issues and Responses to DNFSB 94-1

Stabilization of Americium/Curium Solutions by Vitrification

T. S. Rudisill, W. G. Ramsey, R H. Jones, Jr., T. F. Severynse
Westinghouse Savannah River Company

Approximately 15,000 liters of solution containing isotopes of americium (Am) and curium (Cm) are currently stored in F-Canyon at the Savannah River Site (SRS). These isotopes were recovered during plutonium-242 production campaigns in the mid and late 1970's. The continued storage of these solutions was identified as an item of urgent concern in DNFSB Recommendation 94-1. Currently there are no existing SRS facilities which can be used to stabilize this material for safe interim storage or transport to the heavy isotope programs at Oak Ridge National Laboratory. An analysis of several alternatives has resulted in the recommendation to stabilize the Am/Cm in a borosilicate glass. The Multi-Purpose Processing Facility in F-Canyon will be used for the vitrification process. Pretreatment operations will be performed in canyon vessels to separate the actinides and lanthanides from other impurities (primarily iron) before subsequent vitrification.

Pretreatment operations for the Am/Cm program include adjusting the nitric acid ($HN0_3$) concentration of the solution to <1M, precipitating the actinides and lanthanides as oxalates, washing soluble metallic impurities from the precipitate slurry, solubilizing the precipitate, and adjusting the $HN0_3$ concentration prior to vitrification. The proposed flowsheet for these operations is based on previous development work and plant operating experience. Specific experimental needs include demonstration of the oxalate precipitation to measure the solubility of metallic impurities which have an impact on the glass formulation and measuring the settling rate and specific volume of the precipitate slurry to allow design of the solution transfer equipment. The results of the pretreatment experiments will be used to develop a target glass composition for detailed process design. The primary processing parameters, glass viscosity and glass liquidus, will be optimized. This will allow the glass frit composition to be more accurately determined as well.

An overview of the Am/Cm vitrification program will be presented highlighting both the pretreatment and vitrification development programs.

Session: Issues and Responses to DNFSB 94-1

Unstable Ash Characterization and Stabilization

Richard A. Van Konynenburg, David H. Wood,
Ralph H. Condit and Mark C. Bronson
Lawrence Livermore National Laboratory

Plutonium-oxide-containing reacted ash residues in storage in food pack cans at LLNL were observed to produce gas, pressurizing their containers. The pressure was measured and relieved, and samples were taken of the gas and the residues. Mass spectrometric analysis was performed on the gas samples, and the residues were analyzed for weight loss on heating, sulfur and carbon content, and pitting upon moistening.

The pressurized cans that were sampled were found to fall into two categories. The first category exhibited oxidizing conditions in the gas and basic pH in the residue. The second category showed the opposite conditions for both redox and pH.

We have hypothesized radiation chemical mechanisms for the observed behavior that depend on the relative amounts of available oxygen and carbon. The first category of cans apparently had insufficient carbon to use up the available oxygen from the initial air and adsorbed water. Conditions therefore remained oxidizing, and the carbon dioxide produced by radiolysis was sorbed on the residue and converted to carbonate, displacing adsorbed nitrogen, which pressurized the can.

In the second category, the oxygen was used up by reaction with carbon, eventually forming carbon monoxide, which sorbed on the residue. Under basic conditions, carbon monoxide and water are known to undergo a chain reaction, producing formic acid. This apparently drove the pH down, causing carbon dioxide to be desorbed from the residue. The can was then pressurized by carbon dioxide and hydrogen, the latter coming from radiolysis of adsorbed water.

Stabilization of the ash is in planning. It will consist of washing to remove salts, and recalcining at higher temperature with ample oxygen to drive off adsorbed water and to burn off carbon. The recalcined ash will be maintained under dry atmosphere conditions until it is repackaged in dry inert gas.

Session: Issues and Responses to DNFSB 94-1

Thermal Stabilization of Plutonium Bearing Scrap

Lane T. Cunningham
Westinghouse Hanford Company

Operation of a thermal stabilization process to stabilize reactive plutonium bearing material stored at the Plutonium Finishing Plant (PFP) has started. This inventory of unstable plutonium (Pu) bearing materials does not meet DOE criteria for secure, long term storage. The source of these materials is process residuals and glovebox floor sweepings from the production of defense material at the Plutonium Finishing Plant (PFP). The stabilized material is being packaged for long term storage in one of the vaults at PFP.

Feed material is heated to 1000°C and held at this temperature for one hour to drive off residual volatile components and convert residual plutonium materials to plutonium oxide. For material containing organics, a CO_2 cover gas is added to the furnaces to prevent potential safety hazards from the formation of flammable gases. At this higher temperature, the plutonium oxide is high fired, and becomes less susceptible to absorption of moisture. After firing, the material is sieved through a 4-mesh screen (1/4"), sampled, analyzed for Loss-On-Ignition (LOI), and packaged for storage.

Formation of fluid in the off-gas tubing during furnace operation created operational difficulties during initial processing. A series of changes to the off-gas system have taken place to reduce the effects of the condensation in the off-gas system.

Session: Issues and Responses to DNFSB 94-1

A Proposed Standard for Safe Storage of Department of Energy (DOE) Plutonium Contaminated Residues

Larry R. Crisler
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Nuclear Materials and Reconfiguration Technology Technologies Office at Rocky Flats

D.T. Thorp
EG&G, Rocky Flats Plant

The DOE currently manages a significant quantity of bulk materials (total of 33,109 items, >100,000 kgs, in the DOE residue inventory) contaminated with plutonium (>6,000 kgs), defined as solid process residues. The residues represented feedstock and materials-in-process to nuclear weapon fabrication and nuclear material production until fabrication ceased in 1989. The residues contain materials such as impure plutonium oxides, reactive metals, halide salts, combustibles, ash, dissolver heels, sludges, contaminated glass and metal, and other miscellaneous items. The DOE Implementation Plan to address DNFSB 94-1 Recommendations, February 28, 1995 indicates the following quantities of solid residues at the various DOE facilities; Rocky Flats, 3,000 kgs, 20,532 Items; Hanford, 1,500 kgs, 5,000 Items; Los Alamos, 1,400 kgs, 6,300 Items; Savannah River Classified, 1,306 Items; Lawrence Livermore, 35 kgs, 182 Items; Mound, 3 kgs, 39 Items; Argonne East, <1 kgs, 12 Items; Oak Ridge, 0.1 kgs, 12 Items; Lawrence Berkeley, <1 kgs, 250 Items. Within the solid residue inventory, many of the stored forms are corrosive, chemically reactive and difficult to contain, particularly when they are exposed to air and moisture. Hazards are generated as a result of either poor package design or packaging failure stemming from radiolysis and pressure buildup which contribute to the problem. Like other forms of plutonium, residues in contact with plastics cause radiolysis, hydrogen generation, and pressurization, making these packages susceptible to leaks or ruptures. Many packaging failures have occurred already. These failures have involved highly corrosive salts, fluoride-based reduction slag's, plutonium oxide, and incinerator ash among others. Clearly, not all materials and packaging weaknesses within the inventory have been identified or adequately characterized. In fact, the long-term storage properties of materials are not well known. Action is needed both to respond to emerging hazards as well as to improve understanding of the long-term stability of these residue materials. This effort is focused on arriving at the most desirable pathway to the acceptable end-state (safe storage), using efficiency, cost, ALARA, waste, and facility constraints as elements of the acceptance criteria. Since 1989 these residues have remained in packages in processing areas, vaults, and process lines awaiting disposition. They are not currently in a configuration/condition suitable for long-term storage.

Processing, treatment stabilization, and/or repackaging are required to secure them in a safe, stable end-state. These materials will be stored for periods up to 50 years. The recently issued DOE-STD-3013-94, Criteria for Safe Storage of Plutonium Metals and Oxides (December 1994) can be used as a template for developing Criteria for Safe Storage of DOE Residue/Waste materials until final dispositioning options have been established and implemented.

A draft Criteria for Safe Storage of DOE Residue/Waste materials has been developed and is in the process of being revised per input from DOE technical experts and the residue interim storage (up to 50 years) sites. This paper will review the technical basis for the developing criteria for interim safe storage of DOE residue inventories.

Session: Issues and Responses to DNFSB 94-1

A Technical Basis for Safety Surveillance of Stored Plutonium Metal and Oxide

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Transition Technologies Office at Rocky Flats

Driven by changes in mission (from nuclear weapons production to site clean-up) and Recommendation 94-1 by the Defense Nuclear Facility Safety Board (DNFSB), attention at DOE facilities housing plutonium metal and oxide has shifted to include stabilizing and packaging these materials for safe storage. Previously, plutonium metal had been continually recycled through production lines. Similarly, plutonium oxide was processed to recover metal for feed to manufacturing operations. In both cases metal or oxide were packaged for relatively short periods and the opportunity for problems to develop was small. Now these materials will be stored for periods that could exceed 50 years. There is concern that pre-packaging processing to clean metal pieces and stabilize oxides (including removal of moisture) will indeed be adequate to prevent problems such as container pressurization that have been encountered in recent years.

The recently issued DOE-STD-30130-94 Criteria for Safe Storage of Plutonium Metals and Oxides (December 1994) covers material preparation packaging and basic requirements for safety surveillance. As safety concerns ought to be common at the various sites where these materials are stored there should be a mutually agreed upon technical basis on which individual surveillance programs will be designed. To this end a meeting was held involving technical experts on the metallurgy, chemistry and storage of plutonium from Rocky Flats, LANL, LLNL, and Hanford (with outside participation by Savannah River). These experts discussed what changes in the stored materials and their containers were important to monitor in order to assess the safety of stored packages.

A consensus was reached that the following parameters were necessary and sufficient for surveillance monitoring: 1) Weight, 2) Container leak tightness, 3) Internal container pressure (for stored oxides), 4) Container corrosion and 5) External contamination. These do not necessarily require sophisticated or separate measurements. For example, after performing initial leak tests the integrity of containers can be monitored over time through other data such as weight change. The greatest challenge will be determining gas pressurization of oxide containers, particularly when both material and boundary containers are present.

Other parameters such as temperature and internal gas composition would be desirable to collect data on, though not critical, and thus might be included in a special surveillance program to gather intensive data on a selected subset of stored packages.

This paper will review the technical basis for safety surveillance including considerations discussed at the meeting of technical experts. Some options for collecting data will be described, though each site will ultimately build its own surveillance program around existing capabilities or those that can be reasonably added. Finally, a two-tiered approach to surveillance will be discussed where both basic and intensive data collection are used to status the safety condition of stored packages and also validate predictions regarding behavior of metals, oxides and containers over time.

Session: Issues and Responses to DNFSB 94-1

Impacts of 94-1 Stabilization Activities on Pu Disposition Options

Guy A. Armantrout
Lawrence Livermore National Laboratory

The DOE Fissile Materials Disposition Program is evaluating options for the long term storage or disposition of surplus Pu to reduce future proliferation risks. The principal approaches are: 1) partial burn of the Pu in a nuclear power reactor, with the remaining Pu discarded in a spent fuel matrix, 2) immobilization of the Pu in a matrix acceptable to a possible high level waste repository with a spiking radionuclide for proliferation protection, 3) direct discard of the Pu materials in a deep borehole, and 4) storage in a new storage facility pending future disposition decisions.

Potential 94-1 processing, whose aim is to stabilize existing inventories to assure safe interim storage, will impact the different alternatives in different ways. If all material is processed to meet the current DOE interim storage standard for metals and oxides, then the resulting high-fired oxide will complicate any disposition options which require dissolution of the oxide. Such dissolution operations include the front end purification of the plutonium for the MOX fuel fabrication cycle or the dissolution required to include the plutonium oxide in a ceramic disposition matrix.

Additional complications will occur if stabilization of the residues involve any immobilization of low content matrices in waste forms which are not acceptable for final disposition. An example would be grouting of the ash as an interim measure. Such an interim form would make processing very difficult for any of the current disposition options. Conversely, if 94-1 processing and stabilization involves extensive purification and concentration of the residues, then this processing would be "wasted" for disposition options such as glass immobilization which can directly accept many of the current residue forms.

Clearly, an integrated program plan is required regarding the treatment of current inventories to meet 94-1 stabilization requirements in order to preclude wasted work now or the possibility of greatly complicating future disposition activities.

Electrochemical Processing using Cadmium Electrodes

Z. Tomczuk, J. J. Heiberger and W. E. Miller
Argonne National Laboratory

Plutonium from a Pu-l wt % Ga alloy was recovered by dissolving the alloy in liquid cadmium at 500°C and then electrochemically transporting the Pu to a liquid cadmium cathode using a LiCl-KCl-$PuCl_3$ electrolyte. Pure Pu was then obtained by distilling off the cadmium. Analytical results indicated that the product Pu contained <10 ppm Ga. These results also indicated that all of the added Pu was transported. Prior to testing, a thermodynamic model was developed to help predict the upper limit of solubility of the Pu-Ga alloy in liquid cadmium. Our experimental results are consistent with model prediction.

Session: Pyrochemical Technologies

Recovery of Plutonium Metal by a Hydride/Dehydride/Casting (HYDEC) Process

Carlos Colmenares, Mark C. Bronson and Terry E. Ludlow
Lawrence Livermore National Laboratory

A multi-chamber apparatus for the recovery of large amounts of Pu metal (up to 4 kg), in various geometric shapes, by a hydride/dehydride/cast process (HYDEC) has been built and tested. The hydriding step is carried out in an upper chamber where the Pu-metal is converted to PuH_x which is in the form of loose black powder ($T < 100°C$) or metallic-looking flakes ($T \sim 250$-$350°C$), the latter being the preferred form. The hydride powder or flakes fall into a crucible within the hot zone of a furnace, below the upper chamber, where PuH_x is decomposed into liquid Pu and hydrogen, and the latter recycled to hydride more metal. This recycling greatly reduces (~80%) the amount of H_2 normally required for the stoichiometric reaction. Because the crucible temperature is above the melting point of Pu (640°C), the Pu recovered is molten and is subsequently poured by a special valve through the crucible bottom into a large mass copper mold.

The experimental technique will be described in detail and results without H_2 recycle and partial H_2 recycle presented. We will also give the results of an experiment performed to calibrate the temperature readout of the infrared camera used to measure plutonium temperature.

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract number W-7405-ENG-48.

Session: Pyrochemical Technologies

Processing of C-Saturated Tantalum Hardware for Actinide Pyrochemical Processing

Gerald L. DePoorter
Colorado School of Mines

Keith Axler, Ramiro Pereyra. and Peter Lopez
Los Alamos National Laboratory

A mechanism of deterioration of Ta metal containing molten plutonium is by attack at the grain boundaries and the removal of entire grains of the metal. Carbon-saturated tantalum hardware has been shown to be a suitable material for the processing of Pu in molten salts. Carbon-saturated tantalum consists of precipitates of Ta_2C on the grain boundaries and inside the grains. The Ta_2C on the grain boundaries prevent molten plutonium from attacking the grain boundaries. The results of a detailed kinetic study of the precipitation of Ta_2C from Ta metal saturated with carbon will be presented. Extension of the kinetic data to practical processing of carbon-saturated tantalum hardware for actinide pyrochemical processing will be described. Experimental results on the performance of this material in molten salts-plutonium systems will also be presented.

Session: Pyrochemical Technologies

Uranium Separation and Transport in the Molten Salt Reactor Experiment

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D.H. Metcalf and S. Dai
Oak Ridge National Laboratory

The Molten Salt Reactor Experiment, MSRE, at Oak Ridge has been shut down since 1969 when the fuel salt was drained from the core into two Hastelloy N drain tanks at the reactor site. In January 1994, analytical measurements of gas samples taken from the gas piping circuitry connected with the drain tanks still containing the fuel salt indicated the presence radiolytically generated fluorine, 350 mm Hg, and uranium hexafluoride, 70 mm Hg.

Although fission product radiolysis was known to generate F_2, the formation of UF_6 and its transport from the fuel salt was unexpected. These gaseous radiolysis products have moved through the gas piping to a charcoal bed where reaction with the activated charcoal has produced C-F compounds and probably also, UF_4.

The chemistry of this novel separation of uranium from the solid fuel salt and subsequent transport to the charcoal beds will be discussed and measurements relating to establishing the rate of UF_6 generation relative to that of F_2 will be described. In addition, the stability of the products formed on the charcoal beds and current remediation operations will be presented.

Pyrochemical Techniques for the Separation of Actinides

David Riley
Science Application International Corporation

Bart Ebbinghaus and Mark Bronson
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There are large quantities of actinide containing byproducts, residues and wastes at DOE and commercial facilities. These materials include: residues from the nuclear weapons production facilities, high-level calcine stored at the Idaho National Engineering Laboratory (INEL), fuel rods from nuclear reactors, and waste salts from the uranium reduction. Direct disposal of these materials can be very expensive and consume large portions of proposed waste repositories. If the actinide components were separated, then the majority of the waste could be disposed of as low-level or non-radioactive.

The actinides can be separated from the non-actinides by many different methods. Traditionally aqueous methods have been used to dissolve the actinide containing compounds. Techniques such as ion exchange, solvent extraction, and precipitation have been used to separate the actinides. However, the aqueous processes generate significant amounts of waste solutions that requires additional processing.

Another set of techniques that are available to separate actinides from byproducts, residues and wastes are pyrochemical in nature. Some of these techniques include chloride volatility, salt scrub, and salt filtration. Chloride volatility takes advantage of the differences in vapor pressure between various metal chlorides or chlorides and oxides. This technique has been applied to the separation of actinides in electrorefining anode heels, INEL high-level calcine and pyrochemical salt. The salt scrub process uses a reducing agent to convert actinides to metal and recover them. LLNL has developed a continuous salt scrub process that can remove actinides from materials such as residue salt cakes and uranium reduction waste salts. The salt filtration process is the filtering of molten salt solutions to separate solid oxides from molten salts. It has been used to remove ceramic scraps from residue salts and separation of INEL oxides from salts they contained.

This paper will give an overview of these pyrochemical techniques, how they have been used and how they can be applied to separate actinides. Some of the materials examined will be plutonium pyrochemical salts, INEL calcine, and uranium reduction waste salts.

Session: Pyrochemical Technologies

Distillation Separation of Chloride Salts From Plutonium

Eduardo Garcia and Vonda Dole
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There are a number of residue and waste salts throughout the DOE Complex that have resulted from past pyrochemical processing of plutonium. These salts contain large amounts of plutonium and require treatment for disposal. Distillation separation, of the salt matrix from the actinide content, is a potential treatment technology. This separation process should be capable of producing chloride salts discardable as Low Level Waste (LLW) and a concentrated actinide oxide powder that would be stored as Special Nuclear Material (SNM).

Distillation separation is a simple physical process based on the large difference in vapor pressures between most chloride salts that constitute pyrochemical residues and the actinide oxides. Various actinide species are present in the salt residues, but pre-treatment oxidation processes have been developed to ensure that all actinide components are converted to oxides.

Experiments performed, with oxidized sodium chloride-potassium chloride salts containing plutonium, have shown that distillation separation is viable: the plutonium content of the salt has been reduced from tens of percent to the ppm and sub-ppm range. Approximately 1/4 of distilled salts have been below the LLW criterion. Numerous control experiments with uncontaminated salts in the glovebox have shown that the residual plutonium content of distilled salts is consistent with glovebox background contamination levels. The results of the control experiments support the conclusion that essentially complete and clean separation of the salt from plutonium oxide can be achieved. New equipment, scaled to production levels, is being designed to allow removal of purified distilled salts without exposure to high contamination levels within the glovebox.

Session: Pyrochemical Technologies

Vacuum Evaporation of KCl-NaCl Salts: Vaporization Rate Model and Experimental Results

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A promising means to reduce the storage space and the disposal cost of pyrochemical waste salts is by separating the salts, typically mixtures of chloride salts, from the actinides via a vacuum evaporation process. A simple rate modeling approach based on the Hertz-Langmuir relation was developed and applied to the KCl-NaCl binary system in conjunction with experimental parameters and data obtained from vacuum evaporation experiments in the temperature range of 640-760°C. The effective evaporation coefficient which is the fraction of the theoretical evaporation rate actually realized ranged from 0.1 to 0.4 in these experiments and was found to be temperature dependent. Moreover, this work also included examination of composition and microstructure of the condensate to gain insight to the condensation process. The results generated from this study leads to discussion of other mass and heat transfer factors which are not accounted for in this simple evaporation rate model and also to discuss the relevant engineering considerations in optimizing the equipment design for this evaporative separation process.

Session: Pyrochemical Technologies

TRUMP-S: Pyropartitioning of Actinides from PUREX Wastes

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TRUMP-S (TRansUranic Management by Pyropartitioning Separation) is a pyrochemical process being developed for the recovery of actinides from PUREX wastes. The process is being developed at CRIEPI (Central Research Institute of Electric Power Industry of Japan) as part of Japan's OMEGA Program to reevaluate the nuclear fuel cycle. The process consists of four steps: (1) denitration of PUREX wastes, (2) chlorination, (3) reductive extraction, and (4) electrorefining. Rockwell is participating in the laboratory-scale development of the third and fourth steps, which are chemical and electrochemical pyropartitioning steps, respectively, in which the actinides are partitioned from noble metals, lanthanides, and active metals. The program is conducted by CRIEPI, Kawasaki Heavy Industries of Japan, Rockwell International, and the University of Missouri-Columbia. A special laboratory designed to provide a high purity argon atmosphere and alpha activity containment for U, Np, Pu and Am has been established at the Missouri University Research Reactor.

Progress to date, performance capabilities of a custom designed ICP-MS, and applications to DOE wastes for TRUMP-S will be reviewed in this presentation. Progress to date includes thermodynamic property measurements of actinides and lanthanides in process solvents, measurement of distribution coefficients, evaluation of process kinetics and mass transfer characteristics on a laboratory scale, and preliminary separation tests. Initial laboratory tests indicate that almost all (>99%) of the actinides can be reduced into molten Cd from the chlorinated PUREX waste by addition of a Li alloy. Tests with PUREX-type proportions of actinides and lanthanides have demonstrated that >99% of actinides can be removed from the molten Cd anode and the LiCl-KCl salt phase during electrorefining. In order to meet the more demanding analytical requirements for future tests, an Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) has been acquired to replace an ICP-AES (Atomic Emission Spectrometer). The ICP-MS and its enclosure have been modified to comply with NRC and EPA regulations. A dynamic computer model (not an equilibrium model) based on the thermodynamic measurements, kinetics, and mass transfer characteristics has been developed to predict process performance, and has been verified in tests with rare earths. The model predicts that 90% product actinide content can be achieved through 99% actinide removal from PUREX wastes, and further development work is under way to realize this objective experimentally. Production of a less pure product would allow decontamination of TRU materials to non-TRU levels.

Session: Disposition Technologies

AWE Plutonium Strategy

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Strategies have been developed for different scenarios, which vary from considering either all plutonium to be excess or that all plutonium is a strategic reserve. The facilities, processes and storage requirements necessary to meet the different scenarios are considered. This unclassified presentation gives an overview of the work carried out in developing these strategies, and concentrates on the storage and processing requirement necessary to meet the different scenarios.

Session: Disposition Technologies

Chipless Pit Bisector

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The capability to easily disassemble pits is the first step in processes for plutonium reuse or disposition. This paper describes a chipless pit bisector being developed at LLNL for automated disassembly of most pit types that are in, or have been retired from, the US stockpile. The design is based on:

- Lessons learned at LLNL in FY93 with the proof of concept demonstration in which a bisector with a backed chipless cutter was developed, then used to part a welded stainless steel sphere and the LLNL glovebox robot was used to load each sphere in the bisector, engage the holding fixture, advance the cutting tool during the bisection, and unload/segregate the bisector parts into respective waste bins. (Ref.1)
- The special attributes for the pit bisector pertinent to disassembly were derived from pit configuration and materials properties delineated in the stockpile survey recently completed at LLNL. (Ref 2)

The key features of the bisector are:

- A rotary table oriented vertically for ease in robotic handling of the parts and materials in support of the bisecting operations.
- A pit holding fixture which is a vacuum chuck designed for ease of horizontal pit waist alignment.
- A chipless roller cutter on a platform stage attached to an annular-shape frame surrounding the rotating pit. This frame has a three point mount with a vertical adjustment to align the cutter with the pit waist, and lateral compliance to accommodate slight eccentric run-out on the pit waist.
- A roller cutter subassembly that is backed by two idler rollers also attached to the frame which together form a three-point contact on the pit waist to provide a balanced support for the high forces developed during the parting process.
- A frame subasssembly which can be pivoted out of the way for exchanging vacuum chucks to accommodate differing pit sizes.

The bisector has been designed, fabricated, successfully cold tested and readied in a plutonium glovebox for hot testing.

References:

1. Roy Merrill, "Application of an Interactive Controlled Gantry Robot in glovebox Environment, " International Robotics and Vision Automation Show and Conference, Detroit, Michigan, April 1993.
2. Roy Merrill, "Survey of Pit Disassembly Parameters and Discussion of Disassembly Methods," LLNL Document No. CLYA-95-0007, January 19, 1995 (Secret)

Session: Disposition Technologies

Preparation of PuO_2 from Weapons Grade Pu for use in Mixed Oxide Reactor Fuel Pellets

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We have prepared PuO_2 powders from weapons grade Pu by the HYDOX process, which can be carried out by: 1)Hydriding the metal, Dehydriding the PuHx powder and Oxidizing the metal powder; or 2) Hydriding the metal followed by Oxidizing of the Hydride powder. We found that the second method was the most efficient in terms of the time it took to complete the conversion. PuH_2 powders were prepared at relatively low (100 - 150°C) and high temperatures (350 - 400°C) and the oxidation step was carried out under similar temperature ranges. Both the Hydride and Oxide powders were characterized by sieve analysis to obtain their particle size distribution. The morphology of the oxides, prepared under different conditions, was obtained by SEM.

Session: Disposition Technologies

Electrolytic Decontamination of Oralloy - A Disposition Task

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Throughout the DOE complex there are holdings of contaminated oralloy (highly enriched uranium). Additionally, there are contaminated oralloy parts being produced as part of weapons dismantlement activities. Decontamination of oralloy to swipable plutonium (Pu) and americium (Am) levels $\leq$ 20 disintegrations per minute (dpm)/100 cm^2 is required for shipment of holdings to Oak Ridge Y-12, the nation's disposition site for oralloy. At the Los Alamos National Laboratory plutonium facility, we have successfully demonstrated the electrolytic decontamination of Pu and Am from oralloy. This work started as a joint project with Rocky Flats Environmental Technology Site (RFETS) to find a better method of oralloy decontamination than the hot concentrated nitric acid spray leach process that had been used at RFETS. We have found that not only is the electrolytic method more effective, it also forms a solid waste product that is > 99.9% less in volume than the liquid acid waste produced from the acid spray leach process.

The technical approach of electrolytic decontamination is similar to the common industrial practice of electropolishing. Applying a small voltage across an electrolyte induces anodic dissolution of the contaminants. For our system, a precipitate then forms that leads to easy removal of the contaminant from the electrolyte solution. This precipitate removal enables recycle of the electrolyte. (If ever needed, the electrolyte solution can be discarded as an industrial liquid waste-the radioactivity is less than 6 X 10^5 counts per minute (cpm)/liter.)

Numerous parts have been cleaned with this technique. Recent fixture modifications and upgrades have been successful. Shelf life tests of cleaned parts have not shown any appreciable amount of increase in swipable contamination over time-contamination weeping.

Session: Disposition Technologies

Immobilization of Plutonium in Glass: Issues and Approaches

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It is possible that excess Pu from weapons dismantlement or production will be vitrified for final disposal in a geologic repository. The information available on the formulation and performance of a high-loaded Pu glass is limited. In this paper, the results of experiments that evaluate the performance of high-loaded Pu glasses are presented. The experiments discussed are accelerated by use of parameters that affect glass reaction, and the performance of the glass is measured by the release of Pu to solution, the co-reaction of Pu with potential poisons that were added to the glass, and by the distribution of Pu throughout the reacted glass. Based on the results of these experiments, suggestions are made regarding the interaction between glass formulation, performance, and processing.

Surplus Pu to Pu-Glass using Existing F-Area Facilities at SRS

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Westinghouse Savannah River Company

The DOE is reviewing long-term options for surplus fissile material control and disposition, taking into account technical, nonproliferation, environmental, and economic considerations. This review is being implemented through the Surplus Fissile Material Control and Disposition Project and the preparation of the Programmatic Environmental Impact Statement (PEIS) for Long-Term Storage and Disposition of Weapons-Usable Fissile Material. Options under consideration for the long-term disposition include indefinite storage, minimized accessibility, and elimination.

SRS is proposing the use of existing facilities in F Area to convert surplus plutonium to a condition of minimum accessibility from which the plutonium can be ultimately dispositioned to use in a reactor or direct disposal. The process would include pit disassembly, plutonium dissolution, conversion to plutonium glass logs (vitrification), and interim storage. Both plutonium metal and oxide could be processed. Any HEU components resulting from the pit disassembly could be processed and isotopically diluted to LEU at SRS for use in commercial reactors or disposal, or they could be shipped off-site for disposition.

Adequate space would be available in existing F-Area facilities to house the shipping, receiving, processing, and storage functions. This would include currently uncontaminated facilities, e.g., the new Plutonium Storage Facility (PSF), the New Special Recovery (NSR), and the third-level of the current plutonium finishing line (FB-Line). The little-used Multi-purpose Processing Facility (MPPF) is scheduled to be modified to house a vitrification process for stabilizing the existing americium/curium solutions stored in F-Canyon. These modifications can be expanded to meet the requirements for plutonium vitrification. Most of the remaining F-Area processing facilities would be available when the current stabilization and de-inventory mission is complete. Operation of the large canyon process would not be required and the space could be utilized for interim storage of the plutonium glass logs. The existing SRS infrastructure, including waste treatment and analytical laboratories, would be available to support the process. The finished project would be capable of vitrifying 5 metric tonnes of plutonium per year.

Immobilization of Actinides in Geomedia by Phosphate Mineralization

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A method is being developed to reduce the solubility of actinides in geomedia by conversion to a thermodynamically stable mineral form. The programmatic objective is to transform actinide ions in the near surface environment to less-soluble, less-reactive, thermodynamically stable phosphate mineral phases through the application of surface active organophosphorus complexants. These organophosphorus species are designed to decompose slowly in the environment to release phosphate. The slow-release of phosphate promotes the formation of stable mineral phases while minimizing the potential for application of excess phosphate. The complexant of choice is myoinositol (hexakisphosphoric acid) or phytic acid. We have determined that phytic acid decomposes at a convenient rate, that phosphate minerals are formed as a consequence of the decomposition, and that the lanthanide/actinide phosphates do reduce radionuclide solubility under environmental conditions. This technique also may have important applications in the stabilization of the tailing piles produced as a result of mining operations for uranium/thorium or strategic metals.

Session: Disposition Technologies

Excess Pu Disposition in Zirconolite-rich Synroc

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Australian Nuclear Science and Technology Organisation

The design philosophy of a hot-pressed zirconolite-rich Synroc variant for immobilizing excess weapons Pu is summarized. A multi-phase aggregate of compatible Synroc phases allows chemical flexibility in terms of tolerance to variations in the Pu/precursor weight ratio, and helps to minimize the grain size. The presence of hollandite allows the incorporation of radioactive Cs/Ba to reduce diversion risks. Microstructural data of ceramics containing ~20 wt. % of Pu and ~0.3 wt. % of inactive Cs are presented to illustrate the design strategy.

A description is given of background programs involving:

- the replacement of zirconolite by pyrochlore to increase the Pu waste loading;
- the available chemical substitution mechanisms of Pu and Gd in zirconolite, as studied by X-ray diffraction and microanalysis;
- the partitioning of Pu in zirconolite and perovskite;
- phase analysis of Synroc variants containing different proportions of zirconolite, Pu and Gd
- the influence of chloride on Synroc;
- the production of Synroc and zirconolite-rich ceramics via a microsphere precursor; and
- the zirconolite--> perovskite instability in very reducing conditions and oxygen pressure effects.

Excess Pu Disposition in Zirconolite-rich Synroc containing Nepheline

V.M. Oversby
Lawrence Livermore National Laboratory

E.R. Vance, K.P. Hart, R.A. Day, B.D. Begg
P.J. Angel, E. Loi and J. Weir
Australian Nuclear Science and Technology Organisation

A titanate ceramic designed to consist of 70 wt. % zirconolite ($CaZrTi_2O_7$), and 15 wt. % each of nepheline ($NaAlSiO_4$) and rutile (TiO_2), and containing 10 wt. % of Pu plus 6 wt. % of Gd as a neutron poison, was fabricated by hot-pressing at 1150-1250°C. The nepheline was inserted to lower the consolidation temperature. Detailed microstructural work showed that some of the Na targeted for nepheline had entered zirconolite and some of the minor perovskite ($CaTiO_3$) phase which had formed. Glassy aluminosilicate regions were present, though some devitrification to form nepheline had occurred.

Leaching data on half-disc samples, 10 mm in diameter and about 2 mm thick were obtained at 90 to 200°C by MCC-type methods using deionized water. Seven and 28-day leach rates ($g/m^2/d$) at 90°C of the aluminosilicate phase were in the range of 1-10, and they were a factor of ~ 10 higher at 200°C. Leach rates of Ca were ~0.1, those of Gd, Ti and Zr were $\sim 10^{-3}$, and those of Pu were $\sim 10^{-5}$; the increase in leach rates at the higher temperature was less than that for the aluminosilicate material. These leach rates are discussed in terms of previous leaching studies of Pu from Synroc-C, the Synroc variant originally designed for PUREX waste.

A Zirconium Metal Alloy for the Immobilization of Surplus Fissile Materials

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The immobilization of surplus plutonium from dismantled nuclear weapons may be accomplished by alloying plutonium with other metals to form a strong, corrosion-resistant metal. Stainless steel - zirconium (SS-Zr) alloys are being tested as metal waste forms for the disposal of metal nuclear wastes isolated during the electrometallurgical treatment of spent nuclear fuel (SNF). The nominal alloy waste form compositions for SNF treatment are Zr - 8 wt % SS and SS - 15 wt % Zr. These alloys are highly corrosion resistant and mechanically strong; corrosion and mechanical tests have been done on the base alloys.

We are currently extending our present knowledge of the SS-Zr alloy system by including up to 10 wt % Pu in the alloys. The Zr - 8 wt % SS is the primary alloy candidate for Pu immobilization based upon our current understanding of the alloy phase morphology and corrosion behavior. However, 2 to 10 wt % Pu has been included in both of the nominal alloys to survey the effects of actinide inclusion in either system. The alloy microstructures will be discussed.

One of the primary goals of the Pu immobilization effort will be to make the immobilization form self-protecting through incorporating radioactive fission products separated from spent nuclear fuel. The most effective radionuclide for long-term protection is Cs-137 ($t_{1/2} = 30$ yr); it can provide significant radiation doses for a few hundred years. Although spiking the metal alloy directly with cesium is not favored because of its immiscibility in the liquid alloy and extreme volatility at the alloying temperatures (1200°C to 1600°C), mechanical methods for incorporation of Cs compounds in the metal alloy are being explored.

Another alternative that was considered is the addition of other actinide metals and beryllium to form $AcBe_{13}$ precipitates in the alloy microstructure. The actinide-beryllium compounds would serve as a neutron source through (α,n) reactions. Benchmark calculations made using the MCNP code indicate that the resulting neutron doses would be at most ~5rem/hr (at 1 m), well below the 1000 rem/hr level proposed for self-protection. As a result, an (α,n) neutron source alone is not considered an option, but it may complement other self-protection measures.

Session: Disposition Technologies

Use of Spent Fuel Treatment Technology for Plutonium Immobilization

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Pyroprocessing of spent nuclear fuel can naturally incorporate additional plutonium and concentrated fission products in its metal or its mineral waste forms. In the latter case, separation of transuranium actinides from the waste stream is not practiced. Rather, plutonium is fed along with the spent nuclear fuel, and all the actinides, save uranium, are allowed to enter the mineral waste stream. The mineral waste contains essentially all the long-lived, highly radioactive fission products, but the ^{137}Cs content of the resulting disposal form is easily increased by addition of existing ^{137}CsCl to the pyroprocessing electrolyte. Plutonium is also easily added to the highly stable, strong, and corrosion resistant metal waste form to make a Fe-Zr-Pu alloy that should resist corrosion for well beyond ten half-lives of the ^{137}Cs that would be added to provide radiological protection to the resulting metal disposal form. In this case, the surplus material would be added directly to the melt of noble metal fission products, cladding hulls, and makeup metal that is cast to constitute the metal waste form. Radiological protection is provided by mechanically incorporating stable Cs compounds into the disposal form. The presentation includes a description of the pyroprocess for spent fuel, process modification for disposal of surplus materials, and initial experimental data on the disposal material.

Session: Disposition Technologies

Dry-Blending Processes Applicable to the Denaturing and Disposition of Uranium Isotope Mixtures Incorporating ^{233}U

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Approximately 1.76 metric tons of mixed uranium isotopes including 755 Kg of ^{233}U and 861 Kg of ^{235}U are being stored by the Department of Energy (DOE). This material is fissile and could be used in nuclear weapons. The DOE has determined that this material is excess to DOE needs and is examining methods to blend the material with ^{238}U to produce a material that is non-weapons usable and disposable. One proposed blending approach is to mix the isotopes in nitric acid solution and then to precipitate the uranium out of the solution, producing a blended oxide. However, this approach requires several unit operations and can result in the generation of liquid wastes. Several non-aqueous alternatives for achieving the desired isotopic blend have been proposed and will be presented. These blending alternatives include: dry powder press and sinter, oxide melts, metal melts, and fluoride melts. Oxide melt blending and dry powder blending followed by pressing and sintering have emerged as the most promising of the four. Process descriptions and system configurations will be presented for both of these options.

Full Scale Testing of Extraction Chromatography for Actinide Decontamination of Concentrated Hydrochloric Acid Waste Streams

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Extraction chromatography has been tested at full scale as a method to lower actinide activity levels in high concentration (5-8 M) hydrochloric acid (HCl) effluent streams. Several resins from EIChrom Industries, consisting of amberchrom and silica supports coated with various amounts of n-octyl(phenyl)-N,N-diisobutylcarbamoylmethyl-phosphine oxide (CMPO), tributyl phosphate (TBP) or diamyl amylphosphonate (DAAP), were obtained in quantity and tested. TRU-Spec resin (13% CMPO, 27% TBP on amberchrom) proved very successful for recovery of Pu(IV), while other resins with greater CMPO content (30% CMPO, 10% DAAP) on silica or amberchrom respectively, were more suitable for Pu(III) and Am(III) recovery. The columns performed admirably for removal of gram quantities of actinides under carefully regulated conditions with up to 99.99% alpha decontamination. Problems which degraded actinide decontamination efficiency of some large scale runs included plugging problems (particularly with the silica support) and channeling with some columns and conditions. The usefulness of these resins for actinide removal from HCl effluent streams will be discussed.

Session: Aqueous Separation Technologies

The Effects of Anion Exchange Functional-Group Variations on the Sorption of Pu(IV) from Nitric Acid

S. Fredric Marsh
Sandia National Laboratories

A macroporous, polyvinylpyridine anion exchange resin has been used for more than five years at the Los Alamos Plutonium Facility to recover plutonium from nitrate media. This strong-base anion exchanger, Reillex™ HPQ, offers higher capacity, faster kinetics, and significantly higher resistance to chemical and radiation damage than conventional polystyrene-based resins. In this study, we measure the sorption of Pu(IV) on Reillex™ HPQ and on three macroporous, strong-base anion exchange resins that differ from Reillex™ HPQ only in the alkyl group used to quaternize the pyridinium nitrogen. These four resins, all prepared by Reilly Industries, Inc., are copolymers of 1-alkyl-4-vinylpyridine, where the alkyl groups are methyl, butyl, hexyl, and octyl. We compare the trends in Pu(IV) sorption on these four resins to those obtained in our earlier study of four polystyrene/divinylbenzene anion exchange resins having trimethyl, triethyl, tripropyl, and tributyl ammonium functionality. We measured Pu(IV) sorption from 1 M to 9 M nitric acid in both studies.

Sorption of Americium from Hanford Tank 101-SY Simulant Solution

Zita V. Svitra and Scott M. Bowen
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S. Fredric Marsh
Sandia National Laboratories

We evaluated the sorption of americium and other key elements onto 32 selected absorbers from four variations of Hanford Tank 101-SY simulant containing organics. We chose absorbers for their ability to sorb cesium, strontium, technetium, or actinide elements based on our earlier studies using organic-free simulants. The simulant solution variations for this work were (1) as-prepared 101-SY simulant, (2) 101-SY simulant gamma-irradiated to~35 Mrads, (3) 101-SY simulant after a hydrothermal destruction treatment, and (4) 101-SY simulant after both irradiation and hydrothermal treatment. As expected, the differing organic composition of each simulant variant greatly affected americium and strontium sorption, but not cesium or technetium. We will present experimental results for americium from each of the four simulant variants.

Sorption of Plutonium and Americium from a Generic Hanford Complexant Concentrate (CC) Simulant Solution

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Sandia National Laboratories

Zita V. Svitra
Scott M. Bowen
Los Alamos National Laboratory

As part of a Los Alamos/Sandia absorber screening program, we are evaluating the sorption of plutonium and americium onto 33 selected absorbers from four variations of a generic simulant for Hanford complexant concentrate (CC) tank waste. The simulant variations are (1) as-prepared CC simulant, (2) CC simulant after exposure to ~35 Mrads of gamma radiation, (3) the first solution after a hydrothermal organic-destruction treatment, and (4) the second solution after hydrothermal treatment. The selected absorbers were chosen for their ability to sorb cerium, strontium, technetium, or actinide elements from organic-free simulants in our earlier studies. As expected, sorption behavior is highly dependent on the organic composition of the specific simulant solution. Our experimental results, including the identity of the absorbers that most effectively sorb plutonium and americium from each of the four solutions, will be presented.

Reactivity of Tributyl Phosphate Degradation Products with Nitric Acid: Relevance to the Tomsk-7 Accident

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The reaction of organic solvents with nitric acid or aqueous uranyl nitrate is thought to have caused the chemical explosion at the Tomsk-7 reprocessing plant at Tomsk, Russia in 1993. This accident has prompted further studies of reactions of tributyl phosphate (TBP) solvents with nitric acid and other oxidizers used in nuclear processing plants in the United States. The major organic degradation products of TBP in contact with nitric acid are butyl nitrate and butyl alcohol. These compounds can then react further with nitric acid to produce carbon dioxide, carboxylic acids and significant amounts of heat. The reaction of butyl nitrate and butyl alcohol with nitric acid were studied at several temperatures and the reaction products were identified. Propionic acid, (C_2H_5COOH), butyl alcohol (C_4H_9OH), and butyraldehyde (C_4H_8O) were identified as the main reaction products of butyl nitrate and nitric acid. Carbon dioxide and nitric oxide were the major gaseous reaction products. Butyl alcohol is somewhat more reactive with nitric acid and produces mainly carbon dioxide and nitric oxide. These redox reactions can occur at quite low temperatures (about 50°C) when reacting the compounds nitric acid.

Studies of Solvent Extraction of Actinides in TTA + Diamide Systems

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Diamides have been proposed as separating agents in solvent extraction of actinides in a process which would compete with TRUEX. We have studied the extraction of Am(III), Th(IV), $Np(V)O_2^+$ and $U(VI)O_2^{2+}$ by N,N-dimethyl-N-N'-dihexyl-3-oxapentanediamide (DMDHOPDA) and the thio analog of this diamide. The extraction by the diamides from perchlorate solutions with weak synergism was observed for TTA+DMDHOPDA solutions. The stoichiometric composition of the extracted species has been determined as a function of pH and of TTA and DMDHOPDA concentrations. The speciation indicated was $Th(TTA)_2(X)_2(A)$, $Th(TTA)(X)_3(A)$, $Th(TTA)_3(X)$, $UO_2(TTA)_2(A)$ and $UO_2(TTA)_2(A)_2$. For the Am(III)/Eu(III) pair, the separation factors were found to vary with speciation as follows:

Species	Separation Factor
$M(TTA)_3(A)$	1.0
$M(TTA)_2(A_2)(X)$	1.7
$M(TTA)(A_3)(X_2)$	10.0

Where: $X = ClO_4^-$ and A = DMDHOPDA

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Session: Aqueous Separation Technologies

Zirconium Chemistry in the TRUEX Process Solvent

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Lockheed-Martin Idaho Technologies

Actinide partitioning using the TRUEX process is currently under investigation at the Idaho Chemical Processing Plant (ICPP). Typical ICPP waste solutions contain between 0.001 M to 0.5 M Zr. The TRUEX process solvent has been shown to significantly extract zirconium from both simulated and actual ICPP wastes. Several Zr complexing reagents have been tested to reduce the extraction of Zr into the TRUEX solvent. These complexing reagents have been unsuccessful in preventing the extraction of Zr from ICPP wastes because only small amounts can be added in order to avoid the formation of unwanted precipitates. The extraction of Zr from these ICPP wastes dramatically impacts potential TRUEX flowsheets by: 1) potentially loading the solvent to the extent of suppressing actinide extraction, 2) introducing undesirable chemicals during solvent scrubbing to remove Zr, and 3) causing precipitations to occur during stripping if not efficiently scrubbed. Even though successful flowsheets have been developed that effectively scrub Zr from the TRUEX solvent, an understanding of the chemistry between Zr and the TRUEX solvent is required to develop alternative solutions for preventing Zr from following the actinides.

A study was undertaken to determine the fundamental chemical behavior between zirconium and CMPO (the active extractant in the TRUEX solvent). The nitric acid and CMPO concentration dependencies of Zr extraction have been determined for HNO_3 systems. The reaction stoichiometry and equilibrium constant were experimentally measure based on classical slope analysis techniques for the following reaction:

$$Zr^{4+}_{aq} + 4NO_{3aq} + xCMPO_{org} \leftrightarrow Zr\,(NO_3)_4(CMPO)_{x\ org}$$

It was determined that approximately 2 moles of CMPO react per mole of Zr to form the complex. Furthermore, the equilibrium constant was determined to be $K_{eq} \geq 128$ for the above reaction at 25°C. The experimental and slope analysis methods are complicated due to the extraction of nitric acid by the CMPO and by the speciation of Zr. Therefore, extensive testing was performed to ensure Zr^{4+} was the extractable species, and the extraction of nitric acid by CMPO was considered when determining K_{eq}.

TRUEX Flowsheet Development as Applied to Idaho Chemical Processing Plant Liquid Acidic High-Activity Waste Using Centrifugal Contactors

J. D. Law and R. S. Herbst
Lockheed-Martin Idaho Technologies

Previous batch contact experiments with Idaho Chemical Processing Plant (ICPP) liquid acidic high-activity waste (HAW) simulant and samples of radioactive HAW suggested a potential flowsheet for partitioning actinides using the TRUEX process. The suggested baseline flowsheet includes: an extraction section to remove actinides from liquid sodium bearing waste (SBW) into the TRUEX solvent (0.2 M CMPO, 1.4 M TBP in Isopar-L); a dilute nitric acid scrub (0.07-0.2 M HNO_3) to back extract co-extracted matrix materials (primarily Fe, Zr, and HN03) from the loaded solvent; thermally unstable complexants (TUCS) to back extract actinides and a carbonate wash section for solvent cleanup. The purpose of the flowsheet development studies was to test and develop the baseline TRUEX flowsheet for ICPP HAW under continuous, countercurrent conditions using centrifugal contactors.

Two flowsheets were tested in four experiments using the Centrifugal Contactor Mockup which consists of sixteen stages of 5.5 cm diameter centrifugal contactors. All testing was performed using non-radioactive HAW simulant. Potential flowsheets were evaluated with regards to the behavior of the non-radioactive components known to be extracted by the TRUEX solvent. Specifically, the behavior of the matrix components including iron, mercury, and zirconium was studied. In addition neodymium was added to the HAW simulant as a surrogate for americium. In general, the behavior of the individual components closely paralleled that anticipated from batch testing. The results indicate that eight extraction stages are more than sufficient to reduce the actinide content in the SBW to levels well below the NRC Class A LLW criteria of 10 nCi/g. Iron was effectively scrubbed from the organic and 5% of the total iron reported to the strip product. Zirconium scrubbing was not as effective and as much as 60% of the zirconium in the feed could report to the strip product. The TUCS strip was effective at quantitatively stripping all metals except mercury from the TRUEX solvent. Carbonate washing effectively back extracted mercury from the stripped solvent, resulting in 99.4% of the mercury selectively partitioned from the HAW.

Session: Aqueous Separation Technologies

TRUEX Processing of Idaho Sodium-Bearing Wastes: Treatment of the Extraction Behaviors of Mercury and Zirconium in the Generic TRUEX Model

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Argonne National Laboratory

The extraction behavior of mercury and zirconium in the TRUEX solvent is of interest for the processing of Idaho sodium-bearing and dissolved-calcine wastes. The TRUEX process is a solvent extraction process developed to extract transuranic elements from acidic nitrate media. The Generic TRUEX Model (GTM) has been developed as a tool for (1) designing flowsheets for treating the wide variability of high-level and transuranic waste streams at DOE sites and (2) guiding experimental programs to test the applicability of the TRUEX process for those wastes. Much of the development of the GTM has been directed to treating Hanford wastes; therefore, the extraction behavior of mercury has not been included previously in this model, and zirconium extraction has been given only limited attention. For the same reason, the effects of chloride ion on extraction behaviors have also been ignored. These components are extremely important in designing flowsheets for treating wastes stored at the Idaho National Engineering Laboratory and are being added to the GTM.

To this end, batch distribution ratio measurements are being performed to determine the extraction mechanisms for Zr(IV) and Hg(II) in the TRUEX process. The thermodynamic and mechanistically correct models derived from these data will allow the GTM to predict the aqueous-phase solution chemistry and extraction behavior of these species. Enhancement will also include expansion of the current speciation models to include Hg(II) and chloride ions using literature complexation-stability and acid-dissociation equilibrium constants. (Including chloride ion speciation will affect the extraction behavior, to varying degrees, of all metal ions.) In this paper we will discuss our results and how they affect prediction by the GTM.

Session: Aqueous Separation Technologies

A Comparison of TRUEX and CMP Solvent Extraction Process for Actinide Removal from ICPP Waste

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Lockheed-Martin Idaho Technologies

Two solvent extraction actinide separation processes are currently being evaluated for treatment of Idaho Chemical Processing Plant (ICPP) waste streams. These processes are TRUEX (0.2 M CMPO, 1.4 M TBP in Osopar L®) and DHDECMP (0.5 M CMP, 1.;0 M TBP in Isopar L®). The primary waste streams for which the applicability of these processes are being evaluated are: acidic sodium-bearing waste (SBW) and acidic solutions of dissolved calcines. The objectives of this comparison include developing pertinent flowsheets for both extraction systems amenable to both ICPP waste streams. Initial studies focused on development of baseline flowsheets through laboratory scale batch contacts with waste simulants spiked with radioactive tracers. Distribution coefficients were measured in the batch contacts to: 1) evaluate the extracted species, 2) determine the necessity, appropriate reagents, and composition of scrub solutions; 3) evaluate behavior, composition, and effectiveness of various stripping reagents; and 4) develop flowsheets free of physical problems, i.e., precipitates or third phase formation. A similarity was maintained between the baseline flowsheets to meet these objectives and make a valid comparison; however, adjustments were incorporated to allow representation of each flowsheet in its best light.

With the exception of the TRUEX-calcine flowsheet, the extraction contacts do not require adjustments to the aqueous feed. Feed adjustment may be necessary with the TRUEX-calcine flowsheet to mask Zr extraction, reduce solvent loading and increase the extraction distributions of the actinides. A need for scrubbing in the CMP-SBW flowsheet was not identified. Scrubbing of Zr is accomplished with 0.05 M $H_2C_2O_4$ in 3 M HNO_3 in the CMP-calcine system. A solution of 0.01 M NH_4F in 1 M HNO_3 is used to selectively scrub Zr in both TRUEX flowsheets. Each baseline flowsheet use 0.04M HEDPA in 0.01 M HNO_3 for gross actinide stripping. The recommended flowsheets will be tested in the centrifugal contactor mockup at the ICPP to evaluate process efficiency and hydraulic performance under conditions of continuous, counter-current flow.

The batch contact data confirms TRUEX is the stronger extractant for actinide removal. This should make stripping from the TRUEX solvent more difficult than for the CMP system. These differences in back-extraction are negligible when HEDPA is used as the strip reagent. The TRUEX solvent extracts both Zr and Hg quantitatively; however these components can also be selectively removed from the loaded solvent and a very "clean" actinide fraction is obtained. The CMP solvent very effectively extracts Hg. Zirconium is extracted to a much lesser extent with CMP than with TRUEX. The Zr can be scrubbed from the loaded CMP solvent, but Hg cannot be easily or completely isolated with the CMP system. Although further testing and development is required, a "best" extraction process cannot be recommended at this time. It is possible that the final decision will be based on the behavior of matrix components, such as Zr and Hg, and their impact on downstream processing in the final evaluation.

Session: Aqueous Separation Technologies

Recovery and Separation of Transplutonium Actinides from Irradiated Targets

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The Radiochemical Engineering Development Center at Oak Ridge National Laboratory processes irradiated curium targets to recover and separate the transplutonium actinides. A series of chemical processing steps is required to obtain these elements in purified product fractions. The targets are dissolved in a two-step dissolution process using caustic to remove the aluminum matrix and caustic-soluble fission products, followed by an acid dissolution of the actinides and remaining fission products. The actinide-bearing solution is then processed in a batch solvent extraction process using di(2-ethylhexyl) phosphoric acid as the extractant for impurity removal. LiCl-based anion exchange on Dowex 1-X8 resin is used for fission product-actinide and curium-transcurium separations. Cation-exchange chromatography on Dowex 50W-X8 resin using ammonium α-hydroxyisobutyrate solution as the eluate is used to separate the transcurium actinides into separate product fractions. The individual product fractions are then further purified using solvent extraction and cation-exchange chromatography, and finally, concentrated and prepared for shipment. In the most recent processing campaign (1994), 12 irradiated targets were processed to recover the curium, californium, einsteinium, berkelium, and fermium. The curium fraction will be recycled back into fabricated targets for further irradiation and transcurium element production. The transcurium product fractions were distributed to both research and industrial users. An overview of the processing steps will be presented highlighting the materials recovered and the areas where processing improvements have been made.

Session: Aqueous Separation Technologies

A New Extraction Chromatographic Material for Actinide Cleanup Procedures*

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Argonne National Laboratory

The recently developed chelating ion exchange resin Diphonix™ exhibits an extraordinarily strong affinity for actinides, especially in the tetra- and hexavalent oxidation states. Because of its remarkable actinides sorption ability, Diphonix has found application in TRU and mixed waste treatment and characterization and in analytical procedures. The use of Diphonix in the above procedures, however, suffers from two drawbacks, namely, the substantial interference on Am(III) uptake by other trivalent cations present in the sample matrix and the complicated actinide stripping procedures. The actinides are so strongly retained by the resin that the only effective stripping agents are compounds belonging to the family of aqueous soluble diphosphonic acids, which contain the same ligand group as the resin. After stripping, the aqueous diphosphonic acid has to be thermally degraded for further processing of the actinides, leading to solutions containing high concentrations of phosphoric acid, which, in turn, can generate problems in the successive separation steps. An alternative procedure is to destroy through wet oxidation the whole resin bed used to sorb the actinides. The wet oxidation of several grams of resin is feasible but is time and reagent consuming, because the resin's polymeric backbone must also be destroyed.

The above problems can be eliminated or at least minimized by replacing Diphonix with a newly developed extraction chromatographic resin containing as the active component a new extractant with an even higher affinity for actinides. Both the interference by other cations and the complexity of the stripping procedure are strongly reduced by the new resin. In this communication the properties and characteristics of the new resin are discussed in relation to its application for preconcentration and separation of actinides from a variety of biological and environmental samples.

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Session: Poster

An Overview of the Los Alamos Efforts in Tank History Characterization and Estimation of Chemical/ Radionuclide Inventories for the Hanford Waste Tanks

Stephen F. Agnew, Kenn Jurgensen, Toma Duran, Rob Corbin,
Bonnie Young and John R. FitzPatrick
Los Alamos National Laboratory

Over the past two years there has been an on-going effort at Los Alamos, supported by DOE-RL and Westinghouse Hanford Company to use historical tank fill information to derive a set of inventory estimates for the 149 single-shell and 28 double-shell tanks at the Hanford site. As of April 1995, several data sets have been compiled using Excel 4.0/5.0. These data sets are discussed below.

1. Waste Status and Transaction Records, or WSTRS, are records of waste transactions and level histories for each tank. The solids accumulations are assigned to a particular Defined Waste and are used for tanks where such information is missing. Some of the waste transactions have been validated either by LANL or by Ogden Environmental.

2. Tank Layer Model or TLM, sludge accumulation model, is a volumetric and chronological description of the inventory of each tank. Each solid layer is assigned to a particular transaction (waste addition) or process campaign and is described in the Hanford Defined Waste Report.[1] The TLM analysis therefore gives a description of each tank's solids in terms of sludge layers, salt cake and salt slurry.

3. Supernatant Mixing Model or SMM, is an "ideal" mixing model, describing the composition of each supernatant in the tank. This model describes the supernatant in terms of fractions of each Defined Waste supernatant with corresponding volume reduction due to active evaporation. No Supernate Mixing Model (SMM) was done for the NE, NW, SW quadrants because there is not much liquid in these tanks as compared to the double-shell tanks.

4. Hanford Defined Wastes provides chemical and radiochemical definitions for each Defined Waste Type. Defined wastes provide a basis for estimation of the chemical and radionuclide inventories in each tank.

A discussion of each dataset listed above with examples will be presented along with the progress to date.

Session: Poster

Design of a Furnace-Based Tritium Decontamination System for Plutonium

W.H. Atkins, J.M. Berg, M.S. Blau, D.E. Christiansen
W.R. Dworzak, C.E. Klatt, T.O. Nelson, H.J. Newman
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A system for removing and recovering tritium from plutonium metal has been designed and fabricated. This design is a substantial upgrade to a previous tritium-recovery system and incorporates several significant improvements. The new system will begin operation in the plutonium facility at Los Alamos National Laboratory in the summer of 1995. New design features include the primary process line, where tritium is driven from plutonium metal in a furnace and captured on metal getter beds. This process is secondarily contained within a set of gloveboxes. An effluent treatment system removes residual tritium from the process line and the glovebox atmospheres by oxidizing the tritium and capturing tritiated water in molecular sieve beds. All components are monitored and controlled by a modern process control system. This system provides complete automation of the effluent treatment system and contributes to safer and more efficient operation. The resulting system is flexible and robust. Process efficiency is improved by better control and monitoring of process parameters. Worker safety is improved by secondary containment, reduced handling, and automatic control of key functions. Stack emissions are minimized by the effluent treatment system. System up-time is improved by the use of reliable components, and in some cases, redundant components, allowing continued operation during certain failure modes.

Session: Poster

Design of an Alpha Spectrometer to Determine Pu/Am Fixed Contamination on Uranium

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As part of weapons decommissioning, plutonium (Pu) and americium (Am) contamination is removed from oralloy hemishells before they are sent to Oak Ridge for disposition. Prior to shipment to Oak Ridge, it is necessary to verify the part is free of Pu and Am contamination after cleaning. Presently, this verification is performed by manually swiping the part and detecting contaminating elements via alpha spectroscopy. Although this method works well for determining the swipable contamination, it does not reveal the fixed contamination on the oralloy surface. Thus, there is open speculation on the cleanliness of the part. We have designed an alpha based spectrometer that will determine the fixed contamination on the oralloy surface as low as 500 disintegrations/minute (dpm) levels.

An apparatus is being constructed that will automatically survey the entire part, inside and outside, by rotating the part past an array of surface barrier detectors. By counting all alpha events above the energy associated with the decay of $^{234,235}U$ (4.75 MeV), an accurate measure of Pu and Am contamination can be determined. Part rotation, data acquisition, and data analysis are all performed under computer control.

The use of this instrument enables a more thorough characterization of the oralloy surface than typical swiping procedures. Additionally, the use of this instrument is more amenable to quality control than the swipable technique because human intervention is not required. Moreover, this apparatus provides a standardized means of performing this measurement that can be utilized by all laboratories handling these parts.

This paper describes the mechanical design, the instrumentation and controls, and preliminary results.

Session: Poster

Radionuclide Transport and Colloid Formation During Sludge Washing

W.D. Bond, G.D. DelCul, G.D. Davis
and L.M. Toth
Oak Ridge National Laboratory

As a result of activities related to fuel reprocessing and plutonium recover at Hanford, a large volume of sludges and liquid wastes were generated over several decades of operations.

One of the possible ways to minimize the volume of the stored transuranic (TRU) waste is the washing of the bulk sludges. The aim of the washing is to minimize the amount of TRU wastes by displacing the interstitial aqueous salt solution from the sludge and by dissolution of non-TRU components. During the washing, it is imperative to avoid the transport of transuranic elements into the washing solutions.

Colloids, if formed, could contaminate supernates with TRU elements since colloids are often not easily filtered nor easily coagulated. Colloidal dispersions of the bulk sludge materials might carry Pu and Am as their hydrous oxides by sorbing on or aggregating with the major hydrous oxide (Al, Cr, Zr, Bi, etc.) constituents of the sludge. Determination of factors which promote stability of sludge colloids is necessary so that these conditions can be avoided during the processing of the tank sludges.

A variety of experimental techniques were employed to determine if certain conditions in washing produce colloids. These methods included turbidity, light scattering, streaming current potential, particle size measurements, and settling rates. Solution characteristics necessary for colloidal formation such as pH, conductivity, electrolyte concentration and type were also determined. Special emphasis was devoted to the characterization of the hydrous, gelatinous aluminas precipitated at different pH values and different salt concentrations.

Session: Poster

Criticality Safety when using Neutronic Poisons with Plutonium

Ralph H. Condit and Joseph S. Pettibone
Lawrence Livermore National Laboratory

This note introduces a criticality safety issue having to do with a special case where gadolinium may be used as a neutronic poison in the processing of solutions of highly enriched uranium or plutonium. It applies to a moderated system where the neutrons are degraded to thermal energies and where gadolinium is used as a neutronic poison to control criticality. The reactivity in such a fissile system would increase rapidly with temperature due to the fact that the capture cross-section of gadolinium decreases more rapidly with temperature than does the fission cross-section of the uranium or plutonium. This is a well known feature of gadolinium and has always been taken into account when criticality safety calculations are undertaken. The present contribution is to carry out a computer calculation using codes for estimating energy releases in the event that a criticality condition is reached. It indicates that the release could be larger than normally expected for criticality accidents, in the range of 10^{21} fission, depending on the system. This is because the energy release can increase more rapidly with temperature in such a system than the decrease in reactivity due to its disassembly with time. Further study may be desirable to determine the relevance of this calculation to present and planned fissile material handling.

Session: Poster

Actinide Neutron Source Recovery

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Cecil Brown, Antonio Guillen and Brenda Griego
Los Alamos National Laboratory

Neutron sources were produced at a variety of DOE laboratories beginning in the late 50's and were distributed to Universities and private companies for a variety of research tasks, such as instrument calibration, density measurements and small scale reactors. These sources are now being retired and deactivated at LANL. Because neutron sources present an exposure problem (5 Mev neutron), we have modified a glovebox with polycarbonate shielding along with automated control systems and remote handling to perform decladding and dissolution to meet ALARA goals. Our process involves exposing the plutonium beryllium alloy, dissolving the alloy in hydrochloric acid and using standard aqueous processing to separate the plutonium. We presently have several processing options available, including ion exchange, solvent extraction, DCHP and direct oxalate precipitation. We have modified historical flowsheets with emphasis on waste minimization and a new goal of a long term storage form for plutonium disposition. This poster presents relevant data associated with these flowsheet options.

Session: Poster

Actinide Recoverability From Synroc Ceramics

Bartley B. Ebbinghaus
Lawrence Livermore National Laboratory

Norm M. Levitz
Argonne National Laboratory

A Synroc ceramic is one candidate for immobilization of surplus fissile materials, primarily plutonium. One requirement of any immobilized form is that the plutonium or other fissile materials must be at least as difficult to recover from the form as plutonium is from spent nuclear fuel. Thus, the surplus fissile materials will be at least as unattractive as the larger and continuously growing inventory of plutonium in spent nuclear fuel. The proliferation resistance or difficulty of recovery will be a function of the radiation field around the product, the difficulty of removing and retrieving it from the geologic environment, the degree of safeguards and security used to protect it, the isotopic composition, and the chemical processing required to recover the desired actinide from the immobilized product. This last aspect of proliferation resistance will be entirely dependent upon the chemical composition of the immobilized form chosen. For actinides in glass waste forms, a variety of dissolution and aqueous processing methods are already known and are quite adequate to recover fissile actinides from glass to a weapons usable form. For actinides in Synroc ceramics, however, the best actinide recoverability routes are not known. In this study, all reasonable approaches to actinide recoverability from a Synroc ceramic have been considered, and three likely routes to actinide recoverability have been identified. These hypothetical processes are derived primarily from known processing methods used in the titanium industry and partially from research in nuclear fuel reprocessing. Each proposed method will be accompanied with a discussion of the resources and equipment that would be required to perform the chemical processing.

Session: Poster

ALPHA Vitrification Facility

Philippe Guiberteau
CEA France

Objectives: Effluents vitrification in a specific unit with a small capacity

Effluents are contaminated by Plutonium and Americium (about 40 mg/l, no fission product) but contained lot of elements like: Iron, Calcium, Cadmium, Sodium.....

The process is scaled to treat an annual effluents volume of 7 m^3 and the glove box technology will be used. One liter of effluents produce about 360 g of glass.

Process is divided in:

- effluents preparation,
- effluents evaporation and calcination,
- glass mixing in a melting-pot
- casting in a container,
- container treatment.

Glass is cast in a standard container which is decontaminated. Our objective is to produce glass to concentrate the radio-activity of the effluents in a stable matrix to a future geological storage.

The project planning is:

1995: Studies by SGN
1996 - 1997: Equipment and glove box installation
1998: Cold and hot tests

Total cost is about 5 M $.

Session: Poster

Plutonium Immobilization Hot Press Development

James M. Lawson, Steven T. Mills and Bartley B. Ebbinghaus
Lawrence Livermore National Laboratory

A hot press suitable for immobilizing relatively large quantities of plutonium in Synroc ceramic has been designed and built at LLNL. Initial testing and demonstrations are in progress. The design maximizes use of existing glove box systems and equipment, thus simplifying its assembly and minimizing its impact on LLNL's other plutonium processing capabilities. This hot press has been designed to densify and sinter ceramics in stainless steel bellows at temperatures up to 1300°C and 4750 psi loads. In a similar type of hot press, these conditions have been used by the Australian Nuclear Science and Technology Organisation (ANSTO) on large scale, up to 31 kg of Synroc product, to prepare densified and sintered samples in stainless steel bellows using non-radioactive materials. The hot press at LLNL will be used to demonstrate immobilization of up to 100 g of plutonium in up to 500 g of Synroc product. To date plutonium immobilization in Synroc has only been demonstrated on a scale of about 1 to 2g of plutonium in about 10 to 20 g of Synroc product.

Synroc is an extremely durable and leach resistant ceramic composed of a variety of titanate based minerals. Exact minerals that are used in the formulation depend upon the application and the status of Synroc development at the time. For this application, the minerals used are zirconolite ($CaZrTi_2O7$), hollandite ($BaAl_2Ti_6O_{16}$), perovskite ($CaTiO_3$), *and rutile* (TiO_2). To prepare the SYNROC, plutonium is added to the in the form of PuO_2 or $PuO_2(NO_3)_2$ to a slurry of Synroc precursor containing the desired oxides. Gadolinium, either as Gd_2O_3 or $Gd(NO_3)_3 \cdot 6H_2O$, and cesium, either as $CsNO_3$ or CsCl, are also added as a neutron poison and to simulate the radioactive cesium which will be added. The slurry is blended and died to make a homogenous mixture. This mixture is calcined to remove residual moisture and any nitrates, and is then loaded into the stainless steel bellows and hot pressed.

Session: Poster

Declassification of Non-Plutonium Weapons Components

Douglas P. McAvoy and Bartley B. Ebbinghaus
Lawrence Livermore National Laboratory

As nuclear materials are removed from retired weapons components, classified transuranic (TRU) residues are generated. These residues are composed primarily of beryllium metal and stainless steel. While these parts remain classified, they are unacceptable for disposal at the Waste Isolation Pilot Plant (WIPP). As a TRU waste, no other disposal alternative is likely to be available. A viable method to declassify non-plutonium metal parts is to melt and cast them into buttons or ingots. For beryllium, melting is achieved at about 1290°C, and for stainless steel, melting is achieved at about 1600°C. However, considerably lower processing temperatures can be used if the part is dissolved into a molten alloy. For example, a review of know phase equilibria indicates that either a molten copper-titanium or a molten copper-zirconium alloy can be used to dissolve beryllium metal at temperatures as low as 900°C. For stainless steel parts, similar alloys can be used. As an additional consideration, the melting of beryllium alone is undesirable because residual plutonium will react with molten beryllium to form $PuBe_{13}$ which is an intense source of neutrons. With both beryllium and plutonium soluble in molten copper-titanium and copper-zirconium alloys, $PuBe_{13}$ will be less likely to form. Incorporation of beryllium and residual plutonium into a metal composed partially of titanium or zirconium may also be desirable to increase corrosion resistance and provide a better waste form for disposal.

Session: Poster

Metal Conversion and Oxide Stabilization Systems

Steven T. Mills, Bartley B. Ebbinghaus and Carlos Colmenares
Lawrence Livermore National Laboratory

Recovery of plutonium from weapons components by hydriding and oxidation of the hydride to form PuO_2 powder has been demonstrate repeatedly at LLNL and other sites. This experience provides the basis for a pilot scale process to convert plutonium from retired weapons components to oxide powder suitable for mixed oxide fuel (MOX) or immobilization for long term storage.

Several designs for the conversion of plutonium metal to plutonium oxide are currently being investigated at Lawrence Livermore National Laboratory. Proposed systems that have been considered vary in configuration according to the level of automated handling used. Systems range from a glove box with no automation to a fully automated unit with manual backup capability.

The first is a two step process where plutonium is hydrided in a vacuum vessel filled with hydrogen. The plutonium hydride is then transferred into a flow through reactor where it is oxidized to PuO_2. Feasibility of this approach has already been demonstrated.

The second proposed process requires a single reaction vessel. In this process a fixed volume of hydrogen gas is introduced into a vacuum vessel containing a plutonium sample. After the hydrogen is consumed, oxygen is then slowly introduced into the vessel. The resulting oxidation of the plutonium hydride releases hydrogen, which will continues hydriding fresh metal until all of the plutonium is converted to oxide. Earlier work has shown that this cyclic process is able to completely remove and oxidize the plutonium in weapon components.

Session: Poster

Alpha Swipe Spectroscopy for Oralloy Disposition

Arthur N. Morgan (Lonny) and Timothy O. Nelson
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Los Alamos National Laboratory has demonstrated an electrolytic decontamination method for cleaning oralloy (high enriched uranium) surfaces. This method reduces plutonium (Pu) and americium (Am) contamination to less than 20 disintegration's per minute (dpm)/100 cm^2. For determination of the plutonium (Pu) and americium (Am) contamination on the uranium (U) surface using a swipe, alpha spectroscopy is the method of choice. However, procedures between DOE sites have differed. For the procedure used at Rocky Flats Environmental Technology Site (RFETS), the alpha swipe was read directly. For the procedure used at Oak Ridge Y-12, a more elaborate chemical tracer alpha analysis was done, where the swipe was ashed. The difference in procedures for alpha swipe analysis was believed to be a potential root cause for unacceptable parts received from RFETS at Oak Ridge. We have done a direct quantitative comparison of these procedures.

In addition to the quantitative results, other differences in the procedures should be noted. The RFETS procedure is much less work intensive and follows the general guise of counting a room swipe compared to the Oak Ridge procedure. Additionally, the turnaround time for a count result using the RFETS is much shorter than using the Oak Ridge procedure. Moreover, the waste stream produced following the Oak Ridge procedure is a mixed hazardous low level waste; whereas, the RFETS procedure simply produces a low level swipe as waste.

The Oak Ridge procedure follows a more standard method of quantitative alpha spectroscopy. For the RFETS procedure, major concerns in detecting the alpha particles comes from attenuation to lower energies by the swipe itself that may lead to loss of alpha particle readings and tailing. This tailing to lower energies can be severe enough to obscure the peaks of interest. Newer software however enables the deconvolution of peaks to get rid of this effect; therefore, we were able to compare the two procedures based on the complete area for the Pu and Am peaks.

Session: Poster

Magnetic Swing Adsorption: A New Actinide Separation Process

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Robert L. Kochen
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James A. Ritter
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A new magnetic swing adsorption (MSA) process is under development which utilizes a column of magnetic adsorbent surrounded by a magnetic field to effect separation of certain metal species from solution. In this process, an electromagnet is simply energized for adsorption and shut-off for elution. During the adsorption mode, an effluent stream is produced that is depleted of certain metal species, whereas during the elution mode, an effluent stream is produced that is enriched in these same metal species. Both the magnetic field strength and relative flow rates during adsorption and elution control the degree of enrichment of the metal species.

Magnetic adsorbents in various physical forms can be used in this MSA process for the removal of a variety of actinide and heavy metal ions from waste water. A synergistic effect between a magnetic adsorbent material and an external magnetic field is thought to be responsible for the enhanced removal of metals from waste water. Results presented for the removal of plutonium and americium using a magnetic resin clearly demonstrate this synergy. These results also point the way for the development of a variety of new external field-enhanced separation processes.

Session: Poster

Nuclear Applications for Magnetic Separations

David A. Romero, Ann R. Schake, Laura A. Worl, Dennis D. Padilla
Kevin B. Ramsey, Richard D. Maestas, Robert C. Martinez, Yvette E. Valdez
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Thomas L. Tolt
Lockheed Environmental Systems & Technologies Co.

Magnetic separation is a physical separation process that segregates materials in a mixture on the basis of magnetic susceptibility. The technology relies only on physical properties, and therefore separations can be achieved while producing little or no secondary waste. All actinides and their compounds are paramagnetic (slightly magnetic) and, in a non-uniform magnetic field, move in the direction in which the field intensity increases. Most host materials, such as water, graphite, MgO/sand, and soil, are diamagnetic (non-magnetic) and move in the opposite direction. This serves as the basis for separation such that when the field gradient is of sufficiently high intensity, paramagnetic particles are physically captured and separated from the non-magnetic host material. The paramagnetic contaminants are concentrated into a much smaller volume, reducing downstream processing needs and resulting in significant cost savings.

Two magnetic separation methods are being developed: magnetic roll (or drumtype) separation and high-gradient magnetic separation (HGMS). The magnetic roll separation method is used to separate magnetic particles from dry powders. This method effectively extracts particle sizes ranging from 90 to 850 microns. A variety of hot tests have been conducted on a lab-scale roll separator. Graphite powder, bomb reduction sand (MgO), and sand, slag, and crucible (SS&C) residues have been processed with varying results. HGMS is used to separate magnetic components from solids, liquids, or gases and involves passing a slurry through a magnetized volume. The HGMS method effectively extracts particle sizes ranging from about 0.3 to 90 microns and, thus, is complementary to the roll separator. We are currently developing high gradient magnetic separation (HGMS) for applications to soil decontamination, liquid waste treatment, underground storage tank waste treatment and chemical processing residue concentration.

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Conference Program and Abstracts

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