

66th Radiobioassay and Radiochemical Measurements Conference (RRMC 2023) Abstracts

08:10	Julia Ignacek	Keynote: Technology Transfer: Unraveling the Complexities
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Monday, October 30, 2023

09:20	Masako Ohno	Application of Rapid In-Vitro Bioassay for Determining Internal Contamination of Radiation Workers - To Prepare for Decommissioning in Fukushima Daiichi NPPS
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APPLICATION OF RAPID IN-VITRO BIOASSAY FOR DETERMINING INTERNAL
CONTAMINATION OF RADIATION WORKERS

- TO PREPARE FOR DECOMMISSIONING IN FUKUSHIMA DAIICHI NPPS -

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On March 11, 2011, a tremendous earthquake of a 9.0 magnitude occurred undersea off the northeastern coast, triggering a massive tsunami. Flooding from the tsunami caused the loss of core cooling functions, which led to the evaporation of water in the reactor pressure vessels of Units 1-3 at the Fukushima Daiichi Nuclear Power Station (FDNPS), resulting in an accident that damaged the reactor cores. After the accident, the decommissioning work is being carried out step by step, and it will now be entering the stage of taking on challenges in uncharted territory, on the way to full-scale decommissioning including retrieval of fuel debris.

Radiation workers in decommissioning project who enters reactor buildings should conduct various kinds of tasks in working environment with relatively high concentration of radionuclides such as Strontium-90, Actinides, etc., is constantly present. Even if the workers wear appropriate protective equipment, intakes of radionuclides under unforeseen circumstances may result in internal exposure.

Measurement of radioactive materials taken into the body in the event of an incident can be conducted by in-vivo bioassay using WBC and lung monitors, and in-vitro bioassay using analysis of feces and urine. In FDNPS, all workers who enter the controlled area are subjected to routine individual monitoring of internal exposure by WBC (for screening purposes). For alpha-emitting radionuclides such as actinides and beta-emitting radionuclides such as Strontium-90, in-vitro bioassay measurements are generally used. In case of an internal exposure event, it is necessary to establish a rapid bioassay for judgment of medical treatment and for information collections to develop a plan of action.

This presentation will provide an overview of the decommissioning work at FDNPS and application of a rapid bioassay method for mixed nuclides of Pu, Am, Cm, U, and Sr.

Monday, October 30, 2023

10:00	Allison A. Price	Improvements in Am Separation Chemistry for ^{241}Pu - ^{241}Am Dating of Environmental Samples
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IMPROVEMENTS IN AM SEPARATION CHEMISTRY FOR ^{241}Pu - ^{241}Am DATING OF ENVIRONMENTAL SAMPLES

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Determination of ^{241}Pu and ^{241}Am in environmental samples can provide a powerful chronometer spanning ~100 years from the decay of ^{241}Pu ($t_{1/2} = 14.4$ years) to ^{241}Am ($t_{1/2} = 432.6$ years). However, environmental samples typically have ultra-low levels of ^{241}Am , on average 0.18 pg to 16 pg in surface soil (Boulyga et al., 2003), and accurate measurement requires large sample sizes, systematic chemical purification from complex matrices, and sensitive instrumentation. Our laboratory typically follows a sequential separation and isotope dilution method for the measurement of Pu, Np, and Am from a single aliquot of dissolved environmental material, typically up to 1.5g of soil/sediment matrix (Goldstein et al., 2018). In short, Pu, Np, and Am are purified using diethyl oxalate and ferric hydroxide coprecipitations, then Am is separated from Pu and Np utilizing an anion exchange column. While Np and Pu are further cleaned with a smaller anion column and measured by TIMS, the Am fraction is further purified with anion exchange (MP1) and TEVA resins before measurement by MC-ICP-MS. However, recent measurements of Am process blanks employing this chemistry show apparent ^{241}Am and ^{242}Am process blanks which are roughly 5-10x higher than process blanks from three years earlier, likely indicating a polyatomic interference which was not present previously. We find that the addition of a small (200 ul) TRU resin column following the TEVA column provides sufficient removal of the interferent and allows detection limits of ^{241}Am near 3.5×10^5 atoms (less than a few hundred attograms). We provide data on aliquots of the same process blanks run with and without the TRU clean-up column to demonstrate this improvement, as well as replicate analyses of environmental reference materials IAEA-384: Fangataufa Lagoon sediment, IAEA-410: Bikini Atoll sediment, and IAEA-412: Pacific Ocean sediment.

Monday, October 30, 2023

10:20	Hiromu Kurosaki	Development of Routine Bioassay Procedure for Ra in Urine
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Development of Routine Bioassay Procedure for Radium in Urine

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Nuclear Measurements and Analytical Services at Oak Ridge National Laboratory (ORNL) regularly conducts urine radiobioassay analyses of actinides, fission products, and other analytes utilized by researchers and staff. The purpose of this routine program is to monitor radiological workers for potential occupational internal exposure in accordance with regulatory requirements.

When a new project begins at ORNL involving radionuclide(s) that are not currently part of routine monitoring, the development of new analytical techniques to monitor those radionuclide(s) becomes necessary. ORNL supplies Ac isotopes for use in the radiopharmaceutical field for cancer drug development. In the production process, a ^{226}Ra target is bombarded with neutrons to produce ^{227}Ac . After ^{227}Ac is produced, it must be separated from the ^{226}Ra target materials, packaged, and shipped to customers. Because potential exists for exposure to ^{226}Ra , developing a monitoring program for ^{226}Ra is important.

In this presentation, the development of the ^{226}Ra monitoring procedure will be discussed, including separation methods, tracer selection, and source preparation methods.

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Monday, October 30, 2023

10:40	Bailey Christensen	Neutron Source Classification using Convolutional Neural Network-based Acoustic Analysis of Cavitation Shock Traces from Centrifugally Tensioned Metastable Fluid Detectors
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NEUTRON SOURCE CLASSIFICATION USING CONVOLUTIONAL NEURAL NETWORK-BASED ACOUSTIC ANALYSIS OF CAVITATION SHOCK TRACES FROM CENTRIFUGALLY TENSIONED METASTABLE FLUID DETECTORS

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1. Background

Neutron spectrometry is a critical tool for the detection, measurement, and identification of neutron radiation sources such as fission, accelerator neutron generation, and nuclear particle interactions. Thus, neutron spectrometry is vital in fields ranging from nuclear energy to nuclear medicine to the prevention of nuclear terrorism – one of the top ten 21st-century grand challenges. Conventional neutron spectrometry systems [1] can be time-consuming (often requiring days or weeks), susceptible to contamination from background gamma-beta radiation, expensive (~\$10K-\$200+K), and often require bulky moderators.

1.1. TMFD Technology Overview

Purdue University's Metastable Fluids and Advanced Research Laboratory (MFARL) has developed [2] a real-time neutron-alpha-fission tension metastable fluid detector (TMFD) that has been demonstrated to be 100% gamma-beta blind and operates at high efficiency (80-90%+). TMFDs create sub vacuum, negative pressure (p_{neg}) states in fluids using centrifugal or acoustic force. Fluids in such states experience weakened intermolecular bonds which are broken by deposition of localized energy via particle collisions of high-enough linear energy transfer (LET). By varying its p_{neg} , the detector can tune its sensitivity and discriminate detection capability based on particle energy and type (i.e., to neutrons-alpha-heavy ions & blind to low LET gamma-beta particles). The centrifugally tensioned metastable fluid detector (CTMFD), shown in Fig. 1, controls the p_{neg} state in the central bulb region by varying the rotational frequency. When a neutron of sufficient energy strikes the tensioned fluid, this femto-scale interaction causes explosive cavitation bubbles growing within μs to the mm scale resulting in a recordable event accompanied by an acoustic shock wave as depicted in Fig. 1.

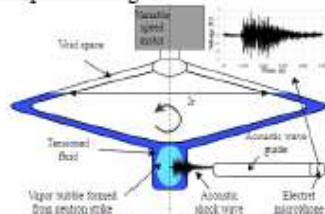


Fig. 1. System Schematic

1.2 Machine Learning and Neural Networks

Machine learning (ML) is a powerful tool that allows a computer to quickly gain "experience" with a

dataset. A neural network (NN) is an ML algorithm that consists of layers of artificial neurons that collectively create a deeply obscure mapping from an input space to a target output space through the use of tunable weights. These weights are optimized to minimize the error between each training sample's output and its targeted label. This layered structure allows the model to seek patterns in incredibly abstract transformations of the data that defy conventional analytic-visual approaches. The convolutional neural network (CNN) is an NN designed specifically to assess data with gridlike topologies, such as images.

2. Experimental Approach

A single CTMFD was used to measure the acoustic shock traces of neutron detections from ²⁵²Cf (fission spectrum) and from a D-T accelerator to ensure distinct neutron energy between sources. All samples collected were assigned their source as a target label. In total, over 32,000 shock trace samples were collected in the form of time-series voltage data describing microphone responses. The time-series data was not found to be source-separable, so spectrograms were generated from the shock traces that depict acoustic power distributed across the frequency and time domains.

3. Results

A CNN was trained on 80% of the dataset of spectrograms to classify the samples as DT or Cf induced. This model was able to classify its training samples with 98% accuracy. The remaining 20% of the dataset was used for blind validation. The trained CNN successfully discriminated the blind validation data, separating the 14 MeV neutron source from the ~0-12 MeV fission energy spectrum source with an accuracy of ~97% as summarized in Fig. 2.

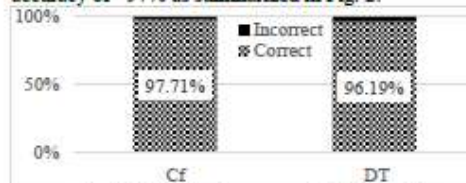


Fig. 2. Model Performance on Validation Data

Acknowledgments

The research work was supported by the USNRC, USDOE, & Purdue University, State of Indiana, USA.

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Monday, October 30, 2023

11:00	Carrie Coralie	Quantification of ^{129}I in Milk at Low-Level for Environmental Monitoring
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QUANTIFICATION OF ^{129}I IN MILK AT LOW LEVEL FOR ENVIRONMENTAL MONITORING

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Iodine is an extremely volatile element which exist in the environment with different oxidation states: -1, 0, +1, +5 and +7¹. These physico-chemical properties complicate its extraction and quantification. Among the 37 iodine isotopes, ^{127}I is the only stable one and ^{129}I is the radioisotope with the longest half-life ($16.1 \times 10^6 \text{ y}$)². ^{129}I is chronically and regulatory released by fuel reprocessing plants *e.g.* La Hague (France) or in case of an accident.

These releases can contaminate the French coastline and the adjacent agricultural land to these discharge zones could be enriched with iodine ^{129}I ³. This radionuclide will thus be present into the food chain and can be ingested by humans. A regular monitoring of ^{129}I concentration in milk is therefore necessary to protect the population and estimate the ingestion dose.

Currently, in our laboratory, the quantification of ^{129}I in milk is carried out by gamma spectrometry 12 times a year. There is a chemical pre-treatment before the measurement, involving a purification on ionic resin and liquid-liquid extraction using 50 mL of liquid carbon tetrachloride (CCl_4).

This analysis has two major drawbacks. First, CCl_4 is a carcinogenic, mutagenic and reprotoxic (CMR) solvent which is no longer marketed in France under the REACH regulation. On the other hand, because of the low efficiency of γ -spectroscopy for ^{129}I determination, the detection limits with this technic are often higher than the environmental background.

In this work, a safe and innovative method to quantify ^{129}I in milk was developed. It is based on an extraction followed by a purification with two different specific resins. The chemical yield determination is based on stable iodine measurement by ICP-MS*. Moreover, the gamma geometry was optimized to improve the limit of detection. Indeed, the use of a well detector has improved detection efficiency and reduced the volume required, enabling a more concentrated sample measurement.

This method has proven its effectiveness, offering a good chemical yield (> 80 %) while making chemical treatment safer and faster.

¹ Kaiho, *Iodine Chemistry and Applications*.

² García-Toraño et al., 'The Half-Life of ^{129}I '.

³ Robens and Aumann, 'Iodine-129 in the Environment of a Nuclear Fuel Reprocessing Plant'.

Monday, October 30, 2023

08:00	Yvotte Brits	Keynote: X-Energy Overview & Development of the Xe-100 Burn Up Measurement System
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09:00	Azza Habibi	Determination of Neptunium-237 in Radioactive Waste with a High Level of Uranium by ICP-MS/MS
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DETERMINATION OF NEPTUNIUM 237 IN RADIOACTIVE WASTE WITH A HIGH LEVEL OF URANIUM BY ICP-MS/MS

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Neptunium 237 is usually quantified in environmental samples and radioactive waste related to some installations of the nuclear cycle (*e.g.*, reprocessing plants).

This long-lived alpha-emitter (2.14×10^6 years) is typically measured by alpha spectrometry [1], [2] after an advanced chemical treatment. The absence of a commercial isotopic tracer for ^{237}Np induces the use of a chemical analogue to calculate its activity by estimating losses during the chemical treatment [1].

Plutonium isotopic tracer is commonly used for the quantification of ^{237}Np since, as a chemical analogue, usually shows a similar behavior during a chemical treatment. When ^{237}Np (α , 4 788.0 keV) is quantified using the plutonium tracer, ^{242}Pu (α , 4 858.2 keV) cannot be used as a tracer due to the energy interference. In this case, ^{236}Pu , often containing the other plutonium isotopes as impurities, is usually used. Subtraction of impurities or double analysis, with and without the tracer, are then necessary. The use of a chemical analogue or a tracer containing impurities generates a bias not only on ^{237}Np activity but also on plutonium isotopes activities.

To optimize the procedure of its quantification and given the favorable electronegativity and half-life of ^{237}Np , ICP-MS is an excellent alternative for the activity determination of this radionuclide [3]. With this technique, the use of ^{242}Pu as a tracer for the quantification of ^{237}Np is possible since the quantification is based on m/z ratio ($m=mass$ and $z=ion\ charge$) [4]. In this case, the isolated plutonium and neptunium are first micro-precipitated or electrodeposited after the chemical treatment to quantify plutonium isotopes by alpha spectrometry. The source containing plutonium and neptunium is then dissolved in a medium suitable for ICP-MS measurement to quantify ^{237}Np .

Furthermore, the development of the ICP-MS/MS technique could give access to the direct analysis (without chemical treatment) of water and even highly contaminated liquid radioactive waste [5].

Indeed, contaminated radioactive waste often contains a high amount of not only ^{238}U but also ^{235}U and ^{236}U . These uranium isotopes generate interferences due to the peak tailing of ^{238}U and to ^{235}U and ^{236}U hydrides ($^{235}\text{U}^2\text{H}$ and $^{236}\text{U}^1\text{H}$). ^{238}U could be eliminated thanks to the double quadrupole of the ICP-MS/MS with a good decontamination factor (tested up to 10^{-12}). To eliminate interferences at m/z 237 and due to uranium isotopes hydrides, the use of gas in the reaction cell is, on the other hand, unavoidable.

In this study, in addition to the determination of ^{237}Np by ICP-MS/MS based on ^{242}Pu tracer and after a chemical treatment, its direct determination in samples containing a significant amount of uranium

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isotopes was investigated. Several reaction and collision gas have been studied with the aim of minimizing uranium hydrides while both maximizing the signal/noise of neptunium and having similar behavior of neptunium and plutonium. The direct measurement prevents the fractionation of plutonium and neptunium generated by a tedious chemical treatment and provides a more accurate quantification. A new and robust method was then developed with excellent figures of merit including excellent decontamination factors and low detection limit.

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- [2] Feng *et al.*, *Nucl Instrum Methods Phys Res* (2009) 609:165-171
- [3] Osvath *et al.*, *J Radioanal Nucl Chem* (2011) 287:459–463
- [4] Huang *et al.*, *Microchem. J.* (2019) 148:597-604
- [5] Huang *et al.*, *Talanta* (2023) 265 :124798

09:20	Frazier Bronson	Tools for Evaluating Groups of Gamma Spectra
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TOOLS FOR EVALUATING GROUPS OF GAMMA SPECTRA

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The new Mirion Data Analyst is a device that is used with spectroscopy detectors to perform continuous repeating quantitative gamma spectral analysis. It is most commonly used on continuously operating industrial radioactivity monitors, for example flowing radioactivity in a pipe, moving detectors on a robot. Consequently, there are a great many spectra generated – hundreds or thousands each month. To serve our users, we have developed two software tools [DA Prospector, ReexaMiner] to simplify analysis of the vast amount of spectra and data generated. This presentation will briefly introduce those tools and focus on how they can be used to gain insight into groups of gamma spectral analyses obtained in a more conventional laboratory setting. These features include: ability to sum groups of spectra together for lower MDA; ability to package groups of spectra into a format allowing viewing nuclide results graphically and exporting results to spreadsheets; ability to extract from groups of spectra a specified single nuclide's activity, FWHM, and Energy.

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10:00	Dustin May	Expanding Radiochemistry Capacity Through Partnerships: Establishing a Radiochemistry Training in Collaboration Between Academia and Public Health
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EXPANDING RADIOCHEMISTRY CAPACITY THROUGH PARTNERSHIPS: ESTABLISHING A RADIOCHEMISTRY TRAINING IN COLLABORATION BETWEEN ACADEMIA AND PUBLIC HEALTH

Presenters: Dustin May, Ph.D.¹ & Royce Riessen, MBA²

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The United States is dangerously close to losing the ability to measure radioactive materials accurately, precisely, and within an effective timeframe to address a radiological disaster and conduct ongoing regulatory testing. This ability requires extensive and unique expertise that has been declining for decades. In 2020, the Association of Public Health Laboratories (APHL) convened an expert radiochemistry workgroup to identify potential radiochemistry workforce solutions. The workgroup wrote a training program proposal to address the gap in training programs for radiochemists. In July 2023, this proposal was funded by APHL through an award from the Centers for Disease Control and Prevention and will establish both an APHL radiochemistry workforce program and a graduate certificate program in radiochemistry. These programs are intended to address critical state and local laboratory radiochemistry workforce needs.

The new APHL radiochemistry workforce program will include an advisory group, a community of practice, and resource development to meet newly-trained and current radiochemist workforce needs nationwide. The advisory group will provide feedback on the graduate certificate program and will steer the development of the community of practice to share national radiochemistry successes, discuss challenges, and develop resources to share solutions. To retain radiochemists in the public health laboratory system, it is critical that newly-trained state and local radiochemists have access to a nationwide network and the resources to ensure they can efficiently conduct their work in a manner that maximizes public health protection.

The radiochemistry graduate certificate program will consist of twelve semester hours of instruction through the Department of Chemistry in the College of Liberal Arts and Sciences at the University of Iowa. Of these twelve semester hours, eight semester hours will be administered online via asynchronous instruction and four semester hours will be hands-on laboratory training. The online courses will cover fundamental concepts in radioanalytical chemistry including: nuclear physics; radiation safety; radiochemical separations; instrumental analysis; data and statistical analysis; and quality assurance. The hands-on laboratory courses will take place during two full weeks at the State Hygienic Laboratory on the University of Iowa campus. These laboratory courses will demonstrate common radiochemical separations across different technologies and the use of radiochemical instrumentation, including gas-flow proportional counters, liquid scintillation counters, gamma spectrophotometers, and alpha scintillation counters. The certificate will be completed in three academic semesters (fall, spring, and summer), with the laboratory training occurring during the summer semester. The hybrid course design allows for students to enroll from anywhere, while still providing the necessary hands-on experience required.

These radiochemistry workforce initiatives will be developed and released over the next year. APHL expects to launch the advisory group and the community of practice by spring 2024 and The University of Iowa expects to enroll its first student cohort for the Fall 2024 semester. APHL, with financial support from CDC, will facilitate tuition, training and travel for public health laboratory personnel enrolled in the program. Overall, these programs are designed to meet the needs of the current and future radiochemistry workforce through a strong partnership between the Association of Public Health Laboratories and the University of

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Iowa. The students trained, credentialed, and supported by these programs will provide a skilled pipeline of new radiochemists to support the field for years to come.

This work is supported by the Centers for Disease Control and Prevention (Grant #NU60OE000104).

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10:40	Dominic Larivière	Comparison of Radium-226 Separation Methods Based on Chromatographic and Extraction Resins for its Determination by ICP-MS in Natural Waters
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COMPARISON OF RADIUM-226 SEPARATION METHODS BASED ON CHROMATOGRAPHIC AND EXTRACTION RESINS FOR ITS DETERMINATION BY ICP-MS IN NATURAL WATERS

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The longer-lived radium isotopes ^{226}Ra ($t_{1/2} = 1600$ y) and ^{228}Ra ($t_{1/2} = 5.75$ y) are ubiquitous in the environment. Because ingested radium can substitute for calcium ions in biological systems, ^{226}Ra and ^{228}Ra radiotoxicity is of concern for health safety. The WHO has defined a limit of 1 Bq L^{-1} for drinking water. At these low levels (ultra-trace), its analysis by inductively coupled plasma mass spectrometry (ICP-MS) requires separation and preconcentration of ^{226}Ra prior to its analysis. Several methodologies have been developed for this purpose, based on resin separations. While their analytical performances have been reported individually, their direct and accurate comparison based only on the literature data is quite impossible due to various sample conditions (matrix, range of ^{226}Ra concentrations) and different analytical instruments or parameters (ICP-MS, ICP-MS/MS, SF-ICP-MS, Apex-Q-ACM) used. Therefore, we selected five methodologies [1-5] based on various resins (AG50W-X8, Analig Ra-01, Sr-Spec), loading (samples volume and pH, flow-rate), rinse and elution (acid/basic) conditions. We applied these methodologies from the same certified waters and analyzed ^{226}Ra concentrations in the final samples by ICP-MS according to the same instrumental parameters. Thereby, we were able to adequately compare them in terms of analytical figures of merit (recovery yield, enrichment factor, method limits, sensitivity, accuracy, turnaround time, cost). This work provides information for selecting the appropriate methodology according to the individual requirements of performance and cost.

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Tuesday, October 31, 2023

11:00	Jonathan Button	Enhancing Emergency Radiobioassay Capacity: Validating the Urine Radionuclide Screen and Refining Sample Analysis Prioritization
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ENHANCING EMERGENCY RADIOBIOASSAY CAPACITY: VALIDATING THE URINE RADIONUCLIDE SCREEN AND REFINING SAMPLE ANALYSIS PRIORITIZATION

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Abstract

We conducted collaborative in-house studies on Cs-137, Ir-192, and I-125 to validate the concept of operations for the Urine Radionuclide Screen (URS). The URS has two layers - a Screening layer and an Identification and Quantification ("ID and Quant") layer. These layers are crucial for prioritizing samples during emergency responses. With the technical ability to effectively triage samples during a large radiation emergency, we can optimize limited capacity for clinical laboratory radiobioassay. Our projects confirmed the accuracy of the Screening layer and helped develop decision algorithms for sample analysis.

Based on three different methods, our measurements demonstrated good accuracy. The Sodium Iodide gross gamma screening and Liquid Scintillation (LSC) gross alpha/beta screening methods correctly identified samples with elevated radioactivity levels. The High Purity Germanium gamma ID and Quant measurements for Cs-137 and Ir-192 exhibited accurate results within 2% and 6% of the respective true values across five activity levels.

To enhance sample analysis prioritization, we compared the performance of the Screening and "ID and Quant" methods for measuring various radionuclides. We effectively addressed timing challenges, such as the gross alpha beta method's lower output and longer startup time. Moreover, we found that the gross gamma result alone is often sufficient for prioritization.

Disclaimer

The findings and conclusions in this study are those of the authors and do not necessarily represent the views of the U.S. Department of Health and Human Services or the Centers for Disease Control and Prevention. Use of trade names and commercial sources is for identification only and does not constitute endorsement by the U.S. Department of Health and Human Services or the Centers for Disease Control and Prevention. The authors declare that they have no competing financial interest.

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08:00	Ralf Sudowe	Keynote: The Radiochemistry Workforce Challenge: How did we end up here and what can we do about it?
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Wednesday, November 1, 2023

09:00	David DiPrete	Ra-226 Analyses on Savannah River Site Radioactive Waste Matrices
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RA-226 ANALYSES ON SAVANNAH RIVER SITE RADIOACTIVE WASTE MATRICES

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Waste cleanup at the Savannah River Site, as well as other DOE nuclear sites, has created an ongoing need to characterize the radiological inventories of various waste tank heels prior to tank closure. Each tank heel has unique chemical and radiological distributions, rendering the use of routine analyses inadequate. Since each tank is a completely new matrix with often-unforeseen interferences, method development must be performed for each tank's residue material. While currently representing only a small fraction of the activity in the various sites' radioactive waste inventories, Ra-226 and its progeny will eventually represent a more significant portion of the inventories due to its relatively long half-life. As a result, detection limit requirements for Ra-226 are often quite low. Ra-226 is an alpha emitter with weak gamma emissions. The required Ra-226 detection limit requirement can be 8 or more orders of magnitude lower than the activities of shorter-lived radioisotopes commonly present in various SRS sample matrices. Therefore, a very efficient separation of Ra-226 from high levels of interfering radionuclides is required. High sample radiological doses require initial radiochemistry to be conducted in SRNL's remote handled shielded cells facility. The separations are then completed in radiological hoods. A series of delayed gamma analyses are conducted for Ra-226 and for the Ra-224 tracer's progeny. Details of the Ra-226 analysis method development and implementation in these high activity matrices will be discussed.

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09:20	Guy Backstrom	If You Can't Beat 'em, Join 'em
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The presenter will discuss how the entire series of Lanthanides from a fresh fission product sample was removed in a few simple separations so that they could be measured using gamma ray spectroscopy. The chemistry of the removal of fresh fission product interferents will also be discussed as well as the work that was done using Mass Spectrometry to show that the Lanthanides are where they were supposed to be in the separation scheme.

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10:00	Henrik Persson	Statistical Review of the Performance of a Large Set of Detector Models Used for Efficiency Calibration in Gamma Spectrometry
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STATISTICAL REVIEW OF THE PERFORMANCE OF A LARGE SET OF DETECTOR MODELS USED FOR EFFICIENCY CALIBRATION IN GAMMA SPECTROMETRY

Henrik Persson

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Kara Phillips and David Sullivan Mirion Technologies (Canberra), Inc.

Developed initially as a solution to create accurate efficiency calibrations for in-situ gamma spectroscopy measurements in the late 1990's, the ISOCS (In-Situ Object Counting System) technology is used today in In-Situ, In-Vivo, Laboratory, and NDA applications. The core of the methodology is a high-quality Monte-Carlo model used for detector efficiency characterization of typically a high-purity germanium detector, which is performed by the detector manufacturer in a rigorous process. The spectroscopist user then combines this characterization with sample-specific geometry definition, which is used by the ISOCS application software and its ray tracing algorithms to generate a full energy peak efficiency calibration for the specific measurement.

Today, Mirion has created over 5,000 Monte-Carlo models and detector characterizations for gamma spectroscopy systems worldwide. In this presentation, we will detail the technical development of the Monte-Carlo model and the ISOCS characterization. The discussion will continue with a description of the stated uncertainties of the characterization. The main focus of this presentation will be a statistical review of comparisons between full energy peak efficiencies from physical calibration source measurements to modeled full energy peak efficiencies of hundreds of recent detector models (from 2016 to 2022).

Wednesday, November 1, 2023

10:20	Dylan Flanagan	Current and Emerging Counting Capabilities within C-NR
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Current and Emerging Counting Capabilities within C-NR

Dylan Flanagan, Matthew Gooden, Lisa Hudston, Dillon Soenke, Jen Romero, Todd Bredeweg, Mike James, Randy Rendon, Mitzi Boswell, Joshua Williams, Elena Guardincerri, Jonathan Zane & Zsuzsana Macisk

Abstract: The counting facility maintained by the Nuclear Chemistry and Spectroscopy Team at the Los Alamos National Laboratory preserves a rich history while continuing to innovate. Dating back to the sandstone test series, the first gas flow proportional beta counters were designed and placed into service in the 1950; continuing advances in gamma spectroscopy helped to drive stepwise improvements. Since 1998, the team has supported the in-vitro bioassay plutonium program. In 2007, liquid scintillation counting of tritium samples supporting this program was also introduced. However, this has presented several challenges in maintaining and upgrading unique systems. A particular focus of late has been expanding and improving our capabilities in forensics and in support of the bioassay program. Current and expanding capabilities will be reviewed while noting the legacy of the historic counting facility. Today, the counting facility operates more than a dozen gas flow beta counters, 50 gamma counters, 200 alpha counters, 4 liquid scintillation counters, and continues to grow.

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10:40	Guy Backstrom	There's Niobium in them thar hills
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A particle from a decommissioning site was analyzed that contained enough Cobalt-60 to hide the Niobium-94 in the Compton Continuum. The presenter will discuss the limitations of “*A-Posteriori*” sample specific Minimal Detectable Activity, the technique used to subsequently remove the Cobalt-60 so that the Niobium-94 and Niobium-93m could be measured and the surprising amount of Nickel-63 that was also found.

Wednesday, November 1, 2023

11:00	Charles M. Folden III	Nuclear Forensic Analysis of Manhattan Project Plutonium
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NUCLEAR FORENSIC ANALYSIS OF MANHATTAN PROJECT PLUTONIUM

Charles M. Folden III

Department of Chemistry and Cyclotron Institute
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Texas A&M University has a longstanding program in nuclear forensics that works to determine the reactor irradiation history of samples of U and Pu. Using a combination of experimental irradiations, simulations, chemical separations, and intra-elemental ratios of specific isotopes, it is possible to determine the type of reactor, the time since irradiation, and the burnup. These procedures have been demonstrated in multiple irradiated samples of U with varying ^{235}U enrichments that have been irradiated in different neutron spectra. One sample was exposed to a varying neutron spectrum in the High Flux Isotope Reactor at Oak Ridge National Laboratory, and this required a detailed study to understand. These techniques were then applied to a legacy sample of Pu from Los Alamos National Laboratory that was shown to have been produced during the Manhattan Project. This talk will discuss these samples, the chemical processing necessary to analyze them, the simulations required, and the details of the Manhattan Project sample.

Wednesday, November 1, 2023

11:20	David DiPrete	Applications of Liquid Scintillation Counting on Savannah River Site High Activity Matrices
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Applications of Liquid Scintillation Counting on Savannah River Site High Activity Matrices

David DiPrete^{1*}, Tad Whiteside¹

¹Savannah River National Laboratory, Aiken, South Carolina, USA

*E-mail: david.diprete@srnl.doe.gov

The Savannah River Site (SRS) is a US Department of Energy nuclear reservation established in the early 1950's as part of the US government's nuclear weapons programs. Currently, SRS is undergoing a process of environmental remediation, emptying waste tanks containing the residues from various neutron activated target processing as well as from the reprocessing of spent nuclear fuel to run the Site reactors. SRS continues to dissolve and dispose of spent nuclear fuel from its past processes as well as that retrieved from various research reactors in the SRS H-Canyon Separation Facility. H-Canyon process solutions, when injected into the waste processing system, is rendered caustic. The waste, when rendered caustic, separates into two phases. The two phases compose of a 6M sodium liquid phase with radiological constituents predominantly made up of Cs-137, and a sludge phase predominantly made up of Sr-90. The bulk of the actinide and lanthanide inventory are also contained in the sludge phase. The caustic phase is decontaminated of Cs-137 using a calixarene-based solvent-solvent extraction facility (Salt Waste Processing Facility). The extracted Cs-137 and the high-level sludge from the waste tanks are vitrified in SRS glass vitrification plant (Defense Waste Processing Facility). The decontaminated solutions are grouted and stored On-Site at the SRS Saltstone Facility.

The Nuclear Measurements Group (NMG) of SRNL is responsible for providing radiological characterizations for these highly radioactive processes, providing analyses for over 100 radioisotopes. Ironically, due to the wide swings in beta versus alpha activities in the various processes, one of the more challenging analyses are simple gross alpha and gross beta analyses. The NMG measures gross alpha and gross beta using a variety of methodologies, including analyses using gas flow proportional counters (GFPC), silicon charged particle spectrometers, and liquid scintillation counters. Liquid scintillation counters offer some advantages over the other methodologies for these higher activity matrices in that the sample aliquots are contained in liquid scintillation vials. LSC vials provide more contamination control, especially when measurement of low levels of alpha activity in the presence of orders of magnitude more beta activity or vice versa is desired. LSC is also less affected by high levels of dissolved solids often found in these waste matrices than are the GFPC or charged particle spectrometers.

The NMG makes heavy use of pulse shape discrimination with liquid scintillation counting to differentiate between alpha and beta activity when making these gross alpha/beta measurements. The laboratory uses both Perkin Elmer Tri-Carb and Hidex scintillation counters equipped with pulse shape discrimination. Performance of the two types of liquid scintillation counters, plus the challenges of using these pulse shape discrimination technologies in a production mode on the wide variety of sample matrices analysed in this laboratory will be discussed.

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08:00	Tad Whiteside	Non-Destructive Assay Systems Deployed at the Savannah River Site
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08:20	Nathalie Wall	Organoclays for the sequestration of technetium and iodine
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Organoclays for the sequestration of technetium and iodine

Nathalie A. Wall¹, Emily Maulden¹, Elizabeth Gager¹, Seaton Ullberg¹, Juan C. Nino¹, Simon Phillpot¹, Carolyn I. Pearce², James E. Szecsody²,

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Clay buffer materials can be used as an engineered barrier for nuclear waste repositories to limit radionuclide transport after the degradation of waste canisters. Bentonite, consisting primarily of crystalline clay minerals belonging to the smectite group, is effective at sequestering cationic fission products owing to its low point of zero charge. However, bentonite requires functionalization to change the surface charge to capture anionic species, such as pertechnetate, iodide, and iodate. We have examined the behavior of organoclays that were functionalized with alkylammonium cations of different sizes and metals. Carbon analyses confirm alkylammonium group inclusion and X-ray diffraction data demonstrate an increased basal spacing for the clays intercalated with the largest alkylammonium moieties. Fourier transform infrared spectroscopy data provide insight into the clay conformation changes upon alkylammonium intercalation. Computational data allow for analyzing the angle between the alkyl chains and the clay plane. Organoclays with less than 15% carbon show negative zeta potential (ζ) values, while clays with more than 15% carbon feature positive ζ . The best pertechnetate, iodide, and iodate retention were obtained with clays functionalized with a multi-branched alkylammonium cations without metal. This work was supported by the U.S. DOE Office of Nuclear Energy's Nuclear Energy University Program, grant DE-NE0008952.

08:40	Beatrice Boulet	Improved analysis of ^{99}Tc in environmental solid matrices in response to an emergency situation.
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IMPROVED ANALYSIS OF ^{99}Tc IN ENVIRONMENTAL SOLID MATRICES IN RESPONSE TO AN EMERGENCY SITUATION.

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Authors: B. Boulet, M. Coupanec, N. Cariou, C. Vivien, E. Cale

The analysis of 99-technetium in solid matrices in the environment can remain a challenge for some laboratories because of its physico-chemical properties and the difficulty of obtaining an isotopic tracer. The challenge is even greater if the analysis is required for less than four days in an emergency situation.

IRSN/LMRE has been measuring ^{99}Tc for several years by liquid scintillation, after dissolving 20 g of dry environmental sample by acid attacks, followed by purification on TEVA resins (Triskem®). This protocol took around 3 weeks to complete, due to the numerous evaporation stages and the need to take great care not to volatilise Tc and the tracer Re. Chemical yields were fluctuated. In addition, in the case of low Re yields, a difference between the results could be observed, which meant that the analysis had to be repeated.

Several studies have been carried out at the lab in recent years to improve the protocol.

Initially, alkaline fusion followed by purification on TK202 and C8 resins was tried. However, new constraints emerged.

Secondly, following an incident that resulted in the possible dispersion of ^{99}Tc in the environment, the laboratory had to quickly adapt its analysis method in order to provide a result in less than a week.

A new analysis strategy was then adopted: reducing the sample size to 1 g; 'simplified' attack of the sample by leaching because deposition had just taken place; measurement by ICP-MS instead of liquid scintillation, with identification and search for all interferences likely to be present at m/z 99 and at the m/z allowing the ^{99}Ru contribution to be estimated. The addition of collision gas was also studied in order to eliminate the interferences identified. The measurements obtained in the blanks were carefully studied and the accuracy of the protocol was validated by analysing reference materials.

The optimised protocol enabled the results of 8 samples to be obtained in 4 days, with chemical yields of over 90%, controlled and corrected interferences and a limit of quantification (LQ) of around 10 Bq.kg⁻¹ dry sample.

Further improvements may be explored in the future, in order to reduce the LQ.

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09:00	Caleb J. Mortensen	Determination of Per technetate and Non-Per technetate Species in Hanford Waste Tank
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DETERMINATION OF PERTECHNETATE AND NON-PERTECHNETATE SPECIES IN HANFORD TANK WASTE

Caleb J. Mortensen and Michael L. Lindberg
Hanford Laboratory Management and Integration, Richland, WA 99354, USA

ABSTRACT

The U.S. Department of Energy's Hanford Site has approximately 56 million gallons of radioactive waste stored in 177 underground storage tanks. It was discovered that some Hanford tank wastes contain technetium in a soluble form inconsistent with the usual Tc(VII) pertechnetate ion or the insoluble Tc(IV). These forms were dubbed "non-pertechnetate." Previous studies were performed on ten Hanford tanks, and four were found to be predominantly in the non-pertechnetate form: 241-SY-101, 241-SY-103, 241-AN-102, and 241-AN-107. The other six tanks showed indications of low concentrations of non-pertechnetate, but were not fully quantified due to varying experimental results.

The determination of non-pertechnetate is relevant in the consideration of using a grout waste form. By distinguishing between the pertechnetate and non-pertechnetate species in Hanford tanks, it can be determined more accurately how effective the addition of a reducing grout will be at immobilizing the tank waste. The grout waste form would rely solely upon the oxidation-reduction chemistry of technetium to determine its effectiveness in immobilizing the various forms of technetium. When pertechnetate-containing waste is mixed with the grout-forming materials, particularly Blast Furnace Slag, the Tc(VII) as soluble pertechnetate is reduced in an oxidation-reduction reaction to insoluble Tc(IV) as TcO₂ or TcS₂. There is a chance, however, that the non-pertechnetate will not behave like the other forms of technetium and thus may not be retained in the solid waste. This has not been tested on direct tank waste, but since non-pertechnetate was found by previous studies to be primarily in the Tc(I) oxidation state stabilized in a carbonyl complex, further reduction would not result in the desired insoluble Tc(IV) form. The Tc(I) would have to first be oxidized to reach the insoluble form, but the Blast Furnace Slag grouting environment is not conducive to such chemistry. This may mean that a grout waste form would be unacceptable because it would not retain the non-pertechnetate form of technetium, and it could leach more quickly than the insoluble Tc(IV) produced from the original pertechnetate.

A method was, therefore, developed at Savannah River National Laboratory for the determination of pertechnetate and non-pertechnetate species in tank waste. This method includes using TEVA[®] resin to extract the pertechnetate, a sulfuric acid and hydrogen peroxide oxidation of the sample, and re-extraction of any newly formed pertechnetate onto TEVA resin. Testing was first performed on Savannah River Site waste, which does not contain non-pertechnetate species. Therefore, testing needed to be performed on Hanford Site tank waste to adapt the method for use at the 222-S Laboratory and to determine the feasibility of implementation in the suite of analyses performed on Hanford tank samples. Three tanks,

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241-AN-107, 241-AW-105, and 241-SY-102, were chosen for these tests due to the expected presence of non-pertechnetate as well as the availability of recent sample material in the 222-S Laboratory sample archive. Initial testing indicated that the amount of tank sample, and in turn the radiation exposure, could be decreased significantly from what was originally used in the Savannah River method while sufficient data could still be obtained. Preliminary data indicated that sufficient separation of the two species was achieved after the sample amount was decreased and that the quantification of the two species is possible using inductively coupled plasma – mass spectrometry.

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09:20	Robert Jones	Integrated Consortium of Laboratory Networks (ICLN), Radiological Laboratory Subgroup (RLS) Rad Lab Hub
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INTEGRATED CONSORTIUM OF LABORATORY NETWORKS (ICLN)

RADIOLOGICAL LABORATORY SUBGROUP (RLS) RAD LAB HUB TECHNICAL ORAL PRESENTATION

Tentative Presenter: John Griggs; griggs.john@epa.gov, ICLN RLS Chairperson

Affiliation: ICLN/EPA NAREL

Additional authors and affiliations: Dr. Andrew Scott (DHS ICLN Program); andrew.scott@hq.dhs.gov

This technical oral presentation will describe in detail a major project accomplishment and product of the Integrated Consortium of Laboratory Networks (ICLN) Radiological Laboratory Subgroup (RLS).

The ICLN is a system of interconnected federal laboratory networks that can quickly respond to high consequence incidents and give decision makers timely, credible, and interpretable data. The ICLN is a partnership of nine federal departments and agencies, including seven national lab networks. The ICLN provides the framework and tools that link these federal departments and agencies, federal lab networks, and lab network members so that they can manage sample analysis, share data, methods, and resources during an emergency.

The ICLN RLS seeks to increase laboratory efficiency across agency network programs and identifies radioanalytical laboratory gaps among the ICLN network radiological laboratories. In addition, this subgroup promotes consistency of analytical performance to ensure quality data supporting homeland security decisions and assessing and filling gaps in radioanalytical laboratory testing.

Over the past year, the RLS has created a new website to serve the radiochemistry community as a Radiological Laboratory information “Hub.” This new resource, referred to as the “Rad Lab Hub,” will be viewable from the public facing www.icln.org website under the “Resources” link.

- The Rad Lab Hub provides the first-of-its-kind, centralized collection of analytical methods, guides, and technical information focused specifically for laboratorians, researchers, scientists, incident commanders and other field response personnel in the radiological sciences.
- The information on the Rad Lab Hub covers radiological laboratory topics related to food, clinical, and environmental samples.
- The Rad Lab Hub currently includes 150 different resources under the following categories: Analytical methods, Operational guides, Training, reference, or educational documents, Documentary standards, Calibration, traceability, or physical standards, Laboratory Analytical Tools and Lessons learned.

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10:00	Robert Jones	Integrated Consortium of Laboratory Networks (ICLN), Radiological Laboratory Subgroup (RLS) Gamma Spec Survey Results
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INTEGRATED CONSORTIUM OF LABORATORY NETWORKS (ICLN)

RADIOLOGICAL LABORATORY SUBGROUP (RLS) RAD LAB HUB TECHNICAL ORAL PRESENTATION

Tentative Presenter: John Griggs; griggs.john@epa.gov, ICLN RLS Chairperson

Affiliation: ICLN/EPA NAREL

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10:20	Guy Backstrom	Technique Used for the Total Dissolution of a 100 gram Soil Sample
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The presenter will discuss a how a 100 gram soil sample was totally dissolved using a potassium fluoride and a pyrosulfate molten salt fusion was performed for the analyses of Strontium-90. The technique results from logical choices to reduce sample mass so that a minimal amount of flux can be used. The presenter will also discuss how the resulting fusion cake is dissolved and how many metals are complexed so that they do not precipitate with the sulfates when the Strontium is precipitated. Uses for potential Actinide and Radium analyses will also be discussed.

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10:40	Mike Schultz	Pb-212 Alpha Particle Therapy for Cancer: Exciting Progress and Radioanalytical Challenges (Zoom Presentation)
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Pb-212 ALPHA PARTICLE THERAPY FOR CANCER: EXCITING PROGRESS AND RADIOANALYTICAL CHALLENGES

Michael K. Schultz PhD

Founder and Chief Science Officer

Viewpoint Molecular Targeting, Inc./DBA Perspective Therapeutics

Abstract

Purpose. The lead-203 (^{203}Pb)/lead-212 (^{212}Pb) elementally identical radionuclide pair has gained significant interest in the field of image-guided targeted alpha-particle therapy for cancer. Emerging evidence suggests that ^{212}Pb -labeled peptide-based radiopharmaceuticals have the potential to be transformative for cancer patients. With the success of newly introduced alpha particle therapy comes radioanalytical challenges due to the complexity of the decay series. Here we provide recent preclinical and clinical evidence that demonstrates the potential for alpha particle therapy for cancer and the power of alpha particle therapy vs beta particle therapy.

Presentation Highlights: New radiopharmaceuticals based on peptides have been designed and synthesized with the goal of optimizing the incorporation of Pb isotopes through the use of a modified cyclization technique; the introduction of a Pb-specific chelator (PSC); and other chemical-structural innovations. The effect of these innovations is exemplified by highlighting improvements in receptor affinity, cellular uptake, and biodistributions in preclinical animal models and clinical human imaging. The complexity of decay series for Ac-225 and Pb-212 are compared and contrasted in the context of challenges to performing accurate and precise determinations of the administered radioactivity prior to the administration to patients.

Conclusions: Receptor targeted alpha particle radionuclide therapy has emerged as a potentially transformative approach to cancer treatment. It is anticipated that as the use of these therapies becomes mainstream in oncology that new approaches to radioactivity measurements will emerge to improve adoption by clinical sites.

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Metrological Basis for Legal Defensibility of Scientific Measurements

Kenneth G.W. Inn^{1,2}

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Abstract

Legal defensibility of scientific/technical measurements is based on doctrine laid down by the Daubert standard

- general acceptability,
- established standards controlling the technique's operation and accuracy,
- a known or potentially known rate of error, and
- the testability of the procedure.

These principles are extended by the ANSI consensus standards N42.22 on traceability and N42.33 on measurement and instrumentation quality assurance. These doctrines are further supported by metrological tools that encompass the use and development of new:

- certified reference materials [CRMs],
- traceable derived secondary reference materials,
- calibrations,
- scoping/pilot exercises,
- performance testing,
- evaluation of preparedness,
- declaration of measurement uncertainties, and
- third-party criteria and assessments,

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to provide the required basis for analytical method development, optimization, validation, and quality control.

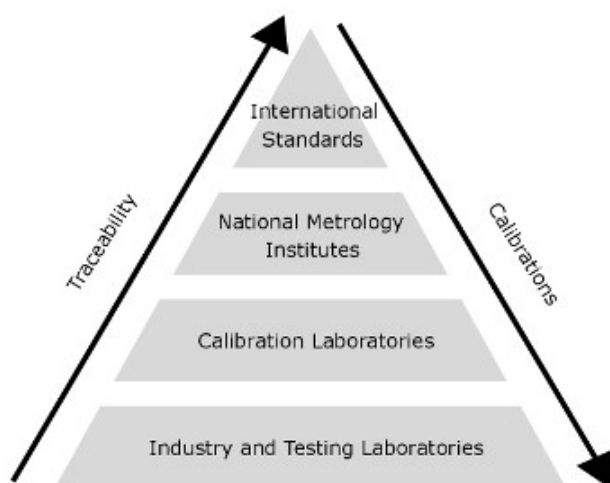
Each of these metrological tools are essential components to the realization of proving the unbroken chain of accuracy depicted in the traceability tree [see diagram] from the SI measurement unit to data obtained for environmental/climate studies. Stated uncertainties are essential to define statistical confidence levels at each tier of the traceability tree.

While it is essential that all of these universally applicable metrology tools are used to provide legally defensible environmental/climate measurement results, it is important to understand that the traceability tree is primarily a reactive rather than a proactive organism. Although the NMIs and Calibration Laboratories respond to national and international needs, it is largely the responsibility of the measurement community to proactively engage them early to make its new and emerging metrology needs known because it takes time to assist them to develop the rationale for altering their mission, finding new resources for the new efforts, develop the new quality tools, and collaboratively define metrology attributes including:

- measurand and required concentration/intensity range,
- required combined measurement uncertainty,
- matrices of concern/interest,
- measurement interferences, and
- time/cost constraints.

It is also important to realize that the tools available among the traceability trees in the international community are not all exactly alike because of varying specialities. The measurement community should collectively look internationally to find the appropriate traceability tree(s) that will provide the metrology fruit that they need. This metrological blueprint for legal defensibility provides the basis for enhanced cooperation and is a key component for international climate response.

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<https://www.adamequipment.asia/aeblog/traceability-metrology-and-mass-measurement>

Biography

Dr. Kenneth G.W. Inn is an independent consultant in the determination of the concentration, distribution, speciation and measurement quality assurance/quality control of low-level actinide, fission product and activation product radionuclides in environmental and biological systems by ultra-clean radiochemistry and ultra-high sensitive and selective measurement methods. His Ph.D. is from the U of Arkansas in Environmental Radiochemistry. Dr. Inn's directed programs in low-level radionuclide environmental-matrix Standard Reference Materials, radionuclide speciation in soils and sediments, and emergency response [nuclear terrorism or unintentional release] and low-level radiochemistry traceability evaluations. He was a Research Chemist at the Nat. Bureau of Standards 78-88; NIST Group Leader, Office of Radiation Measurements 92-94, and Radioactivity Group Project Leader in Low-Level Radiochemistry 95-13. Dr. Inn is member of the American Chemical Society, the Geochemical Society, Journal of Radioanalytical and Nuclear Chemistry Distinguished Reviewers' Board 19-Present, and former member of the EPA/SAB/RAC 15-21 and NTNFC Reference Material Committee 08-13.

Presenting Author Details and Photo

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Thursday, November 2, 2023

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Recent Photograph:



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8:00	Ingrid Lehman-Andino	Strategies for actinide (An) and cesium (Cs) removal from alkaline high-level waste.
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Strategies for actinide (An) and cesium (Cs) removal from alkaline high-level waste.

Ingrid Lehman-Andino^a, Leigh D. Ruddock^b, Rene Panzer^b, Oluwaseun W. Adedoyin^b, David P. DiPrete^a, Konstantinos Kavallieratos^b

^a Savannah River National Laboratory, Aiken, United States

^b Department of Chemistry and Biochemistry, Florida International University, Miami, United States.

The United States and former Soviet Union nuclear arms race resulted in the generation of large volumes of alkaline High-Level Waste (HLW). The majority of this legacy waste still stored in the US in underground tanks at the Savannah River Site (SRS) and the Hanford Site (HS). The stored tank waste is comprised of three layers: (i) the high activity sludge, (ii) the saltcake, and (iii) the supernatant. The sludge and salt cake can directly be processed for vitrification and for saltstone disposal. Current integrated processing of caustic HLW is comprised of a two-step process, the Actinide Removal Process (ARP) separating ⁹⁰Sr and An through sorption on monosodium titanate followed by the Caustic-Side Solvent Extraction process (CSSX) for ¹³⁷Cs separation, which has been more recently evolving into the more effective Next-Generation CSSX process (NG-CSSX). Further improvement of integrated HLW processing could be achieved by incorporating innovative chelators capable of extracting Sr, Cs, and An, and are compatible with the NG-CSSX solvent. Dr. Kavallieratos' group has been identify Sulfonamides derivatives as promising chelators for *f*-elements and investigated their Sm(III) extraction performance from alkaline aqueous solutions. All extractants were tested at SRNL for their radiolytic stability and extraction capability using an HLW simulant (Sr-90, Cs-137, Eu-154, Pu-239, Am-241). Our progress and recent results will be presented.

Friday, November 3, 2023

08:20	Mark A. Jones	Applications of Alternate Actinide Calibration on Inductively Coupled Plasma Mass Spectrometry (ICP-MS) Analysis of H-Canyon Highly Enriched Uranium Legacy Material at the Savannah River Site (SRS)
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Applications of Alternate Actinide Calibration on Inductively Coupled Plasma Mass Spectrometry (ICP-MS) Analysis of H-Canyon Highly Enriched Uranium Legacy Material at the Savannah River Site (SRS)

Mark A. Jones, Henry J. Bonilla, Scott D. Brown

The Savannah River National Laboratory (SRNL) uses Inductively Coupled Plasma Mass Spectrometry (ICP-MS) to support numerous programs at the Savannah River Site (SRS). As part of an objective to prepare high assay low enriched uranium (HALEU), the SRS H-Canyon needed analysis of legacy uranyl nitrate that is stored at its Outside Facilities (HOF). With the application of a standard practice for alternate actinide calibration on ICP-MS, SRNL has successfully demonstrated its capability to characterize the uranium/actinide composition of the HOF tanks that are intended for consolidation prior to preparation of the HALEU. A comparison of the uranium analytical results between recently upgraded SRNL ICP-MS and Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) instrumentation shows the alternate actinide calibration is a valid complementary method when used in conjunction with other analytical techniques (ICP-OES and Davies-Gray Potentiometric Titration). The methods, instrumentation, and ongoing developments on ICP-MS at SRNL will be discussed.

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08:40	Hugo Jaegler	New insights into analysis of ultra-low $^{236}\text{U}/^{238}\text{U}$ isotope ratio by ICP-MS/MS
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NEW INSIGHTS INTO ANALYSIS OF ULTRA-LOW $^{236}\text{U}/^{238}\text{U}$ ISOTOPE RATIO BY ICP-MS/MS

Hugo Jaegler

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Institut de Radioprotection et de Sûreté Nucléaire (IRSN), PSE-ENV/SAME/LERCA, F-78110, Le Vésinet, France

Hugo Jaegler, Alkiviadis Gourgiotis

Uranium isotope measurements are of increasing interest for environmental sciences, nuclear safeguards, and nuclear forensics. Recently, several studies focused on the minor isotopes of uranium in the environment. In fact, minor isotope as ^{236}U is of prime interest for the determination of uranium sources investigation due to the variation of its isotope ratio (i.e., $^{236}\text{U}/^{238}\text{U}$) regarding their origin. The isotopic variations of the $^{236}\text{U}/^{238}\text{U}$ isotope ratio between the geochemical background (10^{-14} – 10^{-12}), the uranium ore (10^{-12} – 10^{-10}) and the global fallout (10^{-9} – 10^{-7}), make this ratio a relevant fingerprint of uranium sources in the environment.

However, detection of ^{236}U in environmental samples is quite challenging due to its extremely low abundance and for this reason specific instrumentation disposing high sensitivity and high resolution capabilities is required. The analytical science community has been working for several years to reduce the detection limit of the $^{236}\text{U}/^{238}\text{U}$ isotopic ratio using mass spectrometry (in particular AMS), and recently by using the inductively coupled plasma tandem mass spectrometry (ICP-MS/MS). Recent works focused on the determination of very low isotope ratios by improving the hydride formation, the peak tailing, etc [1]–[3]. In a previous work, the lowest isotope ratios measured using the ICP-MS/MS technology in UO^+ mode was about 1×10^{-10} [4].

The latest generation of ICP-MS/MS, recently commercialized, has improved performance, including better ion transmissions and an extended dynamic range of mass detection. This allows measurements of isotopes until mass to charge ratio of 280. This is a great improvement as uranium ions could now be measured as UO_2^+ rather than UO^+ which shows important advantages in terms of sensitivity and hydride formation. In this work we explored the capabilities of the new model of ICP-MS/MS, Agilent 8900, in terms of hydride formation rate and the lowest achievable $^{236}\text{U}/^{238}\text{U}$ isotope ratio in UO_2^+ mode. To this end, measurements of certified reference materials and environmental samples were performed.

These improvements as well as the coupling with an efficient desolvating module (APEX Ω) and the addition of oxygen in the collision/reaction cell allowed an important sensitivity increase and an effective decrease of the interferences on $^{236}\text{U}^+$ ion measurements: the sensitivity increased to 4.3×10^6 cps/ppb while reducing the main interference with an hydride formation rate of 7.2×10^{-10} . These values are one order of magnitude better than previous published values and allow ultra-low $^{236}\text{U}/^{238}\text{U}$ isotope ratios measurements.

For the first time a precise determination of the $^{236}\text{U}/^{238}\text{U}$ isotopic ratio up to 10^{-11} was demonstrated [5], validated by the analysis of certified reference materials and environmental samples previously measured by AMS. This new limit is a new milestone in the determination of ultra-low $^{236}\text{U}/^{238}\text{U}$ isotope ratios and open the way to various geochemical applications.

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Friday, November 3, 2023

09:00	Guosheng Yang	⁹⁰ Sr bioassay in small-volume urine by ICP-MS/MS with CO ₂ as the reaction gas in rapid response to radiological emergencies
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⁹⁰Sr bioassay in small-volume urine by ICP-MS/MS with CO₂ as the reaction gas in rapid response to radiological emergencies

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Abstract

As the “core advanced radiation emergency medical support center” designated by the Nuclear Regulation Authority, Japan, the National Institutes for Quantum Science and Technology (QST) is working on developing human resources and enhancing Japan’s preparedness and response to nuclear accidents [1]. To provide timely information in radiation emergency medical treatment, we have developed rapid bioassay methods to measure U, Pu, Np, Am, and Cm radioisotopes in urinary and fecal samples with the combination of radiometric and mass spectrometric techniques [2].

Apart from the mentioned actinides above, it also is critical to perform dose assessment after internal contamination with beta-particle emitting ⁹⁰Sr due to its relatively long physical (28.74 y) and biological (~ 18 y) half-lives [3,4], along with the higher specific activities of ⁹⁰Sr (5.11×10^{12} Bq/g) and its daughter ⁹⁰Y (2.01×10^{16} Bq/g). For rapid public screening after a large-scale radiological/nuclear accident and/or nuclear terrorist attack, we developed a novel ⁹⁰Sr bioassay for small-volume urine samples using triple-quadrupole inductively coupled plasma–mass spectrometry (ICP-MS/MS). After organic matter decomposition of 10 mL urine by HNO₃, stacked DGA and Sr resin cartridges were used directly for chromatographic separation and purification of Sr. Yields of Sr were measured as $94 \pm 5\%$ (n=12) for the whole procedure, using stable ⁸⁸Sr originally in the urine sample as a yield tracer. CO₂ proved to be a better reaction gas than O₂, to overcome isobaric/polyatomic interferences, especially those caused by Zr and Ge. With a conventional introduction system + s lens + 75% CO₂ flow rate in the 4th cell gas line, the method detection limit (MDL) was estimated to be 0.978 pg/L (5 Bq/L) in 10 mL urine, meeting the public screening requirement for judging ingestion/inhalation exposure beyond 1 mSv committed effective dose equivalent, with a reasonable throughput (~ 6 h for 12 samples). For method validation, the standard reference materials provided by PROCORAD (Association for the PROMotion of Quality Control in RADiotoxicological Analysis), France, were analyzed and the results were all in good agreement with the assigned values.

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Friday, November 3, 2023

09:20	Marisa Alfonso	Novel Applications of Eckert & Ziegler Analytics Custom Produced Sources
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NOVEL APPLICATIONS OF ECKERT & ZIEGLER ANALYTICS CUSTOM PRODUCED SOURCES

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Abstract:

Eckert & Ziegler Analytics serves a diverse community within the broad nuclear industry. This presentation will provide examples of recent work to support emerging applications in the fields of emergency response, NORM, metrology, decommissioning, and space exploration. The audience is encouraged to bring comments and questions to guide the next wave of development.

Friday, November 3, 2023

10:00	Andrew A. Reinhard	Application of ATONA Amplifiers to Uranium Isotopic Analyses by Thermal Ionization Mass Spectroscopy
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APPLICATION OF ATONA AMPLIFIERS TO URANIUM ISOTOPIC ANALYSES BY THERMAL IONIZATION MASS SPECTROMETRY

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In nuclear forensic investigations, nuclear safeguards applications, treaty monitoring, and treaty verification, the uranium isotopic composition in a sample provides valuable information about the production history and source of uranium material. The utility of these data has created a continual push within the field to measure uranium isotopic ratios in smaller sample sizes while reducing detection limits and improving measurement precision. Historically, when analyzed by thermal ionization mass spectrometry (TIMS), small uranium samples (< 1 ug) have necessitated time consuming peak-hopping routines such that the minor uranium isotopes (^{234}U , ^{236}U) could be measured using an ion-counting detector. However, the recently developed ATONA amplification system (IsotopX Ltd) uses capacitance-based amplification rather than traditional resistance-based amplification, significantly improving the noise floor and increasing the dynamic range of Faraday cup detectors. These improvements allow for both major (^{238}U , ^{235}U) and minor (^{234}U , ^{236}U) isotopes of uranium to be measured on Faraday cups. By simultaneously measuring masses 234, 235, and 238 on Faraday cups and measuring only mass 236 on an ion-counter a total evaporation technique can be utilized which decreases the total time and the complexity of a measurement. Data will be presented from several certified reference materials (CRMs) with uranium isotopic compositions ranging from extremely depleted (NBL CRM U0002) to highly enriched (NBL CRM U930). Further aliquots of the Columbia River sediment standard reference material (NIST 4350b) were digested, processed through separation chemistry, and analyzed for their uranium isotopic composition by TIMS using the ATONA amplification system. Replicate NIST4350b aliquots of 10, 20, 50, and 200 ng of total uranium were analyzed to determine measurement reproducibility and accuracy of uranium separated from a complex matrix at different sample sizes.

LA-UR-23-29233

Friday, November 3, 2023

10:20	David L. Cook	A New Anion Exchange Protocol for the Separation of Uranium from Environmental Samples
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A New Anion Exchange Protocol for the Separation of Uranium from Environmental Samples

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Separation of trace quantities of U from environmental samples is routinely performed for treaty monitoring programs at Los Alamos National Laboratory. The current separation protocol consists of three ion exchange columns that employ “homemade” columns packed with quartz wool and require numerous reagents. In addition, one of the three columns utilizes H₂SO₄, based on Strelow and Van Zyl (1967). The use of H₂SO₄ requires hot plate dry downs of the U fraction near the melting temperature of the labware, introducing additional hazards to the procedure. Ion exchange properties in various acid media suggest that U could be sufficiently separated without the use of H₂SO₄ (e.g., Saito, 1984; Edwards et al., 1986). Another drawback of the current protocol is the use of H₂O₂ mixed with various molarities of HCl on the second column. The presence of H₂O₂ often leads to bubble formation in the resin bed, which can cause channeling and has the potential to reduce the effectiveness of the separation. We investigated a new protocol with the following goals: to reduce the overall processing time needed for the chemical separation of U, to eliminate the use of H₂SO₄ and H₂O₂, to improve the reproducibility of the method among different chemists, and to potentially reduce the procedural blank of the method by simplifying the overall separation.

A column made from a 2 ml transfer pipette (internal diameter of 0.7 cm), packed with quartz wool, was filled with 1 ml of Eichrom AG1×8 (200-400 mesh) anion resin. The resin was cleaned and conditioned with > 18.2 MΩ water followed by 7.5M HNO₃ prior to sample loading. Samples were prepared by mixing 4 different multi-element ICPMS standard solutions and loading an aliquot equivalent to 1 μg of each element present. Samples were loaded in 7.5M HNO₃; matrix elements were eluted with additional 7.5M HNO₃, followed by 6M HCl. Uranium was then eluted with 0.1M HCl. Aliquots collected throughout the procedure were measured via quadrupole ICPMS and used to construct elution curves. These tests showed a sharp elution peak for U and good separation from the matrix. Subsequent tests using multiple replicates of NIST 4350b (Columbia River sediment) confirmed high yields of U (i.e., > 90%) that is well-separated from the major and minor rock-forming elements (i.e., Na, Mg, Al, K, Ca, Cr, Mn, Ti, Fe), plus numerous trace elements. Future tests will include determining procedural blanks by isotope dilution and measuring isotopic ratios via TIMS on U separated from matrix-appropriate standards using the new purification scheme.

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Friday, November 3, 2023

10:40	Adam Judy	Non-Destructive Assays of Various Geometries for SRNL Mk-18 Program
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Non-Destructive Assays in Support of Shielded Cells Cleanout

Adam Judy

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The Savannah River National Laboratory has been working to clean out, renovate and repurpose the Shielded Hot Cells used for handling and processing high dose material. This is required to create capacity and capability to support new scope in the facility, such as the MK 18 project which will dissolve new fuel types for extraction of rare isotopes. A key part of the process is the removal of legacy equipment and structures, many of which have been in place for many decades. The amount and type of radioactive material remaining on and within these items is critical knowledge for the planning and execution of waste management.

Throughout the campaign, various geometries and techniques were used to quantify the contamination on these items. Using ISOCS, a modeling software from Mirion Technologies, the items were able to be assayed, modeled, and the radionuclide inventories determined to aid in their waste classification and ultimately the milestone project for the laboratory. The processes of developing and executing analysis plans using a combination of non-destructive assay and radiochemistry will be discussed.

Friday, November 3, 2023

1	Olga Piraner, Karlee Eardley, and Jon Button	Alpha and Beta Spillover in Liquid Scintillation Counting Analysis of Urine Samples
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**ALPHA AND BETA SPILLOVER IN LIQUID SCINTILLATION COUNTING
ANALYSIS OF URINE SAMPLES**

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As part of the CDC's mission for radiological/nuclear emergency preparedness, we are developing methods that will be used to rapidly detect and quantify gross alpha and beta-emitting radionuclides in urine samples from populations suspected of internal contamination. Rapid detection and quantification of gross alpha/beta-emitting radionuclides by liquid scintillation counting (LSC) are vital in guiding laboratory response activities to radiation emergencies. Liquid scintillation counters use signal pulse shape to discriminate alpha and beta events in samples but require precise optimization to minimize the spillover or misclassification of those events. The primary focus of this work lies in understanding the relationship between sample activity and spillover in urine, with comprehensive analyses conducted on various LSC instruments. Additionally, investigations into sample matrix and emission particle effects contribute to a more comprehensive understanding of LSC performance in radiation emergency scenarios.

Disclaimer

The findings and conclusions in this study are those of the authors and do not necessarily represent the views of the U.S. Department of Health and Human Services or the Centers for Disease Control and Prevention. Use of trade names and commercial sources is for identification only and does not constitute endorsement by the U.S. Department of Health and Human Services or the Centers for Disease Control and Prevention. The authors declare that they have no competing financial interest.

POSTER SESSIONS, Tuesday, October 31, 15:00 – 17:00, Wednesday, November 1, 14:00 – 16:00

2	Ge Xiao, Jonathan Button, Cynthia Ward, and Liza Valentin-Blasini	Rapid Determination of Np-237 in Urine by Simple Dilution Using Triple Quadrupole Inductively Coupled Plasma Mass Spectrometry (ICP-QQQ-MS)
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Rapid Determination of Np-237 in Urine by Simple Dilution Using Triple Quadrupole Inductively Coupled Plasma Mass Spectrometry (ICP-QQQ-MS)

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Abstract

Determination of Np-237 in urine at low levels is vital for radiological emergency response. Here we describe method development and validation for a rapid method used to measure Np-237. We use a simple dilution of the urine sample followed by triple quadrupole inductively coupled plasma mass spectrometry (ICP-QQQ-MS) determination using “no gas MS/MS” mode. The method delivers accurate results with a limit of detection (LOD) of 0.006 ng/L. This LOD is below the recommended action level of 0.812 ng/L for Np-237 in children and pregnant women set by the Clinical Decision Guide (National Council on Radiation Protection and Measurements Report #161). Results for Np-237 obtained by this method are within $\pm 8.0\%$ of the target values of certified reference materials spiked in urine. This method provides a more rapid and robust platform to operate in comparison to the existing sector field (SF)ICP-MS method, which requires complex sample column purification and a minimum sample volume of 2 mL. Although this method's LOD is higher than that of SF-ICP-MS, the method is fit for rapid emergency response. Carrying out the analysis requires only a minimal 0.5 mL of each urine sample, making the method suitable for a broad range of people, such as young children and infants, who produce limited urine volumes.

Disclaimer

The findings and conclusions in this study are those of the authors and do not necessarily represent the views of the U.S. Department of Health and Human Services or the Centers for

POSTER SESSIONS, Tuesday, October 31, 15:00 – 17:00, Wednesday, November 1, 14:00 – 16:00

Disease Control and Prevention. Use of trade names and commercial sources is for identification only and does not constitute endorsement by the U.S. Department of Health and Human Services or the Centers for Disease Control and Prevention. The authors declare that they have no competing financial interest.

POSTER SESSIONS, Tuesday, October 31, 15:00 – 17:00, Wednesday, November 1, 14:00 – 16:00

3	Matthew I. Warne, et al	Curium/Californium Separation for Cm-242 Determination
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Curium/Californium Separation for Cm-242 Determination

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A rapid separation of curium from californium using a diglycolamide (DGA) based extraction chromatographic resin was developed and validated to determine the Cm-242/Cm-244 ratio in original dissolution solution at the beginning of a californium campaign. The production of californium-252 is obtained by the irradiation of curium targets in the High Flux Isotope Reactor (HFIR) at ORNL. The presence of americium, specifically americium-241, in the target leads to the presence of curium-242 in the irradiated product due to neutron capture. Curium-242 is a short half-lived alpha emitter with its primary alpha energy at 6.11 MeV. This energy overlaps almost exactly with the primary alpha energy of californium-252. The determination of the californium content must then be done exclusively by gross neutron measurements. While californium-252 is the major neutron emitter, it is not the sole contributor, which creates some uncertainty on the mass determination. Isotopic determination has historically been done by ICP-MS analyses on very dilute samples and requiring HPLC separations involving sample transfer. This process, necessary to establish the full range of isotopics for americium, curium, and californium, is lengthy and is typically unable to rapidly provide the percentage of produced curium-242 at the beginning of a campaign. It was demonstrated that a DGA column was a suitable method to quantitatively separate curium from californium and therefore establish the amount of curium-242 compared to the amount of curium-244 as the percentage of alpha emission at 6.11 MeV is then only due to curium-242. In a californium campaign, curium-244 is generally the strongest alpha emitter with a unique alpha energy of 5.80 MeV, making its determination extremely reliable. Being able to establish a solid ratio between curium-242 and curium-244 is the assurance that the amount of Cm-242 can be calculated throughout the campaign and that its contribution can be accurately subtracted from the major 6.11 MeV emission abundance, providing a method to calculate the amount of californium present in solution by alpha spectroscopy. The separation performed was successful in removing californium from curium to a large extent, evidenced by the concentration of nearly all neutron activity into the final elution cut of the separation.

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POSTER SESSIONS, Tuesday, October 31, 15:00 – 17:00, Wednesday, November 1, 14:00 – 16:00

4	Kyung-Min Lee	Investigation and Assessment of Natural Radioactivity in Commercial Animal Feeds in Texas
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INVESTIGATION AND ASSESSMENT OF NATURAL RADIOACTIVITY IN COMMERCIAL ANIMAL FEEDS IN TEXAS

Kyung-Min Lee and Timothy J. Herrman

Office of the Texas State Chemist, Texas A&M AgriLife Research

The radioactivity concentration in animal feed is critical since animals may ingest contaminated feed that could contaminate the human food supply. To date, there has been limited investigation of natural radionuclide activity concentrations in commercial animal feeds in the US as well as in other parts of the world. Approximately 4000 firms in Texas, the United States and abroad manufacture and distribute 23 million tons of feed in Texas each year.

This project involves the assessment of naturally occurring radionuclides in commercial animal feed distributed in Texas and compares those values with those reported in the previous works performed in other countries and US. In this study, we determined natural gamma emitters including ^{40}K , ^{137}Cs , and Uranium (^{235}U and ^{238}U) and Thorium (^{232}Th) series using a high-resolution HPGe detector in select types of animal feeds that were collected and analyzed for regulatory compliance.

The study results showed that the measured activity concentration of natural radionuclides in animal feed produced in Texas was low enough for safe consumption by animals and depended on the type of animal feed. ^{40}K was the predominant radionuclide showing the highest activity concentration in all animal feed types except for vitamin/mineral mix. In vitamin/mineral samples, the ^{40}K activity was in proportional to the level of potassium in samples. The radioactivity concentration of ^{214}Bi and ^{214}Pb in ^{238}U decay series was 1.39 and 1.33 Bq/kg in corn, respectively. This was lower than in other animal feed types. On the other hand, the vitamin/mineral mix samples showed higher concentrations of ^{214}Bi (9.04 Bq/kg) and ^{214}Pb (10.19 Bq/kg). Beef cattle and poultry feed exhibited slightly higher activity concentration of ^{228}Ac and ^{212}Pb in ^{232}Th decay series while the concentrations of these radionuclides were significantly higher in vitamin/mineral mix ($p < 0.05$) than other animal feed types. Gamma radionuclides detected in animal feed appeared to be highly and significantly correlated within each decay series as well as between decay series depending on animal feed type and radionuclide. ^{235}U was present in all feed samples, but the measured concentrations were lower than those of ^{238}U and ^{232}Th decay products. The anthropogenic radionuclide of ^{137}Cs was not detected in samples irrespective of the type of animal feed. It is important to establish a current baseline of radioactivity concentration in animal feed in Texas, where the largest animal feed consumption in the US exists. The Office of the Texas State Chemist can serve as a sentinel lab of future radiological events or naturally occurring contamination for the US food and feed industry.

POSTER SESSIONS, Tuesday, October 31, 15:00 – 17:00, Wednesday, November 1, 14:00 – 16:00

6	William Stephenson	Determination of Sr-90 in Freshwater Fish Near the Fukushima Daiichi Nuclear Power Plant
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DETERMINATION OF SR-90 IN FRESHWATER FISH NEAR THE FUKUSHIMA DAIICHI NUCLEAR POWER PLANT

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Abstract – In the wake of the Fukushima Nuclear Power Plant (FNPP) accident in Japan, commercially sold food products from the Fukushima prefecture have come under increased scrutiny by the public due to potential bioconcentration of radionuclides. The fish that are harvested from fresh and salt waters are one such product of interest. The main isotopes of concern in fish are ⁹⁰Sr and ¹³⁷Cs. These are radioisotopes that have been found to substitute stable Ca and K in bone and muscle tissue, respectively. This research will explore the concentration of ⁹⁰Sr in freshwater fish sampled near the FNPP to determine if they are safe for consumption. This assessment is important in assuaging the fears of the public regarding the safety of products exported from the Fukushima region, allowing for commerce to flourish once again in the Fukushima prefecture.

Fish have been sampled from the Ukedo River located northwest of the FNPP. Bone samples for ⁹⁰Sr have been prepared by separating tissues from the bones and then drying. Then, bone sample aliquots were divided between Colorado State University and Hirosaki University for a comparison study using the same dissolution and separation method. Samples were ashed and dissolved for separation of Sr and Y via Eichrom DGA resin. Analysis and determination of ⁹⁰Sr was completed via liquid scintillation counter at Colorado State University and via inductively coupled plasma mass spectrometer at Hirosaki University. Sample results and method limits of detection will be compared to those obtained by our collaborators at Hirosaki University.

POSTER SESSIONS, Tuesday, October 31, 15:00 – 17:00, Wednesday, November 1, 14:00 – 16:00

RAPID METHOD FOR ACTINIDES IN SEAWATER USING ACTINIDE RESIN

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The marine environment has been contaminated by transuranium since the global fallout resulting from extensive nuclear weapons testing conducted in the 1950s and 1960s. Following the substantial release of radioactive materials from the damaged Fukushima nuclear power plant in 2011, concerns about radioactive contamination in the ocean have also been on the rise. It is challenging to determine actinides in seawater due to its very low limit of detection. Hence, the development of a rapid and efficient method for pre-concentrating large volumes of seawater is of utmost importance. Co-precipitation methods stands as the most widely employed pre-concentration technique. Various co-precipitation techniques using hydroxides, phosphates, and fluorides have been applied for actinides. While these methods are traditional and cost-effective, they tend to be time-consuming and labor-intensive. In recent studies, a novel approach using extraction resin, known for its high selectivity for actinides, were reported to concentrate water samples. Among commercial resins, Actinide Resin[®] (eichrom) exhibited the highest distribution ratios for almost actinides at low acid concentrations. The aim of the present study is to develop a method for the rapid analysis of 20 L of sea water using Actinide resin as an adsorbent. Actinides in seawater were adsorbed onto the Actinide resin at a speed of 50 ml/min per column under pH 1 conditions. A volume of 5 L of seawater was passed through 2 ml (bed volume) of Actinide resin. With the using of 4 columns at once, enrichment of actinides from 20 L of seawater was achieved in approximately 100 minutes. Isopropyl alcohol (IPA) was employed to strip the actinides and extractant together from the Actinide resin. Following this, the eluted IPA was dried on a hot plate for 1 hour, followed by microwave-assisted digestion using a mixed acid solution. For removing high-concentration diphosphonic acid, CaF₂/LaF₃ co-precipitation was employed. Subsequent purification was carried out using TEVA/DGA resin. The determination of actinide concentration was performed using alpha spectrometry and the entire process, from actinides adsorption to the final purification, took approximately 6 hours.

This work was supported by the Nuclear Safety Research Program through the Korea Foundation Of Nuclear Safety (koFONS) using the financial resource granted by the Nuclear Safety and Security Commission (NSSC) of the Republic of Korea. (No. 00231329)

8	Raissa Chunko	The Influence of Trace Metals in Asphalt on Plutonium Uptake on Extraction Chromatography Resins
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THE INFLUENCE OF TRACE METALS IN ASPHALT ON PLUTONIUM UPTAKE ON
EXTRACTION CHROMATOGRAPHY RESINS

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At the dawn of a new nuclear age, standard radioanalytical techniques must be available to analyze radionuclides in unusual matrices. Radiochemical analysis of samples in standard matrices of soil, water, and air are very well established; however, much less research has been conducted on the effect of unusual matrices such as steel, concrete, glass, and asphalt. In the event of a detonation of an improvised nuclear device (IND) in an urban environment, the standard separation techniques used for plutonium separations from asphalt samples originating from roadways and roofing shingles must rigorously be tested to provide useful insight on the characteristics of the special nuclear material. Batch studies were used to determine the changes in uptake of plutonium on extraction chromatography resins in the presence of trace metal components found in asphalt including aluminum, iron, and manganese. Data analysis is still in progress at this time, but it is expected that iron has the potential to interfere with the uptake of plutonium on extraction chromatography resins while aluminum likely will not interfere with the interactions between plutonium and the resins of interest. The uptake of manganese on extraction chromatography resins has not been studied, but due to its oxidation state and atomic radius, manganese does have the potential to interfere with plutonium uptake on the resins of interest. New data will be presented on the influence of unique trace metal as contaminants found in asphalt on the uptake of plutonium on extraction chromatography resins.

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9	Donivan Porterfield	Development of Teva Resin Extraction Chromatography Separation for Np Determination in Pu Materials using Gamma Spectrometry
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DEVELOPMENT OF TEVA RESIN EXTRACTION CHROMATOGRAPHY SEPARATION FOR Np DETERMINATION IN Pu MATERIALS USING GAMMA SPECTROMETRY

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Np-237 is commonly present in Pu materials due to its production in nuclear reactors and being a decay product of Pu ($^{241}\text{Pu} \rightarrow \beta^- + ^{241}\text{Am} \rightarrow \alpha + ^{237}\text{Np}$). Quantifying low levels of ^{237}Np is complicated by the predominantly Pu matrix and high activity of ^{241}Am . Establishing effective redox control presents another considerable challenge for the separation of trace levels of ^{237}Np from a Pu matrix. Most methods of ^{237}Np analysis in Pu-materials utilize either mass spectrometry or radiochemical analysis. However, radiochemical analysis is generally only possible when paired with complex separations techniques to substantially eliminate any Am/Pu interferences. For example, one method involves both a cation exchange (IX) separation and a thenoyltrifluoroacetone (TTA)-methyl benzoate solvent extraction (SX) followed by measurement with gas flow proportional alpha counting, alpha spectrometry, and NaI(Tl) gamma spectrometry. This method often results in low chemical yields, requiring long count durations for sufficient data statistics and quality. In this study, we seek to develop and verify a rapid, robust separations technique to increase sample throughput and eliminate the need for multiple radiochemical analysis techniques, thus improving on the IX/SX method for routine ^{237}Np determination in Pu materials.

The approach investigated within this work couples vacuum-assisted TEVA Resin extraction chromatography with high-purity germanium (HPGe) gamma spectrometry. Similar approaches detailed within the literature have provided evidence for successful ^{237}Np separation and use of a single instrumental analysis. TEVA Resin chemistry, when paired with appropriate valence control, retains Np(IV) on the resin while +III oxidation state actinides (e.g., Am, Pu) are largely eluted through rinsing. High chemical yields from the separation (~95%), determined through the implementation of a ^{239}Np tracer, permit relatively short count durations. This study will compare the TEVA/HPGe method with both the IX/SX radiochemical analysis method and inductively coupled plasma mass spectrometry measurements on various Pu sample matrices. Additionally, a full uncertainty model will be generated for the TEVA/HPGe method. Ultimately, this work will expand upon current methodology through the investigation of the

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method's validity with Pu-material matrices, the use of a secular equilibrium tracer solution, the comparison to various analysis methods, and the development of the overall method uncertainties and detection limits for a new application of the TEVA/HPGe method.

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10	Alejandra Hernandez-Jimenez	Comparing the Accuracy and Efficiency of Different Fitting Routines for Alpha Spectrometry
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COMPARING THE ACCURACY AND EFFICIENCY OF DIFFERENT FITTING ROUTINES FOR ALPHA SPECTROMETRY

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Alpha spectrometry is used widely to quantify alpha particles that are emitted during the radioactive decay process of an unstable nucleus. Alpha particles are highly energetic subatomic particles that are heavy and slow and most of its spent energy occurs at emission making them easy to shield. Alpha spectrometry is the most simple and selective way of quantifying alpha emitting isotopes of elements like plutonium, uranium, thorium, americium and curium. With the help of a chemical separation, it is efficient to quantify the activity of specific alpha emitting radioisotopes using alpha spectrometry. The counting system includes passivated implanted planar silicon (PIPS) detectors coupled with Genie2K software that allows to obtain energy and peak information. The general assumption is that each radioisotope gives a signal translated into a peak form at a characteristic energy or energies that are directly proportional to the amount of the alpha emitting isotope. These peaks are called regions of interest (ROI).

Because not all matrices are the same, chemical separations and plating are not always 100% efficient in all matrices, the regions of interests will vary and will not be perfect bell curve peaks. Some biases include peak broadening, peak shoulders and background noise. Therefore, determining how to proceed when fitting peaks is essential to the process since this will affect the quantification of radioactivity. The region of interest fitting routine of choice is the manual fitting, which allows to eliminate most biases, but is a time consuming technique that requires practice. This research will look into various fitting routines like manual peak fitting, least square peak fitting, peak easy fitting and ruler method fitting. All fitting routines will use $^{238/239}\text{Pu}$ recoveries to determine which fitting routine is the most accurate and efficient when quantifying alpha emitting isotopes in different matrices.

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11	Jung Seok Chae	Determination of Rn-222 and Ra-226 in Water Using Liquid Scintillation Counter
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DETERMINATION OF RN-222 AND RA-226 IN WATER USING LIQUID SCINTILLATION COUNTER

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^{222}Rn and its parent nuclide, ^{226}Ra are ubiquitously present in nature as progenies produced in the decay series of ^{238}U . ^{222}Rn is a major cause of lung cancer, accounting for 3-14% all cases and comprises ~54% of the average individual annual effective dose from natural radiation exposure. Daily drinking water is one of the major route of ^{222}Rn and ^{226}Ra ingestion. More than 99% of radon absorbed into the human body through ingestion decays within a month due to its short half-life ($t_{1/2} = 3.8$ d), but ^{210}Pb produced by the radioactive decay of ^{222}Rn has a relatively long half life and can remain in the body and cause continuous internal exposure. In this study, a simple sequential analysis method for determination of ^{222}Rn and ^{226}Ra in water was developed.

The activity concentrations of both ^{222}Rn and ^{226}Ra were determined by ^{222}Rn -emanation method with ^{222}Rn extracted in the Maxilight+ cocktail. The alpha spectra of ^{222}Rn , ^{218}Po , and ^{214}Po by liquid scintillation counting were used to estimate the ^{222}Rn and ^{226}Ra activity concentrations, meaning that the counting efficiency can theoretically reach 300%. The pulse length index (PLI) value of 12 was applied for liquid scintillation counting for 300SL (HIDEX). The optimal pulse shape analysis (PSA) value of 110 was determined by estimating PSA values obtaining according to E^2/B (E: counting efficiency; B: background count rate) for Quantulus 1220 (PerkinElmer).

Sample for the determination of ^{222}Rn activity concentration was measured approximately 3 hours after sampling to allow the progenies (^{218}Po , ^{214}Pb , ^{214}Bi , and ^{214}Po) of ^{222}Rn to reach radioactivity equilibrium with ^{222}Rn . The activity concentration of ^{226}Ra was determined by measuring the ^{222}Rn and its progenies (^{218}Po and ^{214}Po) extracted into the cocktail 30 days after sampling. The results of this study presented methods and equations for correcting for the decay of ^{222}Rn from sampling to measurement in ^{222}Rn measurements and for eliminating the effect of native ^{222}Rn activity in ^{226}Ra measurements. In addition, the results of the evaluation of MDA as a function of counting time for the method presented in this study are discussed.

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12	Han Chang	Evaluation of Practical Sealing Methods for Preventing Rn-222 Leakage in Gamma-Ray Spectrometry
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EVALUATION OF PRACTICAL SEALING METHODS FOR PREVENTING RN-222 LEAKAGE IN
GAMMA-RAY SPECTROMETRY

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The determination of ^{226}Ra in NORM samples using gamma-ray spectrometry has been based on the detection of gamma-rays of the radon progeny (^{214}Pb , ^{214}Bi). This method requires a storage period of minimum 3 weeks, during which the sample has been tightly sealed to ensure secular equilibrium between ^{226}Ra and ^{214}Pb , ^{214}Bi . Radon, as a noble gas, can leak from the sample container easily. Therefore, special care is needed to prevent radon leakage for ingrowth period. Several studies suggested the various sealing methods for radon tightness. Vacuum packaging of the sample container in a sealed aluminum bag is one of the effective methods. And, it may be a solution to seal between the container body and the lid with a sealant such as plastic tape, paraffin, epoxy adhesive, aluminum tape. In most cases, methods of sealing the outside of the sample container were used. However, if the amount of the sample is not sufficient, and there is an empty space inside the sample container, radon moves to the empty space making it difficult to accurately apply the detection efficiency. Therefore, it may be difficult to use these methods when the sample amount is insufficient.

In this study, we presented a practical sealing method to eliminate the empty space between sample and container lid regardless of the sample amount. The sealing method is as follows. The sample is filled into a cylindrical polycarbonate container having a inner diameter of 60 mm, a height of 54 mm, a wall thickness of 1.5 mm. The top of the sample is covered with an acrylic of 60 mm in diameter and 10 mm in thickness. Then seal the gap between the measurement container and the acrylic with a sealant. Radon leakage may vary depending on the type of sealant used. Two type of experiments were conducted to validate the effectiveness of this sealing method and type of sealant used. [1] To investigate the time of build-up of the progeny of ^{222}Rn , IAEA-434 reference material was sealed with 5 types of sealants (Silicon sealant, Butyl sealant, Modified silicon sealant, Polyurethane sealant, Epoxy adhesive). And 5 samples were periodically measured using a high-purity germanium detector (HPGe). After sufficient build-up time for secular equilibrium between ^{226}Ra and its progenies, the measured ^{214}Pb activity concentration was compared to the Certified reference activity. [2] Radon-rich water was

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prepared into 5 sample containers and sealed with 5 types of sealants. Samples were periodically measured using a HPGe. The theoretical half-life of radon (3.82d) was compared with that of radon calculated through periodic measurements.

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13	Dominic Larivière	Toward the Development of a Cloud Point Extraction System for Cesium
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TOWARD THE DEVELOPMENT OF A CLOUD POINT EXTRACTION SYSTEM FOR CESIUM

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Following nuclear fission of uranium, radiocesium is formed. The environmental release of this radioelement has to be properly monitored. While Cs-137 can be detected at low activities by gamma-spectrometry, Cs-135 detection remains challenging. Several authors have proposed the use of inorganic mass spectrometry such inductively coupled plasma mass spectrometry (ICP-MS) to adequately quantify this isotope[1-3], but the sample preparation required to properly extract and isolate Cs are often time-consuming. Cloud point extraction (CPE), a technique that manipulates temperature and surfactant concentration to move aqueous solutes into a micelle phase for separation. This approach allows for the rapid preparation of the sample and has been shown to be compatible with ICP-MS[4-5]. A new cloud point extraction system for the extraction of cesium is currently in development. In this presentation we will discuss the various strategies that have been explored to render the extraction of Cs by CPE possible and the drawbacks that we encountered.

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2. Magre, A., Boulet, B., Isnard, H., Mialle, S., Evrard, O., & Pourcelot, L. (2023) *Analytical Chemistry*, 95(17), 6923-6930.
3. Asai, S., Ohata, M., Hanzawa, Y., Horita, T., Yomogida, T., & Kitatsuji, Y. (2020) *Analytical chemistry*, 92(4), 3276-3284.
4. Mandal, S., & Lahiri, S. (2022) *Microchemical Journal*, 175, 107150.
5. Basque, J., Lavoie, J., Reynier, N., & Larivière, D. (2023) *Microchemical Journal*, 190, 108724.

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#	Presenter	Presentation Title
14	Annelise Cardon, et al	Improvements to Plutonium Separation Chemistry for In Vitro Bioassay at LANL

IMPROVEMENTS TO PLUTONIUM SEPARATION CHEMISTRY FOR IN VITRO BIOASSAY AT LANL

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The in vitro bioassay program is the foundation of the radiological worker safety program at Los Alamos National Laboratory (LANL), ensuring workplace safeguards are effective against worker exposures to radioactive materials. Though many administrative and engineering controls are put in place to reduce the likelihood of exposure, some duties cannot be performed without risk of an intake. Intakes occur when radionuclides enter the body by breathing, swallowing, skin contact, or through a wound. Certain radionuclides can be detected in vivo with whole-body radiation counters, e.g. Am-241, but determination outside of the body in urine or fecal samples is more sensitive, and allows for the measurement of radionuclides that are difficult or impossible to measure in vivo, e.g. Pu and U isotopes and H-3. LANL's in vitro bioassay program collects urine samples (kits) from at-risk workers which are analyzed by LANL's Nuclear and Radiochemistry Group (C-NR).

Increased hiring of plutonium workers at LANL has resulted in record numbers of Pu kits being collected for analysis by C-NR. To keep up with the increased sample load while ensuring high quality results, we have investigated multiple changes to the Pu chemistry process, including: (1) improving sample-tracer equilibration by more extensive acidification, stirring, and increased wait time before co-precipitation, (2) improving sample digestion by increasing overall solution volume and addition of hydrogen peroxide to organic-rich samples, and (3) optimizing anion-exchange chromatography by changing the column geometry and adding a 9M HCl wash before Pu elution to remove matrix elements (specifically Th).

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15	Annelise Cardon, et al	Traced-Only Uranium Analysis for In Vitro Bioassay at LANL
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TRACED-ONLY URANIUM ANALYSIS FOR IN VITRO BIOASSAY AT LANL

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Increased worker enrollments for Uranium (U) bioassay at Los Alamos National Laboratory (LANL) have necessitated the development of an optimized chemical separation and analysis procedure. Previously, bioassay samples were processed and analyzed in two fractions: the first was a small urine aliquot that was traced with ^{233}U and digested before ICP-MS analysis for U assay; the second was a larger aliquot that was not traced, but went through co-precipitation, digestion, and anion-exchange chromatography before ICP-MS analysis for U isotopic composition. This process was onerous from a chemistry perspective – two fractions of each sample required double the materials and reagents, took up precious hotplate and hood space, and aliquoting each sample twice took a large portion of the overall time needed to perform the chemistry. From the ICP-MS perspective, each sample required two “runs”, doubling the amount of argon and wash solutions used, as well as doubling the time load on the instrument. In addition, and most importantly, the traced fraction does not go through a separation process, so significant amounts of urine matrix went directly into the instrument, causing increased maintenance and down time.

To address these issues and to streamline the overall process, a “traced-only” method was developed. With this method, only one aliquot of urine is taken and traced with ^{233}U before being processed through the U chemical separation procedure (co-precipitation, digestion, anion-exchange). With a single aliquot, both analyses - U assay and isotopic composition - are achieved from a single ICP-MS measurement. To implement this procedure, the major challenges were: (1) Significantly lowering the U procedural blank to approximately 1-5 pg U/mL, so small amounts of ^{234}U (an analyte of interest for bioassay) are not overwhelmed by a large natural U signal; (2) The ^{233}U isotope dilution tracer must be well characterized to quantify the contribution of ^{234}U from the tracer; and (3) Data collection and reduction had to be tailored so the ^{234}U contribution from the tracer is subtracted from the final result with minimal impact on overall ^{234}U uncertainty.

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