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Challenges in Bulk Nuclear Forensics Sample Analysis

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Introduction - Los Alamos National Laboratory

- Over 70 years of actinide nuclear science
- **Mission Statement**

  To solve national security challenges through scientific excellence.
- **Continuing Mission**

  Develop and apply science and technology to ensure the **safety and reliability** of the United States nuclear deterrent; reduce the threat of weapons of mass destruction, proliferation, and terrorism; and solve national problems regarding defense, energy, environment, and infrastructure.

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Introduction - Los Alamos National Laboratory

Continuing Mission - Operational Requirements

- Nuclear Facilities
  - safe handling of actinide materials
  - actinide material processing capabilities
- Analytical Chemistry
  - actinide analysis capabilities
  - quality assurance program
- Quality Assurance Program
  - actinide product certification standard

Introduction - Los Alamos National Laboratory

Continuing Mission – Capability Requirements

- alpha spectrometry
- colorimetry
- controlled potential coulometry
- gamma spectrometry
- gravimetry
- combustion - infrared spectroscopy
- ion chromatography
- neutron counting
- thermal ionization mass spectrometry
- titrimetry
- icp - mass spectrometry
- icp - atomic emission spectroscopy
- x-ray fluorescence

Davies & Gray titrimetry

Capability Application – Actinide Product Certification

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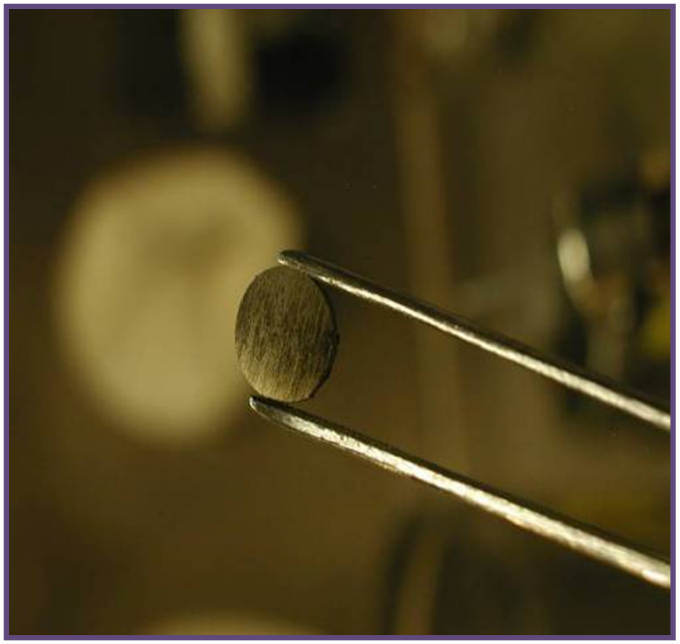
Introduction - Los Alamos National Laboratory

Continuing Mission – Analytical Chemistry Samples

Plutonium Metal
pure
impure
heat source
power source
precursors

Plutonium Oxide
pure
impure
MOX fuels
heat source

Neutron Source
PuBe



Uranium Oxide
pure (EU)
targets (NU)
MOX fuels (EU)
reactor fuel (EU)
metal precursor (EU)
ore concentrate (NU)

Uranium Metal
pure (EU)
targets (NU)

Uranium Fluoride
tetrafluoride
hexafluoride

Introduction – Analytical Chemistry

Continuing Mission – Quality Assurance Standards

QC-1

Implements quality assurance requirements for domestic nuclear industry contractors performing work in relevant product life-cycles. Institutional quality requirements are implemented through Manufacturing Administrative Procedures

NQA-1

Implements quality assurance requirements for nuclear facility applications including design and operation.

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Nuclear Forensics - Analytical Chemistry

Nuclear Forensics Mission - Operational Requirements

- Nuclear Facilities
  - safe handling of actinide materials
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- Analytical Chemistry
  - actinide analysis capabilities
  - quality assurance program

Nuclear Forensics - Analytical Chemistry

Nuclear Forensics Mission – Capability Requirements

- alpha spectrometry
- colorimetry
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- gamma spectrometry
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- thermal ionization mass spectrometry
- titrimetry
- icp - mass spectrometry
- icp - atomic emission spectroscopy
- x-ray fluorescence

- density
- particle size analysis
- optical microscopy
- secondary electron microscopy
- x - ray diffractometry
- x - ray radiography

thermal ionization mass spectrometry

Capability Application – Actinide Sample Analysis

Nuclear Forensics - Analytical Chemistry

Nuclear Forensics Mission – Quality Assurance Standard

ISO/IEC 17025:2005

General Requirements for the Competence of Testing and Calibration Laboratories

ISO/IEC 17025:2005 specifies the general requirements for the **competence to carry out tests** and/or calibrations, including sampling. It covers testing and calibration performed using standard methods, non-standard methods, and laboratory-developed methods.

It is applicable to all **organizations performing tests** and/or calibrations.

Nuclear Forensics - Analytical Chemistry

New Mission – Quality Assurance Requirements

ISO-17025 Procedures

- NF-QA-001, *Nuclear Forensics Analysis Results Review, Approval and Release*
- NF-QA-003, *Calculation of Uncertainties*
- NF-QA-006, *Root Cause Analysis*
- NF-QA-007, *Management Review*
- NF-QA-008, *Review of Requests, Tenders, and Contracts*
- NF-QA-009, *Nuclear Forensics Case Notebook*

Nuclear Forensics - Analytical Chemistry

New Mission – Quality Assurance Standard

ISO-17025 Customer Interaction

- NF-QA-008, *Review of Requests, Tenders, and Contracts*

Name of Customer:_____ Date of Request/Tender/Contract: _____

Written or Oral? _____ Reference Number: _____

Are the **customer requirements** adequately defined, documented, and understood?

Can listed customer requirements be addressed by existing **capabilities** and **resources**?

Do personnel/equipment have the **skills** and **expertise** necessary to perform the tests?

Have appropriate **test methods** been selected to meet the customer requirements?

If any of the above are not met, consult with the customer to resolve, or if not possible, turn down the request, tender, or contract.

List any issues/concerns which need to be addressed prior to acceptance:

Nuclear Forensics - Analytical Chemistry

New Mission – Quality Assurance Requirements

ISO-17025 Quality Testing

- NF-PLAN-006 *LANL Actinide Analytical Chemistry Proficiency Testing (PT) Plan*

The laboratory policy is to run **proficiency test samples** (PTs) where **applicable** and when PTs are **available**. Applicability is based on the analyte/nuclide, available matrix, and concentration range of the PT.

When a PT is not available, appropriate **quality control standards** will be analyzed to ensure the accuracy and precision for quantitative methods. For the qualitative identification, other appropriate schemes within the laboratory test procedures will be implemented.

Nuclear Forensics - Analytical Chemistry

New Mission – Quality Assurance Requirements

ISO-17025 Quality Testing

- NF-PLAN-006 *LANL Actinide Analytical Chemistry Proficiency Testing (PT) Plan*

LANL analytical chemistry has one accreditation discipline, chemistry, with two sub disciplines: general or wet chemistry and spectroscopy. Note there are no ISO/IEC 17043 accredited domestic (U.S.) PT Programs for the bulk **nuclear fuel** or **nuclear material programs**.

Nuclear Forensics - Analytical Chemistry

New Mission – Quality Assurance Requirements

ISO-17025 Quality Testing

- Applicable Proficiency Test Programs Would Require…

1. Appropriate nuclear (Pu and U) materials for distribution
   - Certified Reference Materials
   - Working Reference Materials
   - Viable Control Materials
2. Accurate and precise measured reference values for materials
3. GUM compliant reference value uncertainties
4. Accredited Proficiency Test program administrator
5. Infrastructure and skill to package and ship bulk quantities of nuclear materials

Nuclear Forensics - Analytical Chemistry

New Mission – Quality Assurance Requirements

ISO-17025 Quality Testing Options

~~1. Proficiency Test Programs~~

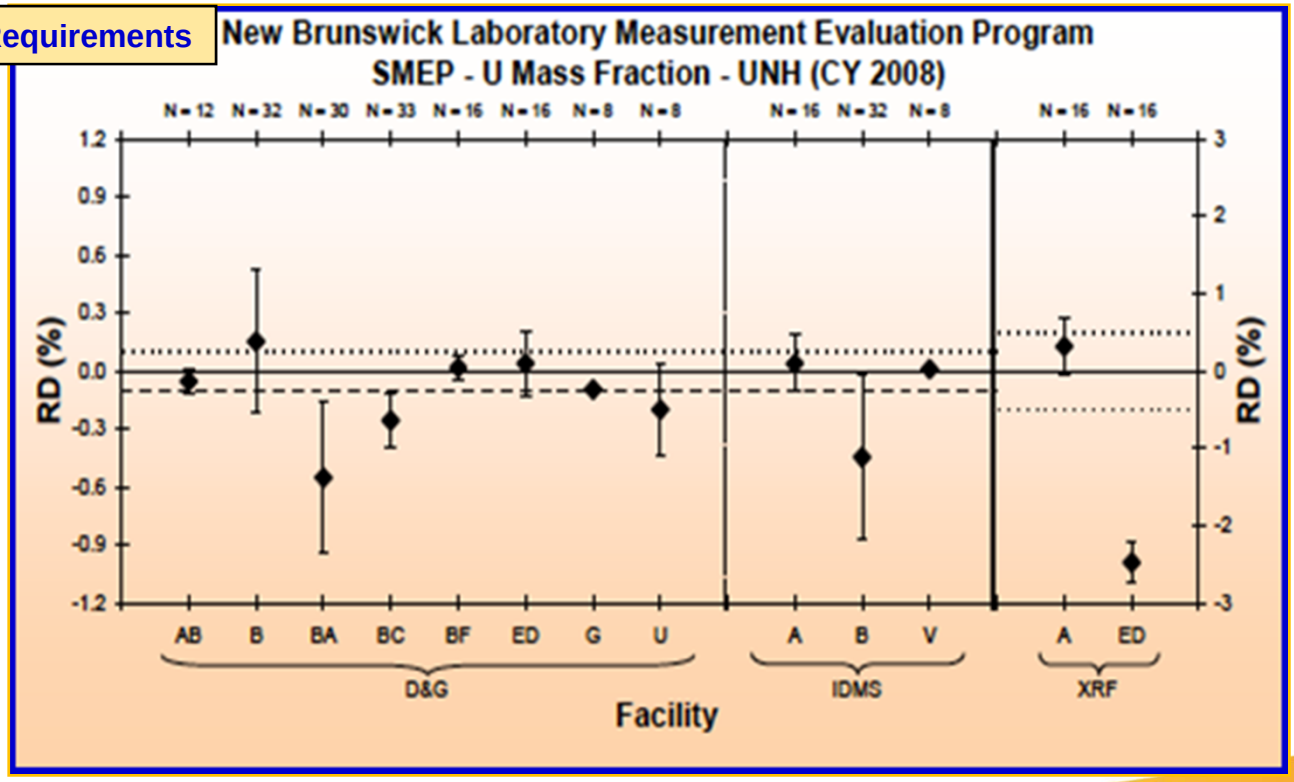
2. Round-Robin Nuclear Material Exchanges

3. Measurement Quality Control Samples

Nuclear Forensics – Reference Materials

- **New Mission – Quality Assurance**
  Round-Robin Uranium Material Exchange
  - Safeguards Measurement Evaluation (SME) Program - NBL

ITV Requirements



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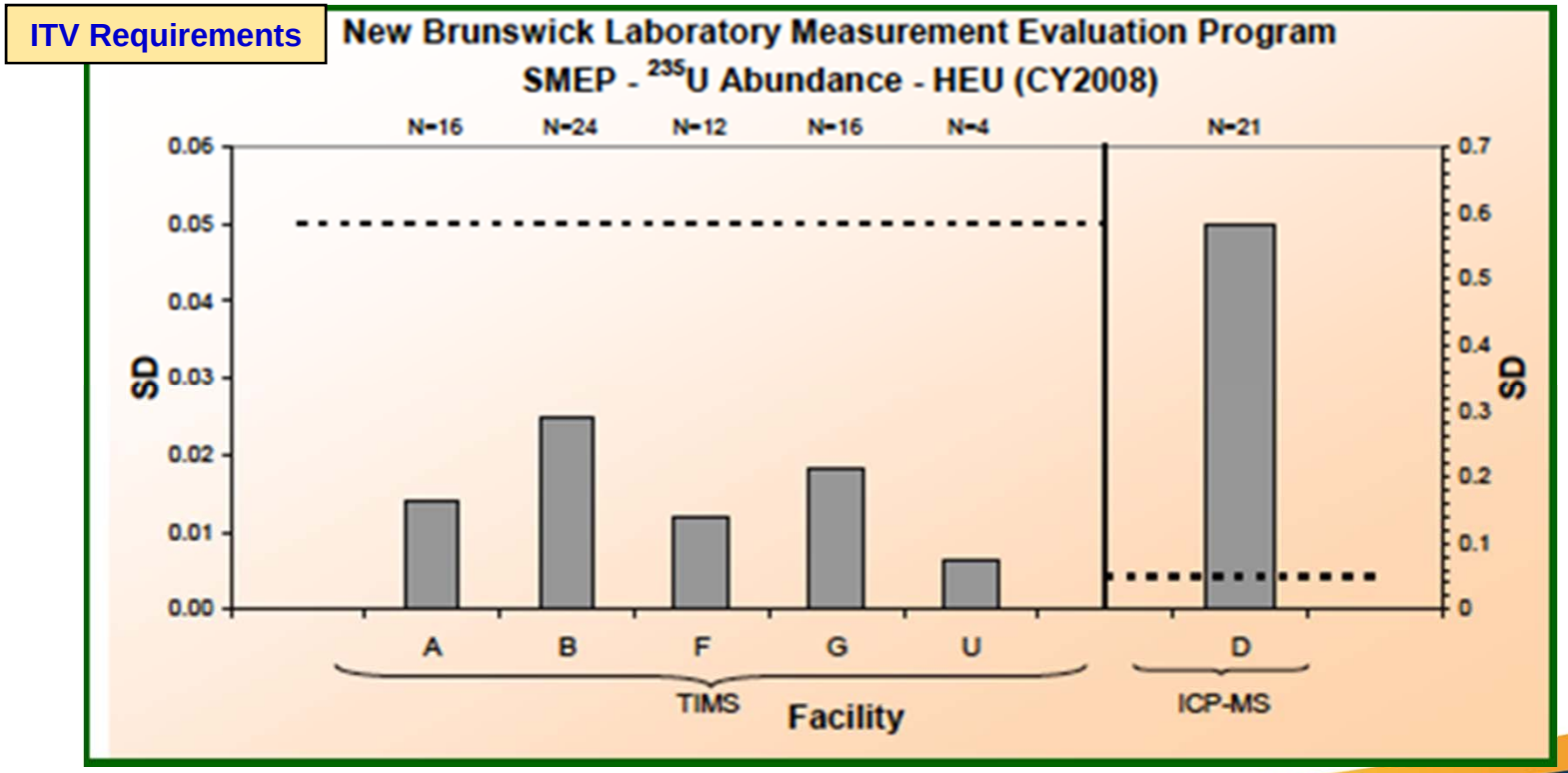
Nuclear Forensics – Reference Materials

New Mission – Quality Assurance

Round-Robin Uranium Material Exchange

- Safeguards Measurement Evaluation (SME) Program - NBL



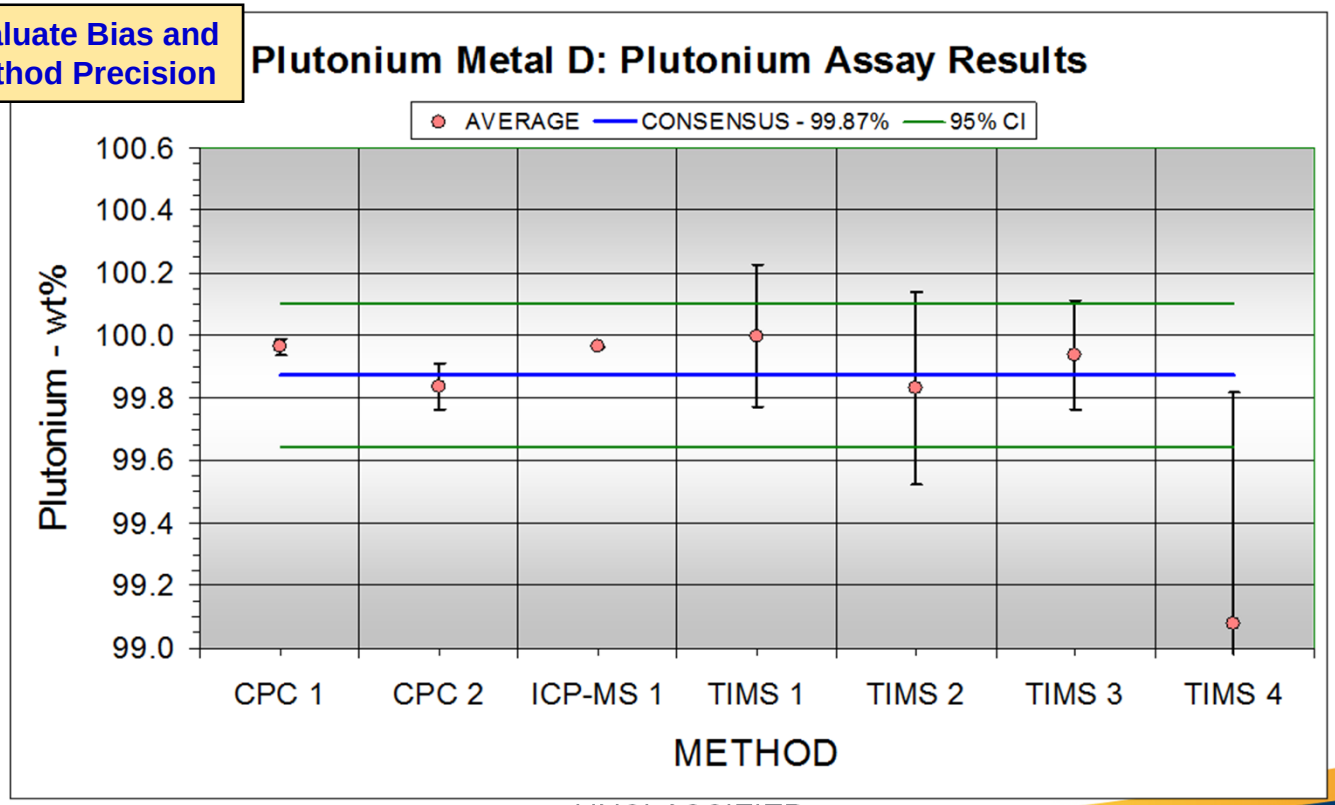
Nuclear Forensics – Reference Materials

New Mission – Quality Assurance

Round-Robin Plutonium Material Exchange

- Plutonium Standards Metal Exchange Program - LANL

Evaluate Bias and Method Precision



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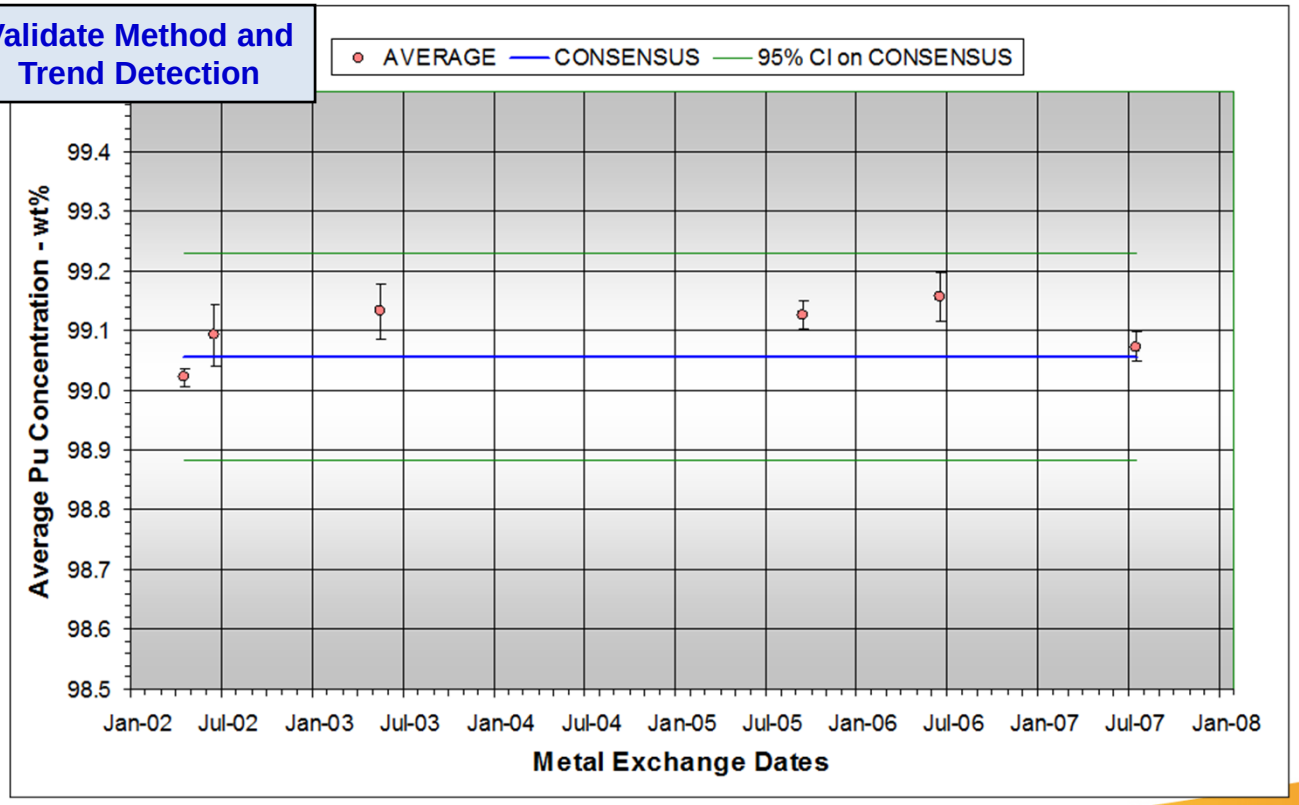
Nuclear Forensics – Reference Materials

New Mission – Quality Assurance

Round-Robin Plutonium Material Exchange

- Plutonium Standards Metal Exchange Program - LANL

Validate Method and Trend Detection



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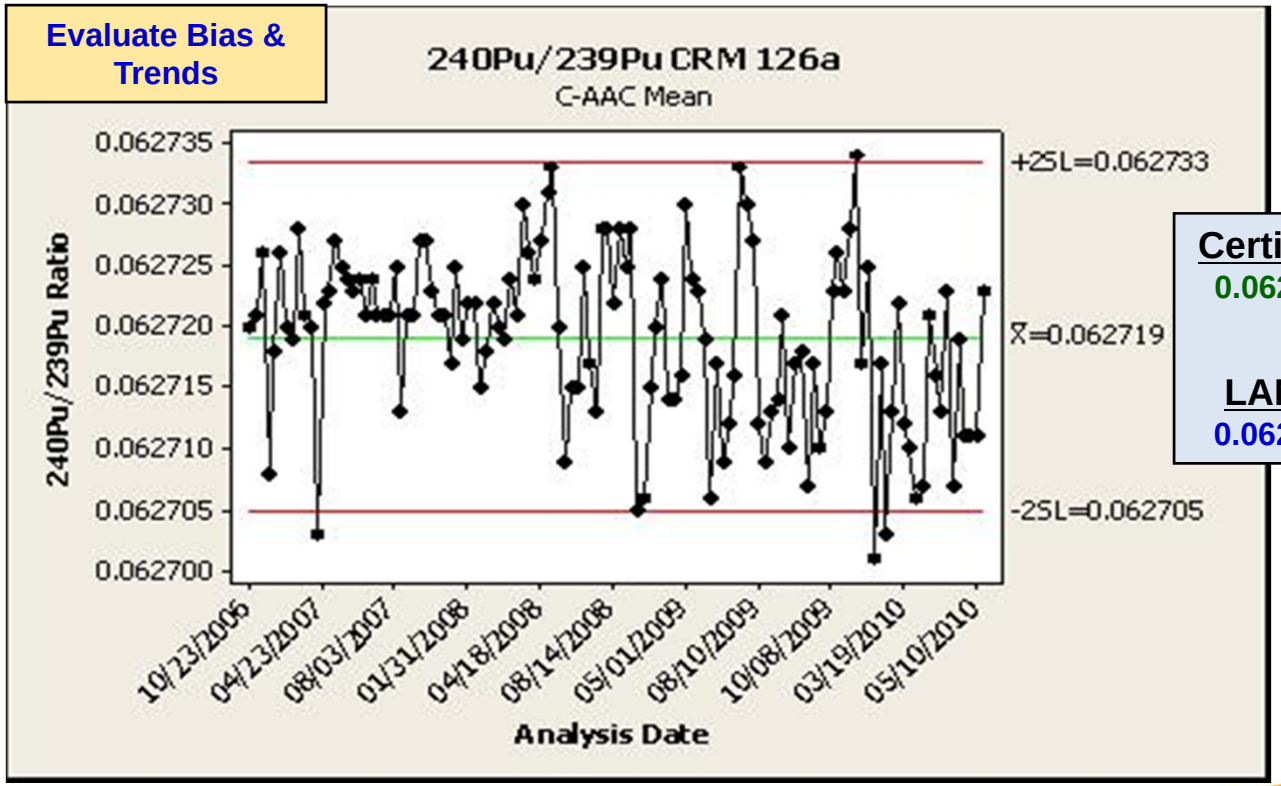
Nuclear Forensics – Reference Materials

New Mission – Quality Assurance

Measurement Quality Control Sample

- Plutonium Standards Metal Exchange Program - LANL

Evaluate Bias & Trends



Certified Atom Ratio

0.062711 ± 0.000016*

** Decay corrected*

LANL Atom Ratio

0.062719 (%RD = -0.013)

Nuclear Forensics - Analytical Chemistry

New Mission – Quality Assurance Requirements

ISO-17025 Uncertainties

- NF-QA-003, *Calculation of Uncertainties*

There are various methods of calculating uncertainties, any of which may be used. If requested by customer, the uncertainty calculation may be performed through the use of **GUM** compliant methods.

GUM
Guide to the Expression of Uncertainty in Measurement (International Organization for Standardization, 1995)

Nuclear Forensics - Analytical Chemistry

New Mission – Quality Assurance Requirements

ISO-17025 Uncertainties

- NF-QA-003, *Calculation of Uncertainties*

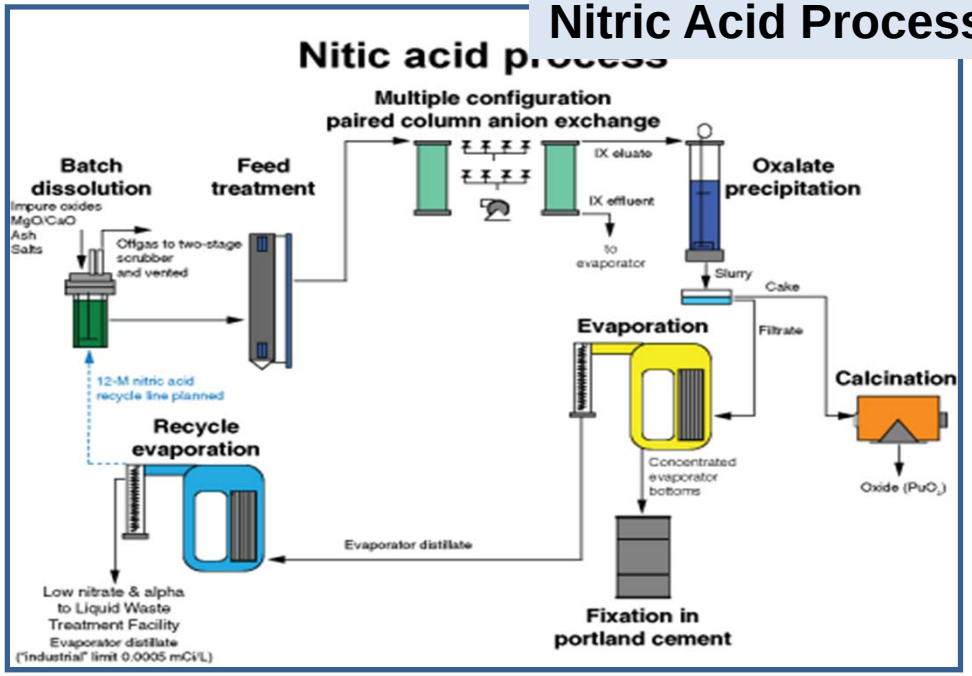
Measurement	Result	n	Replicate Standard Dev. (n=12)	Method Uncertainty (k=2)	GUM Uncertainty (k=2)
Pu Assay	87.60 wt%	12	0.07 wt%	0.08 wt%	0.08 wt%
^{238}Pu content	0.0120 at%	12	0.0003 at%	0.0004 at%	0.0009 at%
^{239}Pu content	93.9645 at%	12	0.0008 at%	0.0007 at%	0.0021 at%

Nuclear Forensics – Reference Materials

- **New Mission – Quality Assurance**

New Reference Material Production – Pu Oxide via Recycle

Nitric Acid Process



Nuclear Forensics – Reference Materials

New Mission – Quality Assurance

New Reference Material Production – Pu Oxide to RM

Nuclear Material Production ● Production Records ● Packaging ● Shipping (Send/Receive) ● Stabilization ● Homogenization ● Blending ● Material Analysis

Nuclear Forensics – Reference Materials

New Mission – Quality Assurance

New Reference Material Production – Sample Analysis

	CRM	WRM
Form	Pu oxide	Pu oxide
Sample Size	160-200 mg	160-200 mg
Attributes	Pu Isotopics (238-244)	Pu Isotopics (238-244)
	Pu assay	Pu assay
	U assay and isotopics	U assay and isotopics
	Am assay (isotopics if appropriate)	Am assay (isotopics if appropriate)
	Np assay	Np assay

Conclusions

- Los Alamos National Laboratory operates capable analytical chemistry and material science laboratories suitable for nuclear material forensic measurements
- LANL analytical chemistry has numerous ISO 17025 accredited measurement processes to support nuclear forensic customers
- LANL analytical chemistry uses numerous means to validate and independently verify that ISO17025 measurement data quality objectives are met

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