Looking Forward through the Past: Status of the United States Nuclear Safeguards Reference Material Program at New Brunswick Laboratory

Jon W. Neuhoff, Director
New Brunswick Laboratory

November 24, 2010
New Brunswick Laboratory – Mission and Programs

- NBL is a U.S. Department of Energy (DOE) laboratory with an enduring mission to improve, build confidence, and strengthen the effectiveness and efficiency of nuclear material measurements in support of DOE and national needs.

- To achieve our mission, we provide unique, metrological science and technology in:
  - Reference Materials
  - Measurement Evaluation
  - Measurement Development
  - Measurement Services
  - Nuclear Forensics
  - Safeguards, Nonproliferation, and National Security Assistance
Laboratory at a Glance

- **Location**
  - New Brunswick, New Jersey (1949-1977)
  - Argonne, Illinois (1977-present)
- **Government-Owned, Government-Operated (GOGO) Laboratory**
- **About 8,400 m² of laboratory space**
- **42 resident staff members**
  - 30 Federal employees
  - 10 contractors
  - 2 post-doctoral appointments
- **11 project-specific consultants**

- **U.S. Government’s Certifying Authority for nuclear reference material**
- **Department of Energy Center of Excellence in analytical chemistry, metrology, and nuclear material measurements**
- **Network Laboratory of the International Atomic Energy Agency (reference materials)**
Early NBL History (New Jersey)

- **1949** - Established in New Brunswick, New Jersey as the official government assay laboratory for quality control of uranium ores, concentrates, and compounds
- **1950** – Staff prepared the first comprehensive compilation of critically evaluated methods for the analysis of nuclear materials
- **1952-1984** - Established and administered the General Analytical Evaluation Program for uranium materials representative of fuel production
- **1953** – Characterized the first uranium oxide material for calibration of uranium assay measurements
- **1957** – Nuclear material standards program established in which NBL conducted the analytical efforts leading to the development of primary reference standards
- **1958** – NBL supplied the first uranium metal samples for use as standard reference material
- **1959** – Plutonium analytical capabilities added
- **1959, 1969** – NBL developed and reported reliable and rapid methods for the assay of uranium and plutonium materials
- **1963-1967** – NBL prepared and characterized the first plutonium compounds for use as standard reference materials for plutonium assay and isotopic measurements
Recent NBL History (Illinois)

- **1975-1977** – Relocated to Argonne, IL
- **1976-1984** – Administered the Safeguards Analytical Laboratory Evaluation (SALE) Program, which provided the largest known critically evaluated base of nuclear material measurement data from laboratories throughout the world
- **1981** – NBL assumed full responsibility for the U.S. nuclear reference material program
- **1984** – Completed design and fabrication of a markedly improved coulometer for plutonium assay measurements
- **1984** – Safeguards Measurement Evaluation Program started and incorporates all previous interlaboratory comparison exercises
- **1996** – Assumed responsibility for the Calorimetry Exchange Program
- **Present** – Continuing historical core missions with nonproliferation, international safeguards, nuclear forensics, reference materials, and measurement evaluation support activities significantly expanding
United States Nuclear Measurement System

SI System

U.S. National System of Physical & Chemical Measurements

Definitive Methods

Certified Reference Materials

NBL: CRMs for Chemical Composition, Isotopic Composition, NDA

NIST: SRMs for Radioactivity, Oxidimetry, Reductometry

Consensus Standard Methods

Reference Methods

Measurement /Method Evaluation, Safeguards (MC&A), QA

Working Reference Material

Voluntary Consensus Standards Organizations: ASTM, ANSI, etc.

Field Methods
NBL Reference Materials Program

- NBL is the only facility in the U.S. (U.S. Government’s Certifying Authority) responsible for preparing, certifying, and disseminating nuclear Certified Reference Materials (CRMs)
- Supports the nuclear fuel/weapons cycle (mining, milling, conversion, enrichment, fuel/weapon fabrication)
- NBL’s suite of reference materials and samples dates from 1944 – present
- Every significant uranium, thorium, or plutonium measurement (assay or isotopic) in the U.S. is traceable to an NBL standard (primarily for safeguards purposes)
Demand for NBL CRMs is Rising Steadily
NBL CRM Usage – Worldwide 2000-2010
NBL CRM Usage – U.S. 2000-2010
Uranium and Thorium Ore CRMs

<table>
<thead>
<tr>
<th>CRM</th>
<th>Type</th>
<th>Description</th>
<th>Size/Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>101-A</td>
<td>Assay</td>
<td>Pitchblende Ore-Silica Mixture (1% U)</td>
<td>50g</td>
</tr>
<tr>
<td>102-A</td>
<td>Assay</td>
<td>Pitchblende Ore-Silica Mixture (0.1% U)</td>
<td>50g</td>
</tr>
<tr>
<td>103-A</td>
<td>Assay</td>
<td>Pitchblende Ore-Silica Mixture (0.05% U)</td>
<td>50g</td>
</tr>
<tr>
<td>104-A</td>
<td>Assay</td>
<td>Pitchblende Ore-Silica Mixture (0.01% U)</td>
<td>50g</td>
</tr>
<tr>
<td>105-A</td>
<td>Assay</td>
<td>Pitchblende Ore-Silica Mixture (0.001% U)</td>
<td>50g</td>
</tr>
<tr>
<td>106-A</td>
<td>Assay</td>
<td>Monazite Sand-Silica Mixture (1% Th)</td>
<td>50g</td>
</tr>
<tr>
<td>107-A</td>
<td>Assay</td>
<td>Monazite Sand-Silica Mixture (0.1% Th)</td>
<td>50g</td>
</tr>
<tr>
<td>108-A</td>
<td>Assay</td>
<td>Monazite Sand-Silica Mixture (0.05% Th)</td>
<td>50g</td>
</tr>
<tr>
<td>109-A</td>
<td>Assay</td>
<td>Monazite Sand-Silica Mixture (0.01% Th)</td>
<td>50g</td>
</tr>
<tr>
<td>110-A</td>
<td>Assay</td>
<td>Monazite Sand-Silica Mixture (0.001% Th)</td>
<td>50g</td>
</tr>
</tbody>
</table>
## Uranium and Thorium Ore and Impurity CRMs

<table>
<thead>
<tr>
<th>CRM</th>
<th>Type</th>
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</tr>
</thead>
<tbody>
<tr>
<td>1-A</td>
<td>Assay</td>
<td>Phosphate Rock Ore (0.015% U)</td>
<td>50g</td>
</tr>
<tr>
<td>4</td>
<td>Assay</td>
<td>Carnotite Ore (0.18% U3O8)</td>
<td>100g</td>
</tr>
<tr>
<td>5</td>
<td>Assay</td>
<td>Carnotite Ore (0.11% U3O8)</td>
<td>100g</td>
</tr>
<tr>
<td>42-A (1-4)</td>
<td>Assay</td>
<td>Pitchblende-Dunite Mixture (0.5, 1, 2 and 4% g U/g)</td>
<td>4x100g</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>CRM</th>
<th>Type</th>
<th>Description</th>
<th>Size/Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>66 (1-7)</td>
<td>Elemental</td>
<td>Thorium Oxide ThO₂ 22 Element Impurity</td>
<td>7x25g</td>
</tr>
<tr>
<td>123 (1-7)</td>
<td>Elemental</td>
<td>Uranium (Normal) Oxide U3O8 18 Element Impurity</td>
<td>7x25g</td>
</tr>
<tr>
<td>124 (1-7)</td>
<td>Elemental</td>
<td>Uranium (Normal) Oxide U3O8 24 Element Impurity</td>
<td>7x25g</td>
</tr>
</tbody>
</table>
## Uranium Assay/Isotopic CRMs

<table>
<thead>
<tr>
<th>CRM</th>
<th>Type</th>
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<th>Size/Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>111-A</td>
<td>Assay/Isotopic</td>
<td>Uranium-233 Spike, Nitrate Solution (99.49% U-233)</td>
<td>0.005g U</td>
</tr>
<tr>
<td>112-A</td>
<td>Assay</td>
<td>Uranium (Normal) Metal (0.99975 g U/g)</td>
<td>4g</td>
</tr>
<tr>
<td>113-B</td>
<td>Assay/Isotopic</td>
<td>Uranium Hexafluoride (4.5% U-235)</td>
<td>1700g</td>
</tr>
<tr>
<td>115</td>
<td>Assay</td>
<td>Uranium (Depleted) Metal (0.99977 g U/g)</td>
<td>75g</td>
</tr>
<tr>
<td>125-A</td>
<td>Assay/Isotopic</td>
<td>Uranium (Enriched) Oxide UO2 (88% U; 4% U-235)</td>
<td>5.4g</td>
</tr>
<tr>
<td>129-A</td>
<td>Assay/Isotopic</td>
<td>Uranium Oxide U3O8 (0.847 gU/g; 0.721% U-235)</td>
<td>25g</td>
</tr>
<tr>
<td>145</td>
<td>Assay</td>
<td>Uranium (Normal) Assay, Nitrate Solution (10 mg U/g)</td>
<td>0.2g U</td>
</tr>
<tr>
<td>145-B</td>
<td>Assay</td>
<td>High-Purity Uranium Assay Solution (10 mg U/g)</td>
<td>0.2g U</td>
</tr>
<tr>
<td>146</td>
<td>Assay/Isotopic</td>
<td>Uranium Gamma Spectrometry Set - HEU (20%, 53%, 93% U-235)</td>
<td>230g</td>
</tr>
<tr>
<td>149</td>
<td>Assay/Isotopic</td>
<td>U3O8 Standard Set For Neutron Counting (93% U-235)</td>
<td>0.5, 1, 1.5, 2, 3, 4kg U</td>
</tr>
<tr>
<td>969</td>
<td>Assay/Isotopic</td>
<td>Uranium Gamma Spectrometry Set – Depleted/LEU (0.3%, 0.7%, 1.9%, 2.9%, 4.5% U-235)</td>
<td>200g</td>
</tr>
</tbody>
</table>
CRM 969 – Uranium Isotopic Standard for Gamma Spectrometry Measurements
CRM 969 – Uranium Isotopic Standard for Gamma Spectrometry Measurements

- Referred to as EC-NRM-171; NBS-SRM-969; CRM 969
- International project between NBL, NIST, JRC-IRMM, JRC-Ispra, IAEA, Euratom, and the ESARDA NDA Working Group
- First example of an internationally-certified reference material for NDA, with the parameter of interest traceable to NBL, IRMM, and the basic SI units
### Uranium Isotopic CRMs – Depleted/LEU

<table>
<thead>
<tr>
<th>CRM</th>
<th>Type</th>
<th>Description</th>
<th>Size/Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>U0002</td>
<td>Isotopic</td>
<td>Uranium (Depleted) Isotopic-U3O8, 0.02%</td>
<td>1g U</td>
</tr>
<tr>
<td>U005-A</td>
<td>Isotopic</td>
<td>Uranium (Depleted)-U3O8, 0.5%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U010</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 1%</td>
<td>5mg U</td>
</tr>
<tr>
<td>U015</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 1.5%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U020-A</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 2.0%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U030-A</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 3.0%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U045</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 4.5%</td>
<td>5mg U</td>
</tr>
<tr>
<td>U100</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 10%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U150</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 15%</td>
<td>10mg U</td>
</tr>
</tbody>
</table>
### Uranium Isotopic CRMs – HEU

<table>
<thead>
<tr>
<th>CRM</th>
<th>Type</th>
<th>Description</th>
<th>Size/Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>U200</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 20%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U350</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 35%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U500</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 50%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U630</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 63%</td>
<td>1g U</td>
</tr>
<tr>
<td>U750</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 75%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U800</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 80%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U850</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 85%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U900</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 90%</td>
<td>10mg U</td>
</tr>
<tr>
<td>U930D</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 93%</td>
<td>5mg U</td>
</tr>
<tr>
<td>U970</td>
<td>Isotopic</td>
<td>Uranium (Enriched) Isotopic-U3O8, 97%</td>
<td>10mg U</td>
</tr>
</tbody>
</table>
NBL Plutonium CRMs

- NBL has conducted plutonium measurements since 1959
  - Developed analytical methods
  - Supported fuels research
  - Produced counting and other standards from pure Pu
- NBL has prepared the primary plutonium metal assay standards in coordination with LANL
  - SRM 949 (1962)  SRM 949d (1972)
  - SRM 949a (1964)  SRM 949e (1975)
  - SRM 949c (1969)  SRM 949g
## Plutonium Assay and Isotopic CRMs

<table>
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<tr>
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</tr>
</thead>
<tbody>
<tr>
<td>122</td>
<td>Assay/Isotopic</td>
<td>Plutonium Oxide PuO₂ (87.79% Pu; 87% Pu-239)</td>
<td>1g</td>
</tr>
<tr>
<td>126-A</td>
<td>Assay/Isotopic</td>
<td>Plutonium Metal Standard (99.96% g Pu/g metal; 94% Pu-239)</td>
<td>1g Pu</td>
</tr>
<tr>
<td>128</td>
<td>Isotopic</td>
<td>Plutonium 239:242 Isotopic, Nitrate 1:1 Mixture (0.99937 Pu-239/Pu-242) – Equal Atom Spike</td>
<td>0.001g Pu</td>
</tr>
<tr>
<td>130</td>
<td>Assay/Isotopic</td>
<td>Plutonium-242 Spike, Nitrate (99.95% Pu-242)</td>
<td>0.001g Pu</td>
</tr>
<tr>
<td>131</td>
<td>Assay/Isotopic</td>
<td>Plutonium-244 Spike, Nitrate (97.895% Pu-244)</td>
<td>0.001g Pu</td>
</tr>
<tr>
<td>136</td>
<td>Isotopic</td>
<td>Plutonium Isotopic, Sulfate Tetrahydrate, 12% Pu-240</td>
<td>250mg Pu</td>
</tr>
<tr>
<td>137</td>
<td>Isotopic</td>
<td>Plutonium Isotopic, Sulfate Tetrahydrate, 18% Pu-240</td>
<td>250mg Pu</td>
</tr>
<tr>
<td>138</td>
<td>Isotopic</td>
<td>Plutonium Isotopic, Sulfate Tetrahydrate, 8% Pu-240</td>
<td>250mg Pu</td>
</tr>
</tbody>
</table>
CALEX I and II Standards

- NBL took over the Calorimetry Exchange Program from Mound in 1996
- Calorimetry Exchange (CALEX) standards are suitable for quality control of gamma spectrometric and calorimetric measurements; investigating suitability for neutron measurements
- CALEX I – not a CRM, but a well-characterized RM
  - Each exchange sample contains about 455 g of PuO$_2$, giving about 400 g of Pu
  - Heat output is about 1 watt
  - Pu-240 content is about 6% of the total Pu
- CALEX-II - CRM
  - Each exchange standard contains about 2kg of PuO$_2$
  - Heat output is about 6 watts
  - Pu-240 content is about 12% of the total Pu
In late 2004, DOE-Washington performed an inspection
- Inspection report identified deficiencies
  - Nuclear safety documentation ("operating license")
  - Fire suppression systems
  - Safety management programs
- NBL stopped all operations and shipping to address
- DOE-Washington allowed limited mission-critical activities to go forward (2005 – 2008) while corrective actions were being implemented
- "Nuclear footprint" reduction required in 2008
  - Successfully and significantly reduced the amount of nuclear material at NBL – mostly legacy/waste materials
- Uranium operations re-commenced in 2009 and are being conducted under a temporary "operating permit"; plutonium areas in process of being upgraded
- Updated and submitted a new "operating license" for approval in September, 2010 – describes controls for the smaller "nuclear footprint" area
- Full operations planned in mid 2011
Nuclear Material Reduction

88% reduction in Plutonium

Plutonium Inventory Reduction

Year

Grams

NBL Fire Protection Upgrade Project

- **Legacy Ductwork Removal (Jun 4 – Aug 31)**
  - *Completed* removal/sealing of legacy ductwork in the Plutonium (Pu) Wing
  - Purpose was to clear the area to construct a fire barrier and reduce nuclear material holdup

- **Fire Protection Upgrades (Jun 2010 – Mar 2011)**
  - 100% complete design delivered November 12
  - Physical construction work to begin in Jan 2011 and complete in Mar 2011
    - Fire separation of the Pu Wing Vault and Fan Loft Area, including nuclear-grade penetration sealing
    - Sprinkler system upgrades in both the Uranium (U) and Plutonium Wings
    - Fire separation in the U Wing
NBL Plutonium Operational Restart

- NBL has conducted plutonium measurements since 1959
- NBL has prepared the world’s primary plutonium standards in coordination with LANL since the 1960’s
  - Used for safeguards measurements and nuclear weapons stockpile work
- NBL took over the Calorimetry Exchange Program from Mound in 1996
- As part of the Fire Protection/Nuclear Safety Upgrade Project, plutonium storage and analytical areas are being upgraded
- Full operations planned in 2011
Looking Forward…

- Next Generation Safeguards Initiative (NGSI) and Materials Protection Accounting and Control for Transmutation (MPACT)
  - NGSI was launched in 2008 to strengthen U.S. domestic policies, technologies, and expertise related to the application of safeguards while also strengthening the international infrastructure to support the international safeguards system as it evolves over the next 25 years.
  - MPACT is developing innovative technologies and analysis tools to enable next-generation nuclear materials management for future U.S. nuclear energy systems.
  - NBL plans to play a major role in NGSI and MPACT in terms of training, assistance, testing technologies, reference materials, measurement evaluation, and measurement development.
Looking Forward...

- With the nuclear renaissance and further adoption of nuclear energy as a clean energy source, the need for reference materials, measurement evaluation, and related services from NBL is expected to significantly grow in terms of customers and materials.
- Increasing efforts as a training center for destructive analytical measurements, reference materials production, and related areas.
- Significant expansion in the uranium ore mining, processing, conversion, and recovery facilities - increased reference materials.
- Growth in uranium enrichment services - increased reference materials and measurements.
Future NBL CRMs - Uranium

- CRM 112-B (Uranium (Normal) Metal Assay and Isotopic Standard)
- CRM 115-A (Uranium (Depleted) Metal Assay and Isotopic Standard)
- CRM 116-A (Enriched Uranium Metal Standard, 93% Enriched)
- UO$_2$ fuel pellet standard
- Y-12 NDA standards
- CRM U0002-A (Uranium Isotopic Standard, 0.2% Enriched)
- CRM U005-B (Uranium Isotopic Standard, 0.5% Enriched)
- CRM U010-A (Uranium Isotopic Standard, 1% Enriched)
- CRM 17-B (Normal Uranium Tetrafluoride Assay Standard)
- Uranium Isotope Calibration Mixes (U-233, U-234, U-235, U-236, U-238) – various mixtures in 1:1
- Uranium impurity standards
Future NBL CRMs – Plutonium

- CRM 122-A (Plutonium Oxide Assay and Isotopic Standard)
- New CRM made from CRM 126-A
  - Solutions or dried nitrates of 10-100 mg sample sizes
- CRM 136-A (Plutonium Isotopic Standard) – 1 mg dried nitrate
- CRM 137-A (Plutonium Isotopic Standard) – 1mg dried nitrate
- CRM 138-A (Plutonium Isotopic Standard) – 1 mg dried nitrate
- CRMs 140-142 (Plutonium Isotopic Spikes) – Pu-240, Pu-242, Pu-244
- CRM 143 (Plutonium Double Atom Spike) – Pu-242/Pu-244
- CRM 144 (Plutonium Triple Atom Spike) – Pu-240/Pu-242/Pu-244
  - 2 mg dried nitrate
- Plutonium Impurity Standards – Pu metal matrix containing metallic/non-metallic impurities
- Plutonium NDA Standards – Weapons grade and reactor grade Pu standards in metal and oxide form; high burn-up; 3013
Future NBL CRMs - Other

- Mixed Oxide (MOX) Standard (U and Pu assay and isotopics with five levels of impurities)
- CRM 66 (Thorium Oxide Impurity Standard)
  - Mostly Th-232 with 22 impurities
- Np standards/spikes (Np-236, Np-237)
- Am standards/spikes (Am-241, Am-243)
- Continue to prepare smaller quantities for ease of shipment/use
- Nuclear forensics – discussed in R. Essex presentation
Summary and Conclusions

- NBL is a U.S. Department of Energy (DOE) laboratory with an enduring mission to improve, build confidence, and strengthen the effectiveness and efficiency of nuclear material measurements in support of DOE and national needs.
- NBL is the only facility in the U.S. (U.S. Government’s Certifying Authority) responsible for preparing, certifying, and disseminating nuclear Certified Reference Materials (CRMs).
- NBL will be fully operational in 2011 under a new “operating license.”
- NGSI, MPACT, new facilities, and the overall nuclear renaissance will require increased reference materials and safeguards measurements.
- New NBL reference materials planned and will come out on an increasing frequency once at full operations.
Congratulations IRMM on Your 50\textsuperscript{th} Anniversary

On behalf of New Brunswick Laboratory, I congratulate you on your 50\textsuperscript{th} Anniversary. Your accomplishments to the field have been noteworthy and you continue to serve an important role. It has been a pleasure working with our colleagues at IRMM throughout the years and we hope for many fruitful years of cooperation and collaboration ahead.

Jon W. Neuhoff, Director
New Brunswick Laboratory

November 24, 2010