#### NTNF CRM Working Group

Activities of the National Technical Nuclear Forensics (NTNF) Certified Reference Material (CRM) Working Group

5<sup>th</sup> Workshop on Isotope Federal Supply and Demand

November 9, 2016

DHS Domestic Nuclear Detection Office







#### What is National Technical Nuclear Forensics?

- <u>Definition</u>: Nuclear forensics is the collection, analysis, and evaluation of predetonation (intact) and post-detonation (exploded) radiological or nuclear (RN) materials, RN devices, and debris, as well as the immediate effects created by a nuclear detonation.
- NTNF is the USG program to develop, sustain, and advance the nation's nuclear forensics capabilities.
- Purpose: The United States must possess the capability to respond rapidly and effectively to actual or intended nuclear attacks against it or its interests. The U.S. nuclear forensics capability supports the USG attribution policy to: deter an initial attack; assist in preventing follow-on attacks; inform the development of strategic response options; and support subsequent criminal prosecutions.



# **Certified Reference Material Working Group**

- Who are we?
  - DHS, FBI, DOD, the U.S. National Metrology Institutes (NIST & NBL), DOE, National Laboratories, and international partners.
- What do we do?
  - Research, develop, and produce nuclear forensic-specific reference materials
- International collaborations
  - Canada
    - National Research Council (NRC) Canada
    - Canada Nuclear Safety Commission (CNSC)
  - Sweden
    - Swedish Defense Research Agency (FOI)
    - Swedish Radiation Safety Authority (SSM)
  - United Kingdom
    - National Physical Laboratory (NPL)
  - France
    - Alternative Energies and Atomic Energy Commission (CEA)



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#### Nuclear Security Summit 2016

#### http://www.nss2016.org/



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#### **Nuclear Forensics**

Fact Sheet: Certified Reference Materials for Nuclear Forensics

Fact Sheet: U.S. Example: National Technical Nuclear Forensics (NTNF) Public Affairs Guide

Fact Sheet: Model Query Process for US National Nuclear Forensics Library

Fact Sheet: Template for Model Query Process for US National Nuclear Forensics Library



# **Certified Reference Material Working Group**

<u>Nuclear Forensics Certified Reference Materials (CRM's)</u> are analytical standards, produced to assure the **accuracy and precision** of nuclear and radiological material analyses that are performed for the purpose of measuring critical signatures that will aid in the determination of a material origin and/or identity.

- Provide measurement capability and confidence for:
  - Method Development
  - Method Validation
  - Measurement Quality
  - Capability Enhancement
- Nuclear forensic signatures isotopic signatures
- CRMs are imperative for nuclear forensic analyses
  - Necessary for scientific evidence to be admissible in a US court of law
    - (Daubert Standard defensible measurements)

DOE Isotopes program is instrumental in our efforts



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### Why We're Producing

- NTNF is not interested in producing or supplying bulk isotopic materials
  - Producing highly characterized materials
    - On their way to becoming SRMs, CRMs
- Identified NTNF reference material needs:
  - Trace Actinide Assay/Composition RMs
  - Trace Element Assay RMs
  - Isotopic Composition Measurement RMs
  - Isotope Dilution Mass Spectrometry RMs
  - Radiochronometric RMs
  - Physical Property RMs
    - Microscopy / particle morphology







#### **CRM Lifecycle Process**





#### **NTNF CRM Priorities & Progress**



#### NTNF Isotope Resources

# Our capabilities and resources are available to the isotopes community

- Idaho National Laboratory Mass Separators
  - Stable Isotope Mass Separator (SIMS)
    - Currently operational
  - Radioisotope Mass Separator (RMS)
    - Testing prior to "hot" separations



Mass Separator (left) Oscilloscope trace Xenon Isotopes (right)





"HOT" Radioisotope Mass Separator



#### Isotope Mass Separators at INL

- Common specifications for both isotope mass separates:
  - Research scale
  - "Scandinavian type" separators
    - 90° sector 1.5 m radius magnets
  - "Dynamic"
    - Can accommodate a variety of ion source and ion collector types
      - Allows for a wide variety of elements and compounds to be introduced into the separators



#### Isotope Mass Separators at INL

Mass Separator Specs	Stable Isotope Mass Separator	Radioisotope Mass Separator		
Ion Source	Radiofrequency (RF) Refractory metals	Hot Cathode Metals (mid-range boiling points – 1000-1500°C), Noble gases		
Acceleration Potential	0 - 40 kV			
Beam Focusing	Extraction electrode, quadrupole, vertical steering plates	Extraction electrode, Einzel lens, vertical steering plates (quadrupole and vertical steering plates considered in near future)		
Beam Dispersion, $d = R \cdot (\Delta m/m)$	For neighbor isotopes (∆m = 1), m = 130, d ≈ 11.5 mm m = [actinides], d ≈ 6 mm			
Beam Current	30 - 100 µ-amp Element-dependent	30 - 100 µ-amp Element-dependent, currently under optimizatior		
Source Efficiency	0.1% - 1.0% Element-dependent			

#### Example using Stable Isotope Mass Separator:

Sample: Barium (2.4% <sup>134</sup>Ba) Collection Rate: 1 µg / hr, (>99% <sup>134</sup>Ba in one pass)



#### Available NTNF Reference Materials

Nuclear Forensics Reference Material <sup>1</sup>	Unit Size	Primary Element or Isotope <sup>2</sup>	Mass Primary Per Unit <sup>3</sup>	Attributes for Certification	Project Status
CRM 125-A (RM 8617)	5.4 g UO <sub>2</sub>	LEU (4.0574% <sup>235</sup> U)	4.76 g	U isotopic composition U assay (g • g oxide <sup>-1</sup> ) Model Purification Date	Certification completed Material available
CRM U045	5 mL soln (1 M HNO <sub>3</sub> )	LEU (4.5153% <sup>235</sup> U)	5 mg	U isotopic composition including <sup>233</sup> U and <sup>232</sup> U	Certification completed Material available (additional attributes under review)
NFRM U-1	1.5 g U <sub>3</sub> O <sub>8</sub>	HEU (93.2330% <sup>235</sup> U)	1.27 g	U isotopic composition Trace Pu isotopic composition U assay (g U • g oxide <sup>-1</sup> ) Trace actinide mass fractions (mg • kg sample <sup>-1</sup> )	Certification completed Material available
NFRM U-2	1.5 g U <sub>3</sub> O <sub>8</sub>	HEU (52.800% <sup>235</sup> U)	1.27 g	U isotopic composition Trace Pu isotopic composition U assay (g U • g oxide <sup>-1</sup> ) Trace actinide mass fractions (mg • kg sample <sup>-1</sup> )	Certification completed Material available
CRM U630 1-g	1.26 g U <sub>3</sub> O <sub>8</sub>	HEU (63.353% <sup>235</sup> U)	1.07 g	U isotopic composition including <sup>233</sup> U and <sup>232</sup> U U assay (g • g oxide <sup>-1</sup> ) Model Purification Date	Certification completed Material available (additional attributes under review)
CRM U630 10-mg	$13 \text{ mg U}_3\text{O}_8$	HEU (63.353% <sup>235</sup> U)	10 mg	U isotopic composition including <sup>233</sup> U and <sup>232</sup> U	Certification completed Material available (additional attributes under review)

Listed in this table are current and future nuclear forensic reference material projects supported by DHS. LEU refers to Low Enriched Uranium, DU refers to Depleted Uranium, and HEU refers to Highly Enriched Uranium.

<sup>1</sup> The reference material designations in parentheses are preliminary numbers assigned to the reference material at the National Institute of Standard and Technology.

<sup>2</sup> The "Primary Element or Isotope" refers to the primary actinide element or radioisotope in the reference material.

<sup>3</sup> The "Mass Primary Per Unit" is the approximate mass of the primary actinide element or radioisotope component in each unit.



#### NTNF CRM WG Isotope Supply

• What we have in reserve that could be distributed to the community

#### **Isotope Supply**

Isotope	Physical Form	Purity and/or specification s	Location of reserve	Size of reserve	Comments
Th-229	Nitrate Soln	>99.9% Th-229	ORNL	20 µg (80 Units)	In review for certification
Am-243	Nitrate Soln	>99.9% Am-243	ORNL	50 μg (25 Units + stock)	Certified and verified
Ba-134	Nitrate Soln	~99% Ba-134	TBD	<1 mg	Currently being processed into 1- $\mu$ g units at INL.
U-233	Nitrate Soln	>99.99% U-233	LLNL	~ 1 g	Currently at LLNL waiting project for unit production.
Pu-244	Nitrate Soln	>99.9% U-244	LLNL	<100 µg	Currently at LLNL (60% of material for IAEA).
Pa-231	Nitrate Soln w/HF	>99.9% U-244	LLNL	<100 µg	Currently at LLNL waiting project for unit production.



#### NTNF Certified Reference Materials DHSCRMInfo@hq.dhs.gov





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