

Nuclear Forensic Reference Materials (RM) for Attribution of Urban Nuclear Terrorism

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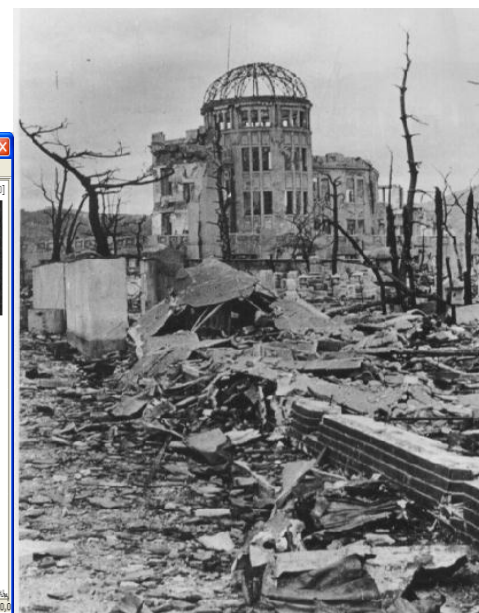
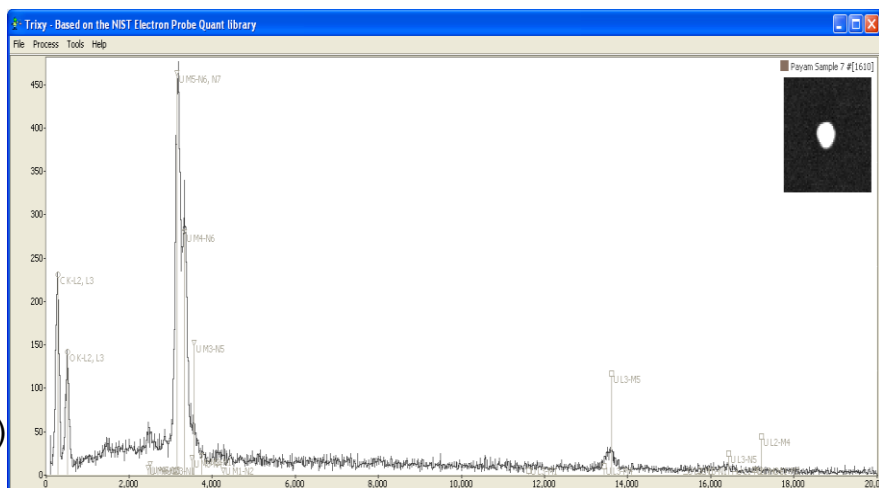
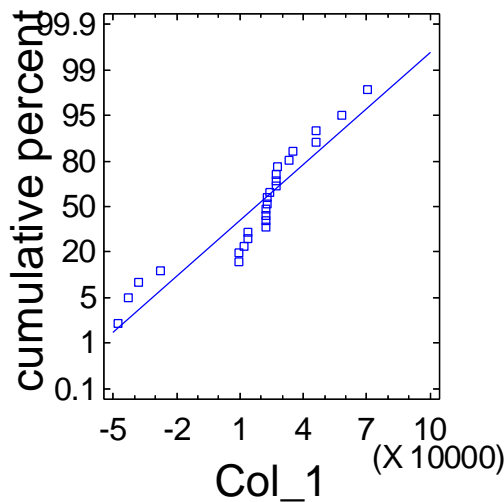
Jeffrey Leggitt & JoAnne Buscaglia (FBI)

Simon Jerome (NPL)

John Molloy (NIST/ACD)

William Pramenko (VIP)

Normal Probability Plot



BLUF

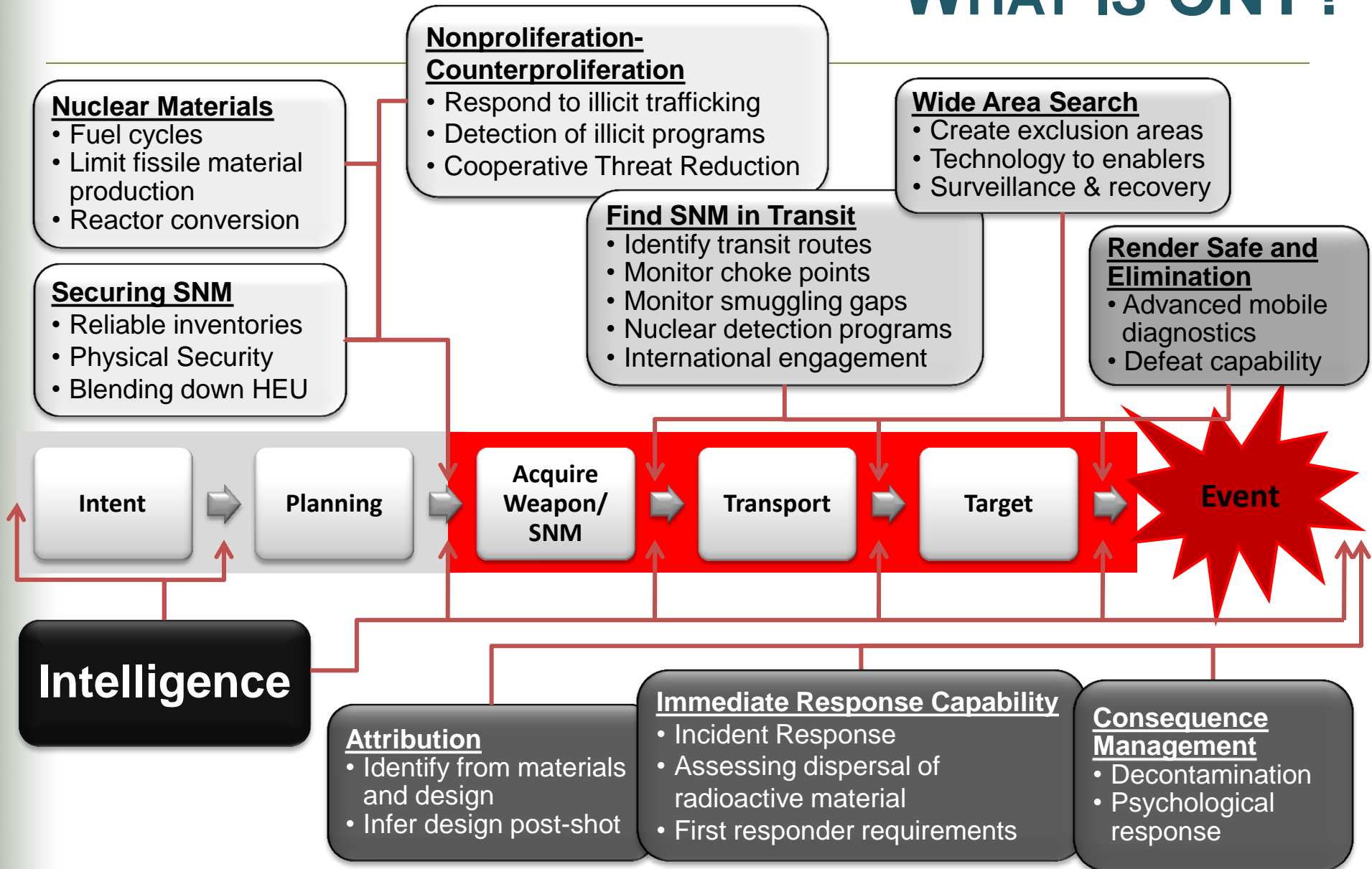
Understanding DoJ, DoD, DHS, DOE needs requires close relationships

Nuclear Forensics require high-level expertise, undisputed signatures & extremely high fidelity measurement capabilities

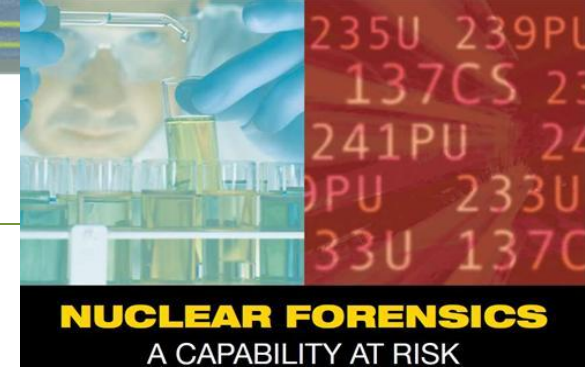
CBRNE countermeasures must withstand legal scrutiny, and requires strong metrology backbone

Requirements for CRMs exceedingly high

WHAT IS CNT?



Definitions



Nuclear Forensics (NF) and Attribution (A)

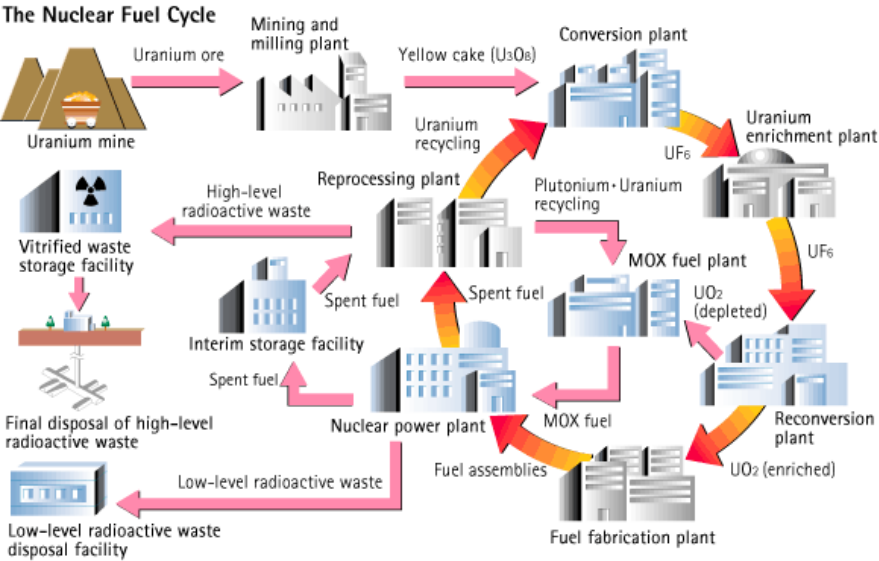
- (NF) - Nuclear forensics is the use of physical and chemical properties to help determine the age, origin, and history of nuclear materials and the design of nuclear devices. Identifying these “nuclear signatures” can help investigators track nuclear materials back to their origin
- (A) Attribution is the integration of all information, including nuclear forensic data, law enforcement and intelligence data, to suggest or exclude the origin of nuclear materials and devices, routes of transit, and responsible groups or individuals

Physical –Chemical –Radiological and Isotopic Characterization

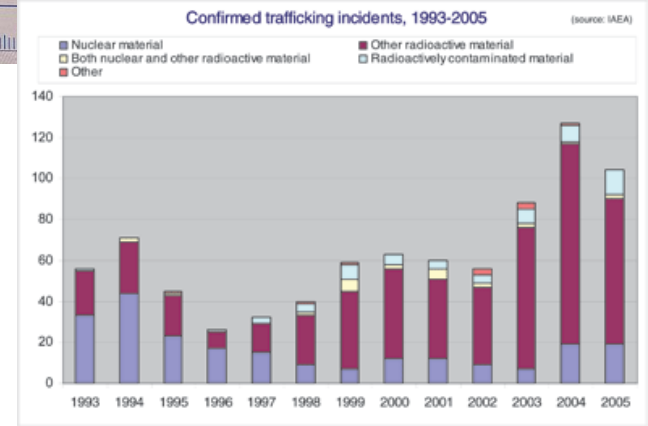
What is it?
How was it constructed?
When?
Where, Who, Why?

Increasing Uncertainty





Signatures are created and erased throughout the life cycle of nuclear materials



Some of these signatures are maintained in Interdicted & Post Detonation Materials

Nuclear Forensic Goals & Applications

Goals

- **Identify** those responsible for nuclear theft or nuclear terrorism
- **Deter** the theft of nuclear materials or acts of nuclear terrorism
- **Enhance** protection, control, and accounting for nuclear materials and weapons
- **Prevent** additional thefts or terrorist acts by plugging leaks and tracking down other missing materials or devices

Applications

- Intercepted nuclear materials or devices
- Debris from a nuclear explosion
- Debris from a radiological dispersal device



Some of these signatures are maintained in
Interdicted & Post Detonation Materials

What Information does Nuclear Forensics Provide?

89 Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm
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Nuclear Forensics – Weapons Fuel Processing

Parameter	Signature	Analytical Technique
Appearance	Material type (e.g. powder, pellet)	Optical Microscopy
Dimensions (Pellet)	Reactor Type	Database
U, Pu, Content	Chemical Concentration, nuclear fuel type, weapon type, and device type	Titration, Hybrid K-Edge Dosimetry, Isotope Dilution Mass Spectrometry
Isotopic Composition	Enrichment--intended use/reactor type, nuclear fuel type, weapon type, and device type	Gamma Spectrometry, TIMS, ICP-MS, SIMS
Impurities	Production Process, geolocation, device type, and weapon type	ICP-MS
Age	Production Date	Alpha Spectrometry, TIMS, ICP-MS
Surface Roughness	Production Plant	Profilometry
Microstructure	Production Process	SEM, TEM

Nuclear Forensics – Weapon Design

Parameter	Signature	Analytical Technique
Fission Fuel	Pu, U Ratio	Radiochemistry, actinide mass spectrometry
Reactor Power and Irradiation time	Fission Product Isotopic Ratios	Radiochemistry, beta/gamma counting
Fuel Sophistication	Enriched U and Pu Isotopic Ratios	Radiochemistry, actinide mass spectrometry
Initiator	$^{210}\text{PoBe}$	Chemical analysis, Radiochemistry, alpha-counting
Boosting	^6Li	Mass Spectrometry
Fission neutron energy	Fission Yield Curve	Radiochemistry, alpha/beta/gamma counting

Evidence Legal Standards

Daubert v. Merrell Dow Pharmaceuticals (1993)

- Has the technique been validated?
- Were the conditions controlling the technique's operation maintained?
- Were the results peer reviewed?
- Does, and at what frequency, the method lead to any erroneous results? (false positives and/or negatives)
- Has the technique been generally accepted in the scientific community?

US Federal Rules of Evidence 702

- evidence is based upon sufficient facts or data,
- evidence is the product of reliable principles and methods, and
- the principles and methods have been applied reliably to the facts of the case

**What is missing? Urgent CRMs to support State-of-the-Art
Measurement Capabilities and Metrology Infrastructure**

**Non-Existent
Not Good Enough for High Fidelity Attribution
in Short Supply
Exceedingly Rare
Long Development Time
Very Expensive**

**Problem? Required CRMs are needed NOW
5 years to develop one CRM
Few have the capability to develop state-of-the-art CRMs
CRM production require specialized non-existent facilities
Process is labor intensive and costly**

How to Close the Gap?

**Enlist increased numbers of capable metrologists
Focus their attention on this mission (affect their priority list)
Establish specialized facilities**

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Participants: Kenneth G.W. Inn (NIST-RBPD), Jacqueline Mann (NIST-RBPD), Jeffrey Leggitt (FBI), JoAnne Buscaglia (FBI), Simone Jerome (NPL), John Molloy (NIST-ACD), William Pramenko (VIP)

Forensic science problem: No fresh fission product urban RM to test nuclear forensics capabilities for attribution

Objective(s):

- Micro-homogeneity Evaluation of UVC material using micro-XRF
- Microwave digestion/fusion dissolution
- Elemental/actinide concentration by ID-HR-ICP-MS
- Stable isotopic “fingerprint” by HR-ICP-MS
- Documentation/Publications
- Elemental/isotopic mass certification of UVC



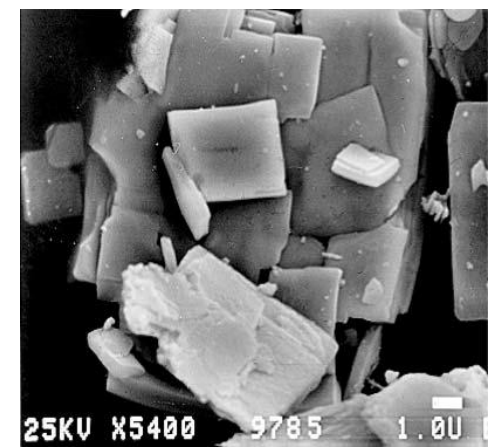
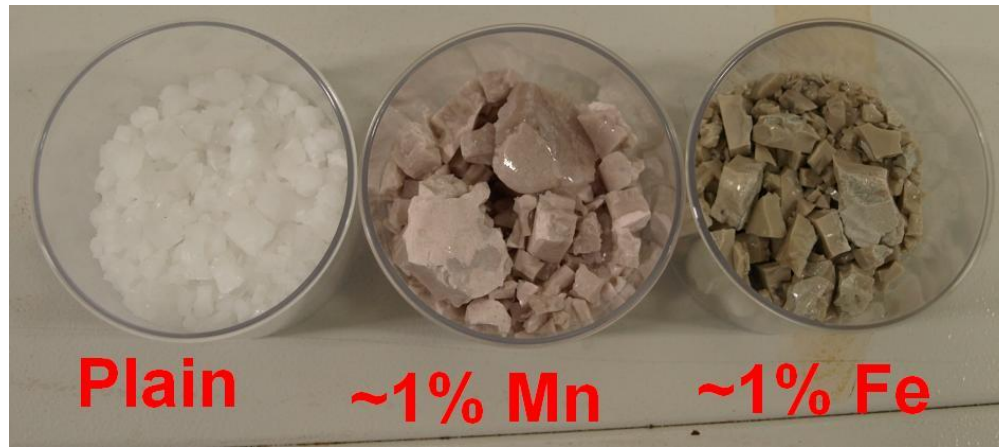
Nuclear Forensic Reference Materials (RM) for Attribution of Urban Nuclear Terrorism

End of project outcomes: Characterization of HEU doped UVC to test the U.S. nuclear attribution capabilities

- Documentation/Publications
- UVC RM certified for Elemental/isotopic mass

Anticipated impact on forensic science: RM for high fidelity attribution capabilities of an urban nuclear terrorist event

- Method development/validation/testing/preparedness
- High fidelity attribution/legal defensibility (Daubert v. Merrell Dow)



Nuclear Forensics Challenge

Figure 1.

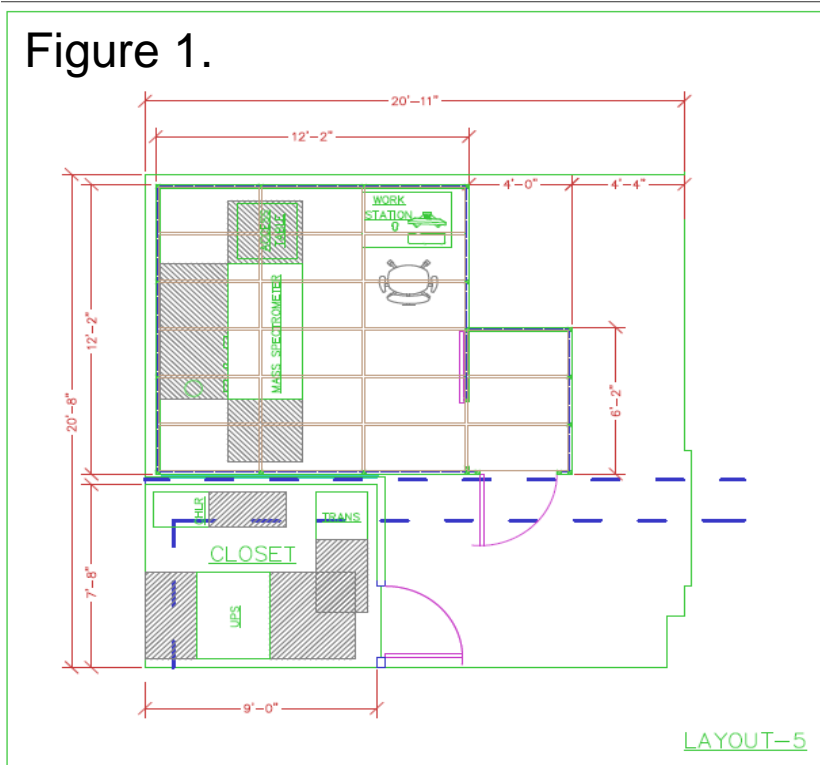


Figure 1. Approved layout for the lab retrofit.

Goal: Lab space with controlled humidity and temperature for optimal operation of new ELEMENT2 mass spectrometer.

**Lab
Retrofit**

Figure 2. Exterior Hard wall clean lab space for new mass spectrometer



Figure 2.



Figure 3.

Figure 3. Interior clean lab space with continuous flooring

Nuclear Forensics Challenge



Mass Spectrometer Purchase



Element2 HR-ICP-MS high-resolution (single-collector) sector field inductively-coupled plasma mass spectrometer

Goal: Establish NIST atom-counting capability for required high-precision/accuracy assay/isotopic trace and major radio-element nuclear forensics certified reference materials

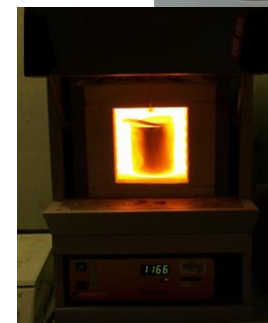


Nuclear Forensics Challenge



UVC Characterization/Certification Plan

- Assess UVC micro-homogeneity with micro-XRF
- Develop dissolution protocols
- Determine elemental/actinide mass concentrations
- Determine Stable Isotopic "Signature"
- Document/Certify UVC elemental/isotopic mass concentrations



Work Done



UVC Dissolution

Procedure: 4 samples (2 of 0.100 g and 2 of 0.200 g):

Step 1 – Digest Al with $\text{H}_3\text{PO}_4 + \text{HCl}$

Step 2 – Digest Si with $\text{HBF}_4 + \text{HNO}_3$

Microwave held at 200 bar, 300° C

Results: Success with 0.100g samples
particles observed with 0.200g samples

Future: Will attempt with pure HF and then
 H_3BO_3 replacing HBF_4

UVC Composition

Element	ppm	Element	ppm	Element	ppm
Si	186000	As	14	Cl	37
Al	7900	B	3	Sr	130
Fe	230000	C	470	Cr	850
Ca	220000	Co	37	Zn	47
Mg	4800	Cu	220	F	260
S	3700	Mo	190	Zr	15
Na	700	Nb	55	(N	2)
K	1900	Ni	450	U	100
Ti	550	Sn	36	W	10
P	200	Ta	25	Th	10
Mn	1200	V	150	(O	Balance)

Conclusions

- **Understanding DoJ [FBI], DoD [COCOMs, JRO], DHS [DNDO/NTNFC], DOE [NNSA] needs requires close relationships**
- **Nuclear Forensics require high-level expertise, undisputed signatures & extremely high fidelity measurement capabilities**
- **CBRNE countermeasures must withstand legal scrutiny, and requires strong metrology backbone**
- **Uncertainty Requirements for CRMs exceedingly high**

Concentration [ppm]	Uncertainty [k = 2, %]
High [500-1000]	1 - 5
Medium [100-500]	1 - 5
Low [1-100]	5 - 30

Thank You

Future Plans

Micro-homogeneity of UVC material using micro-XRF

Microwave digestion/fusion dissolution

Elemental/actinide concentration by ID-HR-ICP-MS

Stable isotopic “fingerprint” by HR-ICP-MS

Documentation/Publications

Elemental/isotopic mass certification of UVC

