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S. Adam Stratz

University of Tennessee, Knoxville

Jonathan A. Gill

University of Tennessee, Knoxville

John D. Auxier II

University of Tennessee, Knoxville

Howard L. Hall

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Modern Advancements in Post-Detonation Nuclear Forensic Analysis

S. Adam Stratz

Department of Nuclear Engineering, Radiochemistry Center of Excellence, University of Tennessee, Knoxville, USA

Jonathan A. Gill

Department of Nuclear Engineering, Radiochemistry Center of Excellence, University of Tennessee, Knoxville, USA

John D. Auxier II

Department of Nuclear Engineering, Radiochemistry Center of Excellence, Institute for Nuclear Security, University of Tennessee, Knoxville, USA

Howard L. Hall

Department of Nuclear Engineering, Radiochemistry Center of Excellence, Institute for Nuclear Security, University of Tennessee, Knoxville, USA

Abstract

Deterring nuclear terrorism is a critical national asset to support the preclusion of non-state actors from initiating a nuclear attack on the United States. Successful attribution of a detonated nuclear weapon, which includes locating the source of the radiological materials used in the weapon, allows for timely responsive measures that prove essential in the period following a nuclear event. In conjunction with intelligence and law enforcement evidence, the technical nuclear forensics (TNF) post-detonation community supports this mission through the development and advancement of expertise to characterize weapon debris through a rapid, accurate, and detailed approach. Though the TNF field is young, numerous strides have been made in recent years toward a more robust characterization capability. This work presents modern advancements in post-detonation expertise over the last ten years and demonstrates the need for continued extensive research in this field.

I. Introduction

In recent years, the United States has called upon the scientific community to address gaps in technology to improve the performance of forensics as a deterrent to nuclear terrorism [1]. The Nuclear Forensics and Attribution Act (NFAA) [2], enacted in 2010, is the legislative embodiment of this directive that stresses the technological readiness such a scenario necessitates and has been approached through an interagency and academic collaboration [3, 4]. Technical Nuclear Forensics (TNF) has been established as the specialized field of science to enhance this technology and analyze nuclear residues of interdicted (pre-detonation) and exploded (post-detonation) nuclear materials. Attribution of these materials employs TNF findings in concert with intelligence and law enforcement evidence to locate the source of these materials. As the following pages highlight, the post-detonation arm of TNF has made recent technological strides in identifying weapon characteristics from nuclear debris to supply timely, high-quality data in support of the attribution process.

A. Legal Benchmarks of TNF Data

As in any field of forensics, data supporting the nuclear forensic analysis process may eventually reach judicial review. Though nuclear forensic evidence may not necessarily encounter the judicial process before the President and his/her national security council make an attribution decision in the event of a nuclear attack, any country wishing to attribute a nuclear incident to another sovereign nation or subnational entity will face intense scrutiny, and as such, must have a high standard of legally defensible forensic methodology. The NFAA does not contain language specifically referring to a defined standard; however, it recommends international cooperation and designates investigative agencies that are bound by legal standards.

The standard most relevant to nuclear forensic methods is the Daubert standard, as it applies to the Federal Rules of Evidence, Article 7, Rule 702 [5–7]. Based on the Daubert standard, judges are given means by which they can assess an expert's scientific testimony on the grounds of reasoning or methodology. Under this standard, the five factors used to assess the validity of a method are (1) whether the theory or technique in question can be and has been tested, (2) whether it has been subjected to peer review and publication, (3) its known or potential error rate, (4) the existence and maintenance of standards controlling its operation, and (5) whether it has attracted widespread acceptance within a relevant scientific community [6].

For the United States, any research effort seeking broad acceptance and government support must meet this standard.

Application of this forensics standard has rightly received rigorous attention in the scientific community [5, 8–11]. In addition, the National Institute of Standards and Technology (NIST) and other researchers are establishing certified reference materials (CRMs) and recognized databases of nuclear information that may act as a known standard for other nuclear materials [12]. Both of these standards generally agree with the requirements for competence outlined in International Organization for Standardization (ISO) code 17025.

B. Essential Steps: Nuclear Forensic Analysis

Post-detonation nuclear forensic analysis begins with the collection of materials produced in the extreme temperature and pressure where the weapon detonates. In the aftermath of a detonation, a specialized type of debris is formed that effectively encapsulates weapon components and fission products in a solidified, glassy matrix [13]. This debris, or nuclear melt glass, is essential for nuclear forensic scientists to conclude weapon characteristics during post-detonation nuclear forensic analysis [14]. Analyzing the debris begins with non-destructive physical and radiological characterization and progresses toward dissolution and destructive analysis. Table 1 (below) as published in *Nuclear Forensics: Strategies and*

Techniques, shows typical analytical techniques to characterize pre-detonation nuclear materials; techniques for analyzing post-detonation materials, as outlined in *Documentation of a Model Action Plan to Deter Illicit Nuclear Trafficking* (Table 2), are performed in a similar manner, with radiochemical separations and radiological characterization having the largest contribution to subsequent attribution.

Table 1: Characterization Techniques for Pre-Detonation Nuclear Materials (IAEA) [15]

Techniques/Methods	24 h	1 Week	2 Months
Radiological	Total Activity		
	Dose Rate (α , β , γ , n)		
Physical	Surface Contamination		
	Visual Inspection	SEM/EDS	TEM (EDS)
	Radiography	XRD	
	Photography		
	Weight		
	Dimensions		
	Optical Microscopy		
	Density		
Traditional Forensics	Fingerprinting, Fibres		
Isotope Analysis	γ spectroscopy	SIMS, TIMS, ICP-MS	Radiochemical Separation
	α spectroscopy		
Elemental/Chemical		ICP-MS	GC-MS
		XRF	
		Titration	
		IDMS	

C. Collection of Species

Collecting ground samples of nuclear fallout debris is the essential first step toward forensic attribution. Samples must be taken from a site sufficiently close to the detonation source or fallout plume to ensure the samples were created in the fireball and encapsulate the necessary fission products, activation products, and anthropogenic materials needed during forensic analysis. Debris collection falls outside the scope of this work and will not be discussed here.

II. Synthetic Nuclear Debris

Rapid sample analysis is essential for forensic attribution in a post-detonation scenario. A recent multi-agency effort between the Federal Emergency Management Agency (FEMA), the Department of Homeland Security (DHS), and the Defense Threat Reduction Agency (DTRA) addressed the repercussions of an urban nuclear event and the uncertainty associated with samples of urban nuclear debris for forensic analysis [4]. While samples of nuclear melt glass (both surface and aerodynamic debris) are available to the academic community from the Trinity test, many fission products have decayed and the Trinitite samples are only quasi-representative of the signatures that would be obtained from a newly acquired sample. Therefore, much work is being dedicated to creating realistic synthetic samples of nuclear melt glass for the experimental development of post-detonation analytical techniques. These surrogates began as simple highly enriched uranium (HEU)-doped sol-gel glass, as reported by Carney *et al.* in 2013 [16]. The glass was impregnated with 93% HEU and neutron irradiated for 15 minutes in order to simulate, on a first-tier basis, the fission and activation products that would be found in nuclear debris.

Many papers followed that advanced the elemental accuracy of synthetic nuclear debris. Trinitite, the most accessible nuclear debris to the academic community, was first synthetically modeled by Molgaard in 2014 [17] and the technique was subsequently published in 2015 [13]. Studies ensued, determining the

physical, chemical, and radiological accuracy of this synthetic debris as compared to actual trinitite; researchers achieved excellent correlation [13, 18].

The need for synthetic nuclear melt glass representative of an urban environment was the next step toward developing analytical techniques for attribution purposes. Giminaro *et al.* recently addressed this need in a study detailing city-specific formulation techniques to identify the elemental composition of any given city using land use data [14]. Two representative samples (Houston, TX and New York, NY) were modeled and synthesized in order to demonstrate the procedure. The need for synthetic nuclear debris, which can be directly compared to actual debris and those that represent a hypothetical urban event, was addressed for the first time in recent years; efforts to improve the realism of the samples are ongoing. These samples provide a more credible baseline for developing forensic techniques for real post-detonation debris [19].

III. Analysis

Analyzing nuclear debris to characterize its physical, chemical, and radiological signatures is a vital component of the technical nuclear forensics process [20]. The procedure aims to reverse-engineer the design of a detonated weapon using the debris it generates. The community of analytical nuclear forensics has achieved significant strides in recent years toward improving the timeliness and accuracy of these techniques.

Upon the detonation of a nuclear weapon, the resulting debris consists mainly of oxidized materials that contain a variety of radiological and elemental forensics signatures [21, 22]. [22, 23][22, 23]Elemental and radiological signatures can be detected using a variety of methods. Elemental signatures consist of the elements captured by the heat and pressure of detonation and are incorporated into the final composition of the melt glass. Radioactive signatures consist of unstable elements that have a tendency to decay. It should be noted that many of the techniques used in traditional forensics can be used in nuclear forensics [23]. Table 2 contains a variety of techniques that can be used for post-detonation nuclear forensics [23]. It is important to note that Table 2 does not have the associated time component that Table 1 contains because attribution should proceed as quickly as possible in a post-detonation scenario.

Table 2. Post-Detonation Nuclear Forensic Techniques [23]

Techniques/Methods	Instrumentation	Pre-Preparation
Radiological	Alpha (α) spectroscopy	Remove stable element contamination
	Beta (β) counting	Immerse in liquid scintillation fluid to determine gross count rate
	Gamma-ray (γ) spectroscopy	No preparation needed other than similar counting geometry to standard counting source
Physical Characterization	Radiography	None
	Photography	None
	Weight	None
	Dimensions	None
	Optical Microscopy	None
	Density	None
Isotope Analysis	Gamma-ray (γ) spectroscopy	No preparation needed other than similar counting geometry to standard counting source

	Alpha (α) spectroscopy	Remove stable element contamination
	Secondary Ion Mass Spectrometry (SIMS)	Dissolution to appropriate concentration, removal of isobaric interferences
	Thermal Ionization Mass Spectrometry (TIMS)	Dissolution to appropriate concentration, removal of isobaric interferences
	Inductively Coupled Plasma – Mass Spectrometry (ICP-MS)	Dissolution to appropriate concentration, removal of isobaric interferences
Elemental Analysis	Laser Ablation – ICP-MS	None
	Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDS)	Samples must be polished prior to analysis
	X-ray fluorescence (XRF)	None
	ICP-MS, SIMS	Dissolution to appropriate concentration, removal of isobaric interferences
	LA-ICP-MS	None
	X-ray absorption near edge structure (XANES)	Dissolution to appropriate concentration

As shown above, some of the mass spectrometry techniques useful for identifying elements are also useful in determining isotopic ratios. The subsequent sections describe the techniques used in the literature regarding the analysis of trinitite and similar fallout [13, 18, 24–36].

A. Elemental Analysis

The elements found in nuclear melt glass are largely found in their oxide (and occasionally chloride) form due to the excessive oxygen and extreme temperatures found in the toroidal region of the blast [24]. When performing elemental analysis on a debris sample, it is important that the analytical techniques are performed so that the spatial integrity of the sample is preserved prior to interrogation. Physical characterization—requiring largely non-destructive techniques—includes morphology of the sample, texture, stratification, and other statically observable characteristics of the debris. The presence of elements in the sample matrix may be indicative of several weapon characteristics and is an important aspect of physical characterization. Some of the more important constituents are plutonium and uranium; these elements are important because the debris contains trace levels of fissile material resulting from an incomplete detonation—no reaction is one hundred percent efficient—and provide useful indications of the initial state of the fuel. Previous reported literature took advantage of alpha spectroscopy on thin vertical slices of trinitite to identify deposits of U and Pu; however, a recent study by Donohue *et al.* integrated several additional techniques, including laser ablation inductively-coupled plasma mass spectrometry (LA-ICP-MS), electron microprobe analysis (EMP), energy dispersive X-ray fluorescence system (XRF), scanning electron microscopy (SEM), and back scattered electron analysis (BSE), in addition to alpha spectroscopy, to obtain a clearer picture of the distribution of elements of interest. Pu deposits were found up to 10 mm deeper in the sample than previously reported [36]. It is clear that more work is still necessary to validate and advance the physical characterization of post-detonation nuclear debris.

B. Scanning Electron Microscopy/Energy Dispersive X-ray Spectrometry

When analyzing nuclear melt glass, it is important to gain insight into the amount of homogeneity or heterogeneity of the elements of interest prior to performing techniques requiring destructive analysis. One rapid technique for determining the spatial resolution of matrix elements is scanning electron microscopy/energy dispersive x-ray spectrometry SEM/EDS; however, the limiting factor for SEM/EDS is debated in literature [37, 38] and it certainly provides poorer sensitivity than other TNF methods. It is generally agreed that SEM/EDS has the advantage of providing the “whole picture” of elemental dispersion, but has only provided elements with an atomic number greater than 5, targeting boron as a problematic constituent due to its low photon energy, and thus, low x-ray yield [39]. The concentration of the element is required to be greater than 0.001 wt. percent, though it is preferred if the elements of interest have a concentration > 0.01 wt. percent. For amorphous matrices such as nuclear melt glass, the major elements of interest are Si, Ca, Na, K, B, Sb, and Fe. Traditional trace-level elements include Al, As, Fe, Ba, and Mg. To complement this technique, most analysts refer to LA-ICP-MS.

C. Laser Ablation Inductively-coupled Plasma Mass Spectrometry

Laser ablation inductively-coupled plasma mass spectrometry (LA-ICP-MS) is an appealing post-detonation analysis technique because of its ability to introduce samples into the instrument without prior sample dissolution. This is a powerful technique that is particularly beneficial for samples that may fully dissolve during a dissolution process. Essentially, the laser evaporates or sublimates a portion of the material for detection, precluding any need for dissolution. LA-ICP-MS is used to determine elements at the ultra-trace level (at concentrations of less than 0.0005 wt. percent), commonly including Ti, Cr, Co, Ni, Zn, Zr, Y, Nb, Tc, Ru, Sn, Te, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, W, Th, U, Pu, and Am [36]. This is useful for detecting both impurities in a sample and ultra-trace elements of interest to the forensic scientist. Additionally, due to the potent capabilities of ICP-MS, it is possible to distinguish relevant isotopes as discussed in the following section.

D. Isotopic Analysis

It is important to note that isotopic signatures of stable isotopes are a useful source of information when performing the analysis of post-detonation debris. A key interest is determining stable oxygen ($^{18}\text{O}/^{16}\text{O}$) ratios, as noted by Keoman *et al.* [40]. In particular, it was determined that the large neutron flux was incapable of producing a significant change in the $^{18}\text{O}/^{16}\text{O}$ ratio greater than the ordinary variation observed (11.2 – 15.5%). Likewise, Bellucci *et al.* reported the use of stable Pb ratios of ^{208}Pb , ^{207}Pb , ^{206}Pb , and ^{204}Pb with LA-MC-ICP-MS to determine the geographic source of the Pb in the weapon from the Trinity test (the debris is the only openly available weapon debris in existence). The distinct overlapping isotopic ratio in both the trinitite sample and the Buchans mine demonstrated that the lead originated in the Buchans mine in Newfoundland, Canada [27]. These heavy metal isotopes shed light on both the ores used to create components of the device or infrastructure surrounding the device and the heavier elements (uranium, thorium) from which it decayed. ^{204}Pb is alone in its natural primordial origins; the other three isotopes stem directly from the long-lived decay products of ^{232}Th , ^{235}U , and ^{238}U [27]. Such interrogations are extremely useful during the sourcing process following technical analysis, though an urban detonation would certainly produce a more complex elemental matrix than the relatively simple elemental matrix of a sandy desert. Isotopes can be detected using a variety of instrumentation including ICP-MS, where a sample is broken into its elemental constituents and a mass-to-charge ratio is measured.

E. Oxidation States

Research by Nelson *et al.* [41] has noted the relevance of x-ray absorption near edge structure (XANES) when determining the oxidation state ratios of $^{5+}\text{U}/^{6+}\text{U}$ and $\text{Fe}^{2+}/\text{Fe}^{3+}$. Some variation in the oxidation of the iron was found, with the Fe ratios varying from 33–55 percent Fe^{2+} , implying reducing conditions and concluding that the dominant species of plutonium was Pu^{4+} .

IV. Radiological Signatures

The presence of radiation is a unique characteristic of nuclear forensic samples when compared to forensic samples in other scientific disciplines. Radiation signatures from post-detonation debris can greatly advance the forensic interrogation of the sample.

A. Gamma-ray Spectroscopy

The most common (and typically, first to be employed) form of radioactivity analysis is gamma-ray spectroscopy [18, 24, 25, 30, 34, 42]. In natural trinitite, ^{133}Ba , ^{137}Cs , ^{152}Eu , ^{154}Eu , ^{155}Eu , ^{239}Pu , ^{241}Pu , ^{241}Am , and ^{60}Co have been reported, whereas synthetic versions of these materials also showed ^{24}Na , ^{140}La , ^{42}K , ^{59}Fe , ^{47}Ca , ^{132}I , ^{46}Sc , ^{95}Zr , ^{130}I , ^{133}I , ^{103}Ru , ^{131}I , and ^{132}Te . There have been four studies on the radioactive nuclides in trinitite as measured by gamma-ray spectroscopy—the resulting isotopes and their specific activities (Bq/g) are summarized in Table 3.

Table 3. Reported Radioactive Nuclides in Trinitite Activities in (Bq/g) for Specific Isotopes of Interest

Isotopes	Schlauf [42]	Parekh [34]	Bellucci [28]	Belloni [25]
^{133}Ba	9.9 ± 0.6	9.8 ± 0.26	53 ± 39	NR
^{137}Cs	90 ± 9	121.8 ± 0.1	85 ± 72	
^{152}Eu	27 ± 1	25.84 ± 0.38	50 ± 27	
^{154}Eu	4.8 ± 0.6	2.74 ± 0.67	158 ± 134	
^{155}Eu	NR	NR	0.9 ± 0.6	
^{239}Pu	NR	NR	11208 ± 3907	86
^{241}Pu	NR	63.0 ± 1.8	NR	
^{241}Am	2.9 ± 0.5	4.14 ± 0.06	13 ± 11	0.4
^{60}Co	29.4 ± 4.4	44 ± 4	NR	0.015
^{90}Sr	NR	NR	NR	13

It is important to note that there are large discrepancies between the reported radionuclides, many of which may be attributed to potentially massive sampling errors. Cook *et al.* [18] reports on the comparison of surrogate nuclear melt glass (synthesized to appear chemically and morphologically similar to trinitite, as reported by Molgaard *et al.* [13, 18] to actual trinitite. In this study, a number of radionuclides were observed, but the only correlation between synthetic samples and those found in trinitite was ^{152}Eu .

B. Alpha Spectroscopy

Direct alpha spectroscopy and preparation of intact debris samples is a useful technique in nuclear forensics; however, its use in studying nuclear melt glass is limited to one study performed by Eaton *et al.* [43]. Sample preparations consisted of a very thin slice of nuclear melt glass placed in an alpha spectrometer, where a distinct peak at 5.157 MeV was observed, correlating to both ^{239}Pu and ^{240}Pu .

Additional peaks exhibited energies of 5.486 and 5.499 MeV (corresponding to ^{241}Am and ^{238}Pu , respectively). The activity ratios were subsequently calculated.

V. Separation Techniques

To perform mass spectrometry analysis of nuclear samples, it is important to reduce isobaric or radiation interferences using analytical separations. A particular concern before performing separations is the dissolution of unique matrices that can encompass a large array of elemental constituents. Post-detonation materials are likely to include a suite of elements that are not found in traditional nuclear melt glasses such as silicon-rich trinitite. Advanced dissolution techniques are imperative for proper dissolution of complex matrices, and several recent publications have introduced innovative approaches to this challenge. Subsequent separations can be performed on these dissolved matrices following proper dissolution of the initial sample. Recent dissolution and separation techniques addressing post-detonation debris are highlighted in the following sections.

A. Dissolution and Laser Ablation

Destructive analysis of nuclear materials provides a useful platform for a variety of analytical methods. These methods provide data on both major and minor elemental parameters, producing information that can help identify the materials' intended uses (radiological dispersion, nuclear weapon, etc.), component/material age and source (do the isotopics point toward a specific mining operation?), reactor information (are there fingerprints indicative of specific reactors?) [44], and production processing [45, 46]. From 2005 to 2015, a variety of methodologies for the preparation of destructive samples were investigated; however, the focus has largely highlighted capabilities of laser ablation (LA) and methods of liquid dissolution.

Laser ablation multi-collector inductively-coupled plasma mass spectrometry (LA-MC-ICP-MS) has seen a recent increase in application since the introduction of laser-induced breakdown spectroscopy in the 1980s [46–54]. Continuous-wave CO_2 lasers remain the preferred method due to their power, wide availability, and wide application. Regarding nuclear melt glass, LA proves useful for specific actinides (e.g. plutonium isotopes), but struggles with many fission fragment nuclides [46, 47]. In particular, Ga–Rb and Mo–Cs suffer from volatilization loss with LA [46]. Even with these shortcomings, the appeal of the introduction of a direct sample into a mass spectrometer will continue to drive research; however, the complexity of laser methods appears to keep effective solutions out of reach.

The dissolution of trinitite, synthetic trinitite, and other melt glasses has been heavily reported in literature [29, 30, 32, 46, 55]. Sharp *et al.* discusses a mixture of concentrated HNO_3 and concentrated HF for 72hrs on a hotplate at 180°C [55]. Eppich *et al.* [26] reports a multi-stage approach whereby a 2.5:1 ratio of concentrated HNO_3 and concentrated HF react for 24 hrs until a white fluoride precipitate is formed. After the precipitate arises, concentrated HClO_4 is added and dried. Finally, dissolution is performed using concentrated HCl drying the dissolution with 3M HCl. This method provides excellent elemental identification with trinitite and should be a method for application to trinitite surrogates and other melt glasses. Hubley *et al.* reports using NH_4HF_2 in a mass ratio of 200mg of NH_4HF_2 to 50mg of trinitite heated in screwtop PFA vials to 230°C for 3hrs then cooled to 160°C . After cooling, concentrated HNO_3 is added, the solutions are evaporated to near-dryness at 160°C , then re-dissolved in a 1:1 ratio of H_2O and concentrated HNO_3 . The final mixture is heated to 120°C for 1 hr yielding a clear, colorless solution. Final dilution is completed with 2% HNO_3 [32]. Hubley *et al.* also reports using a 1:1 ratio of concentrated HNO_3 and concentrated HF (total volume of 2mL) in a pressure microwave vessel. The samples are heated at 400W to a temperature of 140°C , followed with 25min at 600W and allowed to cool. Twenty-three mL of 4% H_3BO_3 was then added. The vessels are first heated at 400W to 140°C for 10min, then for 25min at 600W. The samples are allowed to cool and are diluted with H_2O [32].

Regarding dissolution of synthetic melt glass surrogates, Liezers *et al.* reports a method whereby 2mL of concentrated HF and 0.2mL of HNO₃ are heated at 353K for 36 hrs with intermittent ultrasonification treatment. The samples are then evaporated, and both concentrated HNO₃ and concentrated HCl are added for final dissolution [47]. Maxwell *et al.* reports a method for the dissolution of urban materials such as Mixed Analyte Performance Evaluation Program (MAPEP 24) and asphalt, acting as surrogate material [56]. He reports a NaOH fusion process wherein 1g samples are placed in a graphite crucible with 15g of solid NaOH, heated for 15min, and allowed to cool for 10min. The samples are then transferred to a hot plate and H₂O was added as needed. After partial dissolution, samples are cooled to ~0°C and the suspended solid residues are separated from the liquid by centrifugation at 3,600rpm for 6min. The solid residue is then dissolved with 60-80mL of 1.5M HCl and diluted to 170mL with 0.01M HCl. After dilution, the samples are treated with 25mL of 28M HF, mixed, and allowed to stand for 10min prior to centrifugation. The supernatant liquid is kept, and the remaining solids are dissolved in 5mL of 3M HNO₃ – 0.25M H₃BO₃, 6mL of 7M HNO₃, and 7mL of 2M Al(NO₃)₃. This method shows success with select actinide separation and recommends methods for fission product separations. The separations for this dissolution method are discussed in the subsequent section. To date, the dissolution of trinitite has seen success; however as new surrogates are developed to replicate trinitite and other weapon scenarios, challenges will likely arise and persist with forensically relevant dissolution methods. Unfortunately, present nuclear threats are not likely to produce debris resembling the Alamogordo desert, which is the site of the Trinity test [57].

B. Gas-phase Separations

A major method of interest is founded upon the methods of chromatographic chemistry, largely utilized by the super heavy element community [58, 59]. It is proposed by Hanson *et al.* and Garrison *et al.* that thermochromatography could be employed as a separation technique for nuclear forensics [60, 61]. Following this work, Auxier *et al.* reported the use of a variety of ligands that could be used to synthesize volatile organometallic compounds, specifically of lanthanides, for rapidly separating and detecting useful rare earth signatures [62, 63]. In this work, the organic compounds 1,1,1,5,5,5-hexafluoroacetylacetone, 2,2,6,6-tetramethyl-3,5-heptanedione, and 6,6,7,7,8,8,8-heptafluoro-2,2-dimethyl-3,5-octanedione combined to produce 7 and 9 coordinate compounds that volatilize at temperatures ranging from 140 to 220°C. These complexes show promise in a rapid separation technique using a coupled gas chromatography - ICP-TOF-MS for elemental and isotopic identification and quantification, as well as a separation step for other measurement methods.

C. Mass Spectrometry Interferences

Sharp *et al.* reports that prior to mass spectrometry analysis, rare earth interferences can be removed using a 12cm × 2cm Dowex AF50W × 8400 mesh cation exchange column in the H⁺ form, followed by treatment with both 2.5M HCl and 4.5M HNO₃, respectively. The individual lanthanides Nd and Gd are removed using a Dowex AG50W x 8 (30 × 5 cm) column pre-treated with NH₄⁺ to remove the H⁺, with subsequent elution using α-HIBA (α-hydroxyisobutyric acid) at a pH of 4.7. The cuts are dried and dissolved in 0.8M HNO₃[55]. Eppich *et al.* [64] reports a similar process prior to performing analysis, wherein the matrix elements are removed using a 1.8mL U-TEVA ion exchange resin conditioned with 10mL of 4M HNO₃. The samples are loaded onto the resin and washed with 5mL of 4M HNO₃, 3mL of 9M HCl, and 4mL of 5M HCl. The U remaining on the column is removed with 8mL of 0.1M HCl, then dissolved in 0.1mL of concentrated HCl. It is also reported that the matrix elements can be removed using a 1.8mL AG-1 X 8 (100-200 mesh) anion exchange resin conditioned with 1 mL of 9M HCl. The samples are loaded onto the column and washed with 7mL of 9M HCl. The uranium is eluted with the addition of 8mL of 0.1M HCl. The samples are dried down and dissolved in HNO₃. Further pre-treatment is reported

prior to performing isotope dilution mass spectrometry (IDMS) techniques. Due to the complexity of pre-treatment, most researchers have resorted to using LA-ICP-MS in place of chemical separations.

VI. Conclusion

Modern research in nuclear forensic technology continues to address the timeliness and accuracy of post-detonation forensic analysis techniques toward an effective *in extremis* national security capability. As a vital component of the attribution process, it is critical for the TNF community to remain abreast of revolutionary technology to provide increasingly accurate and timely data into the attribution cycle. Evolving analytical approaches toward the rapid analysis of post-detonation materials is essential, and several works presented here have generated innovative solutions to the challenges posed by nuclear forensic science. It is imperative for progress to continue down its current path in support of a robust and rapid analytical TNF capability.

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VIII. Authors' Bio and Contact Information

Adam Stratz, works under Dr. Howard Hall as a PhD Candidate in the University of Tennessee Radiochemistry Center of Excellence and is Department of Homeland Security Nuclear Forensics Graduate Fellow. His research focused on the effects of climate change on fundamental hydrologic control variables used in the design of major hydraulic dams, projection of extreme precipitation patterns in dynamic climate systems, and dam fortification for post-construction developments in probable maximum precipitation patterns. Adam is currently pursuing his PhD in Nuclear Engineering from the University of Tennessee. Preliminary research efforts were focused on the detailed development of urban nuclear melt glass matrices and fabrication techniques to propagate the effective generation time of resulting laboratory samples. Accurate formulation techniques were developed for citywide applications.

His current research focuses on rapid radiochemical separations of fission and activation products from nuclear detonation debris. Gas-phase chemistry is exploited to develop and improve separations, with a particular emphasis on faster and higher specificity separations. A key research objective is to better constrain the thermodynamic data regarding the interaction of the gas-phase species with the separation column in order to model and optimize a large-scale matrix separation.

Contact Information: sstratz@vols.utk.edu

Jonathan Gill is a PhD candidate and graduate research assistant for Dr. Howard Hall at the University of Tennessee with the Radiochemistry Center of Excellence. He received a bachelors of science from the United States Military Academy in Nuclear Engineering (2005) and a masters of science from the Missouri University of Science and Technology in Engineering Management (2010). During his undergraduate study he completed a summer internship at Lawrence Livermore Laboratory investigating executive decision making regarding nuclear weapons use. Upon commissioning from the Military Academy, he served as an engineer officer in the United States Army from 2005 to 2013. During his service, he led a construction engineer platoon to combat in Iraq and a sapper engineer company to combat in Afghanistan, earning bronze stars during deployments for his accomplishments in combat. Jonathan completed his service as a strategic planning officer for US Army construction operations in Europe, Western Asia, and Africa. Jonathan returned to graduate school after completing his service in 2014 and earned a master of science in Nuclear Engineering from the University of Tennessee (2015) researching acidic and caustic dissolution techniques for trinitite and various nuclear weapons debris surrogates. Jonathan is currently conducting his PhD research on single ion detection in solid state nuclear track detectors for heavy charged particles. His PhD research is in collaboration with BWX Technologies Inc. and the University of Tennessee.

Contact information: Email: jgill14@utk.edu

John D. Auxier II is currently a research assistant professor at the University of Tennessee (UTK) in the nuclear engineering department and the Institute for Nuclear Security. He also holds joint positions with Y-12 National Security Complex, Oak Ridge National Laboratory, and Los Alamos National Laboratory (LANL). Prior to this effort, he worked as a Post-Doc under Dr. Howard Hall in standing up the National Nuclear Security Administration (NNSA) Radiochemistry Center of Excellence (RCoE) at UTK, which focuses on the development of novel nuclear forensic methods for post-detonation scenarios. Dr. Auxier received his Ph.D. from UTK with research efforts that focused on the development of materials for thermal neutron detection, and research efforts at LANL that focused on development of alpha spectroscopy development software. His current interests involve research in nuclear forensics, radiochemical separations, and radiation detection applications. As part of this research he has published numerous scientific articles on nuclear forensics and development of radiation detection material, along with a patent on rapid separations. He has been awarded a number of grants from sponsors including DHS, DTRA, and DOE NNSA in areas related to nuclear forensics and analysis.

Contact information: jauxier@utk.edu

Dr. Howard Hall is the University of Tennessee and Oak Ridge National Laboratory Governor's Chair in Nuclear Security. He is a Senior Fellow in Global Security Policy at the Howard H. Baker Jr. Center for Public Policy, where he directs the Global Security Policy Program. He also directs the UT Institute for Nuclear Security. Before joining UT, he spent 20 years at Lawrence Livermore National Laboratory. His research interests include nuclear security applications at the intersection of science, security, and public policy. Professor Hall is a member of the American Nuclear Society, the American Chemical Society, and the Institute of Nuclear Materials Management. He is a Fellow of the American Association for the Advancement of Science and the American Institute of Chemists.

Contact information: hhall6@utk.edu