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Case Studies of a Nuclear Forensics Examination

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Abstract

States are increasingly using nuclear forensics as a preventive and response to counter the threat of nuclear and other radioactive material out of regulatory control. A review of published nuclear forensics case studies through the lens of the nuclear forensics model action plan draws insights from recommended international guidance on the conduct of an examination to its actual practice in the field. Diverse case studies demonstrate that nuclear forensics is successfully applied to mitigate risks from diverted materials, prosecute those responsible for malicious acts, as well as inform and improve nuclear security practice globally.

Key Words

Nuclear forensics, nuclear forensic case studies, nuclear security investigations, nuclear forensics model action plan, national response plan

Introduction

Case studies provide insight into the effectiveness of a nuclear forensic examination in response to incidents involving nuclear and other radioactive material encountered out of regulatory control (MORC). These materials constitute a threat involving a diverse array of radioactivity originating from the civilian and military nuclear fuel cycle as well as radioactive sources used in science, industry and medicine. These examinations are as varied as the individual incidents and materials involved. Despite these differences, case studies provide crucial insight into the efficacy of nuclear security measures, including nuclear forensics, designed to protect these materials.

Outcomes from case studies can inform improvements to the nuclear forensics state of practice and subsequent examinations and are important because they offer objective lessons that illuminate the nature of the threat as well as procedures taken to secure the materials (1, 2, 3). As such they are illustrative of the many complexities, infrastructure, and expertise that is required to implement a nuclear security investigation.

The purpose of this chapter is not to exhaustively review individual nuclear forensic case studies in detail but rather to use diverse published case studies to extract lessons learned and assess the strength of nuclear security measures designed to prevent and respond to the diversion of MORC. An additional objective is to evaluate whether nuclear forensics in support of investigations, including the initial response to a nuclear and radioactive material out of regulatory control, provides states a means to better meet their nuclear security responsibilities.

Nuclear Forensics as a Security Preventive and Response

Nuclear forensics as a discipline of forensic science has emerged globally in the prevention and response to incidents involving MORC. Incidents in central and eastern Europe involving the interdiction of up to kilogram quantities of fissionable uranium and plutonium began to be reported after the former Soviet Union disbanded at the end of 1991. Concerns regarding high consequence terrorist acts involving nuclear material were heightened after the 11 September 2001 attacks in the United States of America (4). In parallel, international concerns mounted about the security of large inventories of fissionable materials in the Commonwealth of Independent States, and elsewhere, as well as the urgency to best secure these materials in production, use and storage. Scientists and law enforcement officials came together in the mid-1990s to address technical questions surrounding seized, but unattributed, nuclear material (5). These experts recognized that nuclear science, radiochemical and analytical skills maintained during the Cold War by the nuclear weapons states, and utilized in nuclear weapons production and underground testing, could also be adapted to provide insight into the origin and history of smuggled or poorly secured nuclear materials.

Inherent to a nuclear forensic examination are measurements of the physical characteristics (e.g., density, color, grain size) and the chemical properties (e.g. isotope composition, major and trace elements) of the material under examination as they can provide information on its geologic and manufacturing history (e.g. isotopes separated for enrichment and fissionable nuclear fuels) (6, 7, 8).

A review and an assessment of case studies is made in the context of a definition for nuclear forensics supporting nuclear security and law enforcement investigations. Nuclear forensics, as a subdiscipline of forensics science, involves the examination of nuclear or other radioactive material, including evidence contaminated by radionuclides, in the context of national or international legal instruments related to nuclear security (7). A priority are nuclear forensic examinations that support successful potential criminal prosecutions under national laws. These require that evidence returned from nuclear forensic examination, in a majority of cases, must be admissible to a court of law. To meet this requirement, an examination plan acceptable to law enforcement and judicial officials is necessary.

The Lens of the Model Action Plan: A Guide to Nuclear Forensic Examinations

In 1995, to address international concerns overs reports of illicit nuclear trafficking, the Nuclear Smuggling International Technical Working Group was founded. Later renamed the Nuclear Forensics International Technical Working Group (ITWG), this informal, unaffiliated association of nuclear scientists, law enforcement officials, nuclear regulators, forensic science specialists, and nuclear security professionals has focused on sharing best approaches in the areas including guidance for the conduct of a nuclear forensics examination, nuclear forensics evidence collection, implementation of interpretative and analytical collaborative material exercises, outreach, as well recent results of the work of international nuclear forensics laboratories (9, 10).

One of most significant contributions since the founding of the ITWG has been the formulation of the nuclear forensics model action plan as a guide to the conduct of a nuclear forensic examination. This plan was embraced by the IAEA in the formulation of its guidance to Member States and originally published in 2006 and revised in 2015 as part of its nuclear security series (7).

In this discussion of published nuclear forensics case studies, the review is facilitated through the common lens of the model action plan. The evaluation follows the sequence of a generalized plan for examination. While nuclear forensics includes the analytical aspects of an investigation, the model action plan also incorporates the initial safe and secure response to incidents of MORC to include the management of a radiological crime scene as well as the recovery of evidence (Figure 1).

Figure 1. The principal elements of the nuclear forensics model action plan (7). Photographs from author.

The plan specifies a generalized approach to an examination of the traditional evidence recovered at a radiological crime scene (including fingermarks, DNA, digital evidence, fibers, explosive residues) as well as nuclear forensics evidence (nuclear and other radioactive material as well as traditional evidence contaminated by radionuclides) Often traditional forensic and nuclear forensics analysis are done in parallel by separate specialized laboratories. As each case is unique, planning and the resulting implementation of forensic examinations will each be different.

Nuclear Forensic Categorization and Characterization as Part of a Nuclear Security Investigation

The initial stages of incident response to a radiological crime scene involve protection the public and the integrity of potential evidence. Initial categorization at the radiological crime scene involves determination of the nature and extent of radioactivity above background; the specific radionuclides present (e.g. 239-plutonium, 238-plutonium, 235-uranium, 192-irdium, 137-caesium, 90-strontium, 60-cobalt and others) and their dose rate (e.g. micro-sievert or milli-sievert/hour) (11). This information will inform access to the radiation area, health physics considerations, as well as considerations for safe handling, packing, and transport of the evidence to the appropriate laboratory or laboratories for further analysis. The forensics examination plan involves collection of evidence contaminated by radionuclides as well as evidence that is not contaminated and might be encountered outside the radiation cordon (12). Crucial is establishing an unbreakable chain-of-custody that documents the control over the sample to include transfer of the samples sequentially from the point of collection through their ultimate disposition.

Nuclear forensics analysis and interpretation involves measurements of material characteristics, or signatures, comprising isotopes, trace elements and physical properties that are indicative of their geologic and industrial origins (6, 7, 8). Analyses are tailored to the meet requirements set by the lead investigator. Because the laboratory results may be used in legal proceedings or to provide information on the effectiveness of the nuclear regulatory regime, careful handling of evidence as well as a clear understanding of the confidence levels for the findings resulting from the analyses is paramount. Nondestructive analyses are typically prioritized first before destructive analyses involving sample digestion. Gamma spectrometry of radioactive material (e.g. 137-caesium, 60-cobalt), coupled with physical determination of sample weight, density, morphology and molecular composition using x-ray techniques can provide an abundance of information in the initial stage of a nuclear forensics examination. Subsequent characterization involving digestion or destruction of a solid sample is typically required for the mass spectrometric measurements of the isotope ratios to determine diagnostic sample properties such as the enrichments in uranium, plutonium and transuranic metals and oxides, the decay of radiogenic parent isotopes to their daughters to determine the “age of materials” (i.e. their date of last radiochemical purification), and measurement of trace elements concentrations (at dilute levels of parts per billion – nanogram per gram - or lower) that can sequester signatures of processing steps.

As findings are returned from the examination and are interpreted, a national nuclear forensics library, or a network of laboratories, maintained by the state may be used to identify the origin and history of MORC (13). The library contains reference information and access to expertise in nuclear or other radioactive material used, produced or stored within the state aiding in the comparison of samples. In particular such a library is helpful to indicate whether diverted materials originated within the state or elsewhere.

Categories of Nuclear Forensics Case Studies

Nuclear forensics case studies are organized in the frame of categories recognized by the International Atomic Energy Agency’s Incident and Trafficking Database (ITDB) which compiles confirmed incidents reported by Member States (14). These incidents involve: malicious use or illicit trafficking involving nuclear or other radioactive materials, incidents with insufficient information on whether the incident is connected or not to malicious use or illicit trafficking, and incidents where there is sufficient information to determine incidents are not connected to malicious use or illicit trafficking. All cases discussed here were reported by states to the ITDB.

The High Enriched Uranium (HEU) Seizures in Bulgaria, France and Moldova

The most prominent series of cases involve the seizures of highly enriched uranium in Ruse, Bulgaria in 1999, in Paris, France in 2001, and in Chisinau, Moldova in 2011. These cases are unique because of the examinations were informed by the model action plan and a significant amount of information on the ensuing nuclear forensics examination was subsequently published (15, 16, 17). Nuclear forensic findings strongly suggest not only was the isotopically enriched uranium oxide analyzed in the three cases similar nuclear material, but other evidence to include packaging and shielding used to smuggle this material was comparable. The further significance is the more than ten year time span between the cases which suggests illicit trafficking of these materials involve organized and lasting networks of sellers and buyers extending from western

Europe to the Black Sea region. The technical and law enforcement response to these cases informed subsequent case work, with particular emphasis on the role of the involvement of the law enforcement partner and preparedness through a response plan to nuclear and radioactive materials encountered out of regulatory control.

The Ruse, Bulgaria Seizure in 1999

The 29 May 1999 seizure of 4 grams of 72.2 atom per cent enriched uranium-235 as uranium oxide particles in Ruse, Bulgaria on the Danube River has been well documented as has the ensuing nuclear forensics examination of this HEU both in Bulgaria as well as within the United States of America (18, 19). A sealed transparent glass ampoule containing the HEU was recovered by alert Bulgarian border officials from a vehicle driven through a checkpoint. Once recovered, the HEU was only one component of a diverse inventory of evidence that was subsequently exploited by investigators in this case.

The response to the Bulgarian seizure underscored the many opportunities available to forensic examiners. Evidentiary and investigative streams could be divided into three general categories: the vehicle and the driver transporting the HEU, the packaging and related evidence not involving the HEU, and the nuclear material itself. Interviews with the driver indicated that the material had transited south along the western edge of the Black Sea where the driver then headed to Turkey to potentially sell this material to an interested buyer. When the sale fell through, the driver transited back along the same western Black Sea route before being stopped by officers during the night at the Ruse checkpoint. The driver displayed a suspicious demeanor during the on scene interview. A subsequent search of the vehicle returned an abundance of clues to include written paper documentation relevant to the smuggled material, as well as a cast lead cylinder filled with yellow paraffin wax that was shielding a glass ampoule containing approximately 4 grams of fine black uranium oxide powder. Categorization of the material and other evidence by the Bulgarian authorities were expanded. The sample was then transferred to national laboratories in the United States.

Using gamma spectroscopy and optical measures, initial categorization by the Bulgarian authorities confirmed the material consisted of uranium oxide particles with an enrichment in excess of 20 atom per cent uranium-235. These measurements confirmed this was a high priority nuclear smuggling case. In 2000, the Bulgarian authorities commenced a bi-lateral partnership with the United States government to characterize the uranium using technical assets and expertise at its national laboratory complex. Analyses indicated the uranium oxide was also enriched in uranium-236 to 12.1 atom per cent as well as with trace quantities of plutonium. Residual fission products of antimony-125, cesium-134 and cesium-137 were detected. Sulfur, bromine and chlorine were also elevated. Radiochronometry provides the time since the last chemical purification of the sample where radioactive parents are separated from their decay daughters (e.g. natural decay of uranium-235 to protactinium-231, decay of plutonium-241 to americium-241 and others); in this instance the time of last separation was determined to be October 1993 (measurement uncertainty of 50 days). Taken together, these findings indicate the seized material was derived from a reprocessing enterprise involving nuclear fuels.

The examination of evidence returned from the Bulgarian seizure also included the non nuclear, or traditional evidence to include documentary evidence, lead shielding, the yellow wax and a glass ampoule containing the HEU. These technical results include a 'billing of lading' collected from the vehicle and written in Cyrillic, as well as indicates the lead container for shielding was hand fashioned, the yellow paraffin wax contains a concentration of barium chromate colorant not common to North America, and the glass ampoule was indicative of industrial scale production. Taken together, these findings suggest the material was diverted from an industrial processing facility exclusive of a North American origin and likely from within the former Soviet Union (17).

The smuggler was tried and convicted in a Bulgarian court (18); due to the absence of national laws in Bulgaria that prohibited the unauthorized possession, use and transport of nuclear materials at the time of the seizure and trial, the judiciary only sentenced the perpetrator to violating customs formalities and the individual was released from jail for time already served with a reduced fine.

The Paris, France Seizure of 2001

In July 2001, French authorities reported on arrests and the seizure of 468 milligrams of highly enriched uranium in Paris (20, 21). In this case, an arrest was made for the unauthorized possession of gram sized quantities of high enriched uranium oxide powder offered for clandestine sale which was detected during an inspection of a motorized transit van involved in the smuggling operation. The uranium sample was contained in a glass ampoule secured within a shielded lead container lined with a bright yellow paraffin wax. Both the uranium oxide sample and the packaging materials were analyzed by France's Commissariat à l'Energie Atomique for forensic evidence.

The isotopic abundance of the material seized by the French was 72.6 atom per cent uranium-235 and 12.2 atom per cent uranium-236. Trace concentrations of plutonium were also detected. The French examiners reported the radiochemical date of last purification was November 1994 (with a measurement uncertainty of 100 days) and note similarities between their results and those reported after the Bulgarian analyzes with the suggestion that the two analyzed materials have a similar origin (21).

Analyses of the packaging material had remarkable similarities to the material seized in Bulgaria. The lead container was fashioned almost identically to that recovered in the Bulgarian case, is enriched in antimony, the protective yellow wax encompassing the sample vial was enriched in barium chromate and the shape of the glass ampoule mimicked that seen in Bulgaria (20). The comparative evidence returned by examiners in these two cases implicates the transboundary nature of illicit trafficking (17, 20).

Investigations suggested that the nuclear material was smuggled through the Black Sea region on its way to Paris and, like the Bulgarian case, involved reprocessed spent nuclear fuel originating in Russia (17). In 2003, the smugglers in this case were brought to trial in France.

The Chisinau, Moldova Seizure of 2011

Of significance for investigations of nuclear trafficking, and the utility of nuclear forensics as a response to these incidents, was the June 2011 seizure of 4.4 grams of high enriched uranium oxide powder in Chisinau, Moldova. As part of a sting operation involving arrests to counter the illicit trafficking of nuclear material, evidence collected by law enforcement officials included a lead container, paraffin shielding, a glass ampoule, and fine grained uranium oxide powder remarkably similar to those recovered in Bulgaria and France. Subsequent nuclear forensic analysis of this uranium by the European Commission's Joint Research Centre in Karlsruhe, Germany indicate the uranium particulates intercepted in Chisinau have a uranium-235 enrichment of 72.7 atom per cent and a uranium-236 enrichment of 12.1 atom per cent (16, 22). Age dating of swipe sample of uranium oxide indicates a purification date of December 1992 (measurement uncertainty of 1.0 year) (22). Based on the many analytical parallels between the traditional and nuclear forensic evidence in the Bulgarian, French and Moldovan cases, taken together, these investigations conclude the materials involved a similar origin. The smugglers were later convicted for the unauthorized possession of nuclear materials.

Lessons Learned from the Bulgaria, French and Moldovan HEU Cases

The forensics examinations performed on traditional as well as nuclear forensics evidence, as well as the subsequent publication of findings from all three cases, highlight the crucial role nuclear forensic science plays in investigations of cases including trafficked high enriched uranium.

The nuclear forensics findings of the three cases indicate the materials involved are similar. The isotopic abundance measurements of the high enriched uranium, determinations of the time (age) of the last radiochemical purification, the documentation of the uniform and nanometer grain size of the uranium oxide powder, as well as the traditional forensics examination of the confiscated lead containers, the bright yellow paraffin wax within owing to the presence of barium chromate, as well as the manufacture of the sealed glass ampoule containing the uranium powder are all consistent sample-to-sample and suggest a common origin from a nuclear fuel fabrication or reprocessing facility. For analyses of smuggled uranium, access to mass spectrometry was important to provide the required accuracy and precision to compare measurements of isotopes of uranium-234, uranium-235, uranium-236 and uranium-238 measured in these 3 cases. As well, differences in the radiochemical age of purification may be interpreted as differences between production batches. Comprehensive scientific forensic studies involving the origin of the material analyzed in Bulgarian seizure with links to the French and Moldovan cases indicate an exact source of this material remains inconclusive but a North American origin can be discounted (18).

The role of law enforcement in these cases cannot be underestimated. In particular, the Bulgarian case commenced due to the vigilance of an alert Bulgarian border guard who was suspicious of the driver transiting north to Romania and who took appropriate action to include detaining the driver for additional questioning, as well as initiating a search of the driver and his vehicle. The role and preparedness of law enforcement was also crucial in the French and

Moldovan cases to disrupt the attempted sale of these materials (18, 21). The response to each of these cases was predicated on prior knowledge of national forensic capabilities to fully exploit both the nuclear as well as the traditional evidence consistent with requirements of the lead investigative authority. Additionally, in the Bulgarian and Moldovan cases, bilateral arrangements facilitated the sharing of the confiscated samples and the full characterization of the uranium oxide powder in nuclear forensic laboratories of the United States and the European Union.

The seizures of gram-sized quantities of nuclear material were far less than the published significant quantity of 25 kilograms of uranium-235 in high enriched uranium required to construct a fissionable nuclear device (23). However, these smaller quantities do provide insight into the smuggling enterprise and in particular emphasize vulnerabilities associated with the physical protection and detection of smaller samples and the response to interdictions of these materials. In these and other cases, the seized gram sized nuclear materials were represented by smugglers as examples of much larger inventories of nuclear materials that might be transferred later. Nevertheless, these cases indicate that similar nuclear material was misappropriated as a result of deficiencies in the nuclear security regime for more than a decade (from 1999 to 2011), or perhaps even longer. Together, these case studies teach investigators not to treat individual cases in isolation, but to share results, as possible, and compare the findings between the individual cases, more broadly. The comparative aspects between the three cases is an additional endorsement for states to collaborate internationally and to establish a national nuclear forensics library to help in the identification of nuclear material under their jurisdiction (13).

The traditional forensics evidence returned from these three cases was extremely useful to the goals of the investigation. In each, those responsible for the trafficking of this material used a lead container to shield the material from detectable radiation emissions, a bright yellow paraffin wax to cushion the sample and crafted sealed glass ampoule to contain the material. This evidence suggests similar, careful and extensive preparations went into the diversion and smuggling of these samples. For the purposes of the investigation, responders and examiners must look broadly at the entirety of evidence available. Exploiting traditional forensics (e.g. fingerprints, digital evidence) is powerful in linking a suspect to MORC.

That the samples from the 3 cases were encountered out of regulatory control remains worrisome. The samples disrupted the potential transfer of these materials between sellers and buyers who recognized these materials were rare, of strategic importance, and could be used for malicious purposes. The smugglers were organized in their approach and methods. The similarity and timing of these three cases suggests the presence of a network of sellers, middlemen and buyers, possibly working together that extended over many years. The presence of buyers for this uranium has implications for nuclear terrorism (17). Less certain is whether the highly enriched uranium in the three cases was diverted out of regulatory control during a single episode, or over a longer period of time.

Response to the Loss of a Cobalt-60 Source in Mexico

A 60 gram, 95 terabecquerel cobalt-60 source located within the head of a teletherapy source belonging to a hospital and being transported by a truck transiting from Tijuana to a repository site in central Mexico was stolen by armed gunman in December 2013. The medical radiotherapy source and shielding was removed from the truck in the town of Hueypoxtla more than 40 kilometers from where the truck was first commandeered. The source was extracted from the shielding and moved. Elevated radiation levels were detected in the vicinity using vehicle borne radiation detectors. The entire site was subsequently secured by the Mexican authorities and a search, using a robot, ensued for the exact location of the cobalt source. Two days after the theft, the Mexican authorities informed the public about the radiation hazards associated with proximity to or handling of the source. The source was subsequently identified in an agricultural field approximately a week after the theft and was secured in a shielded container provided by the authorities using the robotic technology (24). Ten individuals with proximity to the source, to include one person who assisted the Mexican authorities in locating the source, were thought to be exposed to gamma radiation and necessitated assessment for acute radiation exposure (24). Biodosimetry indicated this one person exceeded the Mexican regulation for radiation exposure (500 mSv annual whole body effective dose). The individual required medical intervention. Criminal cases resulted in the arrests of individuals responsible for the theft.

Lessons Learned from the Response to the Loss of a Cobalt-60 Source in Mexico

The Mexican case underscores the importance of a national response to criminal acts involving MORC as well as the management of a radiological crime scene. Other than nuclear forensics categorization, the response involved recovery and re-establishing regulatory control over the source without extensive nuclear forensics laboratory analysis. The radiation levels encountered were extremely high; this was a Category 1 source which is defined by the IAEA as most dangerous to human health (25). Responders and examiners must be prepared to re-secure the source despite the presence of high levels of radiation. The case further reveled how a criminal act involving the theft of a truck ultimately had grave implications for the security and safe handling of radioactive materials. The exact motives of the gunman were unclear but likely involved their intention to steal the truck without a broader plot to weaponize the cobalt-60. However, organized and sophisticated criminal activity involving gangs and cartels throughout the region remains a concern and the outcomes thereof could have implications for potential terrorist acts (26). A study of the case also points to the particular vulnerabilities associated with local transport of highly radioactive material within the state; while the cobalt-60 source was secured within the confines of the approved facility, once it left by truck, the source became susceptible to criminal acts that resulted in the radioactive source being diverted out of regulatory control (27). For this reason, the case emphasizes the necessity of maintaining the integrity of nuclear security measures throughout its lifecycle.

The case also underscores the need for responders to address evolving nuclear security cases that occur at different locations and over weeks of time involving high levels of radioactivity. The case was also unique as it involved many different national entities to include the national nuclear regulatory body, civil protection agencies, the federal police, the military, nuclear plant operators, as well as the national institute for nuclear research. Due to the extraordinarily high levels of radiation encountered, field categorization involved the use of robots and remote area surveys to locate the source. This case required intervention by medical practitioners to survey for acute radiation exposure as well as provide bioassay. Taken together, lessons learned have implication for the development of a national response plan to a nuclear security event.

Nuclear Forensics Examinations of Contaminated Metal Scrap

Nuclear and other radioactive material often gets inadvertently incorporated in the salvaging streams of metal recyclers. Sealed cobalt-60, iridium-192, caesium-137 gamma-emitting sources are commonly used in the oil refining and manufacturing industries and can be lost or improperly disposed along with benign excess or waste metal (28). Scrap metal is a global and profitable business that continues to be a lucrative market driven by higher prices for raw materials sourced in recycled materials and reflective of demands by the global supply chains during the viral pandemic beginning in 2020. The problem of scrap metals contaminated with radioactive sources and materials persists (29, 30). While cases involving contaminated scrap metal do not involve malicious intent, they do underscore deficiencies in the nuclear security regime that allows these materials to be diverted out of regulatory control and be transported to scrap metal recycling yards. The principal concern is removing radioactive contamination from the waste stream before it is smelted; the cost to a recycler to decontaminate and clean a metal smelter is prohibitive. Despite these precautions, there are reported incidents of radionuclides (to include cobalt-60) that have made their way into recycled household goods (28). For this reason, radiation portal monitors are outfitted at many scrap yards to screen entering haul trucks; scrapyard handling claws affixed to crane manipulators are also fitted with gamma detectors. Nuclear forensic examinations have been conducted to subsequently categorize and characterize the radioactive materials encountered in scrap to determine the inherent radiation profile and any potential threat, how these sources can be handled and secured, as well as provide insight into their history.

Beside sealed gamma emitting sources, highly enriched uranium has been discovered within contaminated steel scrap sent to European recyclers. The HEU is affixed as powders, solutions or gases to the surfaces of the host metal (31). Nuclear forensic analyses of contaminated samples using both non-destructive and destructive bulk and micro-analytical techniques indicated variable enrichments that were the result of the inhomogeneity of the distribution of uranium in the samples (32). The highest uranium-235 enrichment in these metal scrap was 28.3 weight per cent measured in bulk samples by gamma spectroscopy with the corresponding highest uranium-235 enrichment of 25.9 weight per cent measured in bulk samples by thermal ionization mass spectrometry. Individual uranium particles had a maximum enrichment of 94.4 weight per cent uranium-235 using spatially-resolved laser ablation inductively coupled plasma mass spectrometry (32). The detection of uranium-236 in these samples is indicative that the samples were reprocessed.

Lessons Learned from Nuclear Forensics Examinations of Contaminated Scrap Metal

Nuclear forensic examination of contaminated scrap metals highlights the role of industry working in concert with national regulators to ensure that nuclear and other radioactive materials are detected and secured. Although nuclear security is a national responsibility, working with national regulators, industry has a financial incentive to utilize nuclear forensics to categorize radioactive materials and keep them out of recycled metal product streams to alleviate contamination of the manufacturing process and final products. The recycling yards serve as 'choke points' where larger quantities of metals can be aggregated and routinely surveyed and nuclear and other radioactive removed and secured. The awareness and preparedness of recyclers and regulators to the threat has resulted in the removal of dangerous materials from metal scrap and heightened measures to prevent future incidents. Nuclear forensics also plays a role. The outcome of these examinations illustrates lapses in the originator's regulatory regime where radioactive sources and other radionuclides have become inadvertently incorporated with metals bound for recycling yards as well as shortcomings in transport during which time these offending radioactive materials are shipped without detection to a recycler. In the case of the nuclear materials encountered by European recyclers, scrap streams blend lots of metal from many different sources, and forensics findings represent this extreme heterogeneity, the uranium-235 and uranium-236 enrichments, as well as other data characteristics, have proven useful to determine the processing history of the uranium out of regulatory control to include distant points of origin.

Nuclear Forensic Case Studies and Nuclear Security

These cases focus on both the challenges and opportunities in conducting a nuclear forensics examination as well as provide essential lessons learned that can improve the state of practice. Most importantly, results from examinations provide demonstrated confidence in the conduct of nuclear security investigations to include criminal prosecutions. While information concerning incidents of MORC, and the associated results of nuclear forensic examinations is typically managed and controlled by the state, the results directly inform broader nuclear forensic methodologies as well as contributions to a national response plan. When this information can be shared, outcomes from case studies further benefit regional and international partners who may need to respond to similar threats using the tools of radiological crime scene management and nuclear forensic science as capabilities to meet their nuclear security responsibilities.

Utilization of Nuclear Forensic Case Studies in Nuclear Security Investigations

Early cases emphasized the crucial role of law enforcement officials who, in the case of the Bulgarian seizure on 1999, thoroughly questioned and searched an individual who was behaving suspiciously. Follow-on cases in Paris in 2001 and Chisinau in 2011 involved intelligence driven investigations where sting operations and information alerts allowed nuclear materials to be intercepted.

The threat of nuclear and radioactive material is ever present with over 100 incidents being registered each year by the IAEA's ITDB. Over the past two decades, lessons from diverse case studies emphasize the crucial role of a national response plan to a nuclear security event. Movement of MORC occurs regionally as well as internationally and involves different jurisdictions. In the Mexican example, the cobalt source was recovered in a rural agricultural setting that necessitated remote recovery operations including specialized robotics the result of high levels of gamma radiation. Additionally, medical intervention was also required. Collectively the ability of first responders, regulators, law enforcement officials, health and environmental specialists and transport experts working in a coordinated and comprehensive manner was essential to the response. These cases also point to the need for responders to request and receive specialized assistance that allows high confidence in the material identification and the nature of the threat to include use of instrumentation to detect radionuclides, protect the health and safety of responders and securely collect nuclear forensics or traditional forensic evidence. The ability to categorize evidence in the field and develop a forensics examination plan is a necessity to include the receiving laboratory where the ensuing examination and necessary characterization will be conducted.

Nuclear Forensic Casework to Link People, Places, Materials and Events

Nuclear forensics case studies yield information on those individuals, groups or networks who would possess, use or store nuclear and other radioactive materials for malicious purposes, as well as the nature of the materials that are stolen or trafficked. Nuclear forensic case studies provide confidence to examiners, law enforcement officials and the judiciary that nuclear forensics is a viable and effective tool returning findings that support the criminalization of unauthorized acts.

Recent nuclear security investigations involving nuclear forensic examinations in Romania emphasize 'a change in response strategy by the national authorities' (33). A more than two-fold increase in nuclear forensic case work within Romania reflects the intention of prosecutors to use evidence returned from a nuclear forensics examination to prosecute those responsible for unauthorized acts involving radioactive sources, nuclear materials and traditional evidence contaminated with radionuclides. The increased case load, however, is not indicative of a higher volume of trafficked materials but rather highlights increased national awareness of, and confidence in, the legal procedures required in response to a nuclear security event (33). Through the shared experience of examiners and law enforcement officials in Romania, nuclear forensics provides defensible evidence leading to convictions in the court room.

Case work is the ultimate test of nuclear forensics as a preventive and response to the serious threat of MORC. These studies inform the international community that nuclear and other radiological material continues to be diverted outside of regulatory control; the studies provide information of what the material is, what threat it may pose, as well as its origin and history. When shared, the cases allow direct comparison of materials one to another; this was the example from the Bulgarian, French and Moldovan nuclear forensic examinations that collectively spanned more than a decade (14, 15). Without compromising sources and methods that may need to be protected as part of an investigation, dissemination of conclusions from case studies demonstrates the successful conduct of the model action plan encompassing a nuclear forensics investigation leading to successful prosecutions of smugglers. For this reason, more nuclear forensic cases need to be publicized internationally. This outreach also demonstrates the availability of nuclear forensics as a key tool to assist states in meeting their nuclear security obligations. In this regard, by linking materials to people, places and events, nuclear forensics can further serve as a preventive measure to deter those who might contemplate unauthorized acts as well as remedy deficiencies in physical protection of detection measures that allowed this material to be diverted. Findings from case studies continue to strengthen the nuclear security regime globally.

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References

1) Christopher Hobbs and Matthew Moran, *Insider Threats An Educational Handbook of Nuclear and Non-Nuclear Case Studies*, (London: King's College London, 2015).

2) Geoffrey Chapman, Robert Downes, Christopher Eldride, Christopher Hobbs Luca Lentini, Matthew Moran, Alberto Muti and Daniel Salisbury, *Security Culture An Educational Handbook of Nuclear and Non-Nuclear Case Studies,* (London: King's College London, 2017).

3) Wyn Bowen, Matthew Cottee, Christopher Hobbs, Luca Lentini, Matthew Moran, Sarah Tzinieris, *Nuclear Security Briefing Book, 2020 Edition,* (London: King's College London, 2020).

4) Graham Allison, *Nuclear Terrorism The Ultimate Preventable Catastrophe*, (New York: Henry Holt and Company, LLC, 2004).

5) Sidney Niemeyer and Lothar Koch, 'The Historical Evolution of Nuclear Forensics: A Technical Viewpoint' in *Advances in Nuclear Forensics: Countering the Evolving Threat of Nuclear and Other Radioactive Material out of Regulatory Control Summary of an International Conference* (Vienna: International Atomic Energy Agency, 2015).

6) Michael Kristo, 'Chapter 13 – Nuclear Forensics' in *Handbook of Radioactivity Analysis, Volume 2, Fourth Edition*, edited by Michael L'Annunziata (Elsevier, 2020).

7) International Atomic Energy Agency, *Nuclear Forensics in Support of Investigations*, IAEA Nuclear Security Series No. 2-G (Rev. 1) Implementing Guide, (Vienna: International Atomic Energy Agency, 2015).

8) Klaus Mayer, Maria Wallenius, and Zsolt Varga, 'Sample Characteristics and Nuclear Forensic Signatures' in *The New Nuclear Forensics*, edited by Vitaly Fedchenko (Oxford: Oxford University Press, 2015).

9) Benjamin Garrett, Klaus Mayer, Paul Thompson, Boon Kin Pong, Gauthier Lasou 'The Nuclear Forensics International Technical Working Group (ITWG) An Overview' in *Advances in Nuclear Forensics: Countering the Evolving Threat of Nuclear and Other Radioactive Material out of Regulatory Control Summary of an International Conference* (Vienna: International Atomic Energy Agency, 2015).

10) David Smith, 'The ITWG: 20 Years of Science Supporting Law Enforcement and Nuclear Security Investigations' in *ITWG Update No. 1,* edited by Vitaly Fedchenko (International Technical Working Group, 2016).

11) Vitaly Fedchenko, 'The Process of Nuclear Forensic Analysis' in *The New Nuclear Forensics*, edited by Vitaly Fedchenko (Oxford: Oxford University Press, 2015).

12) International Atomic Energy Agency, *Radiological Crime Scene Management*, IAEA Nuclear Security Series No. 22-G Implementing Guide, (Vienna: International Atomic Energy Agency, 2014).

13) International Atomic Energy Agency, *Development of a National Nuclear Forensics Library: A System for the Identification of Nuclear or Other Radioactive Material out of Regulatory Control*, IAEA Non-Serial Publication IAEA-TDL-009, (Vienna: International Atomic Energy Agency, 2018).

14) International Atomic Energy Agency, Incident and Trafficking Database (ITDB), accessed 30 June 2021, https://www.iaea.org/resources/databases/itdb.

15) Michael J. Kristo, Amy M. Gaffney, Naomi Marks, Kim Knight, William S. Cassata and Ian D. Hutcheon, 'Nuclear Forensic Science: Analysis of Nuclear Material Out of Regulatory Control' in *Annual Reviews of Earth and Planetary Sciences*, v. 44 (2016), 555.

16) Elizabeth Keegan, Michael J. Kristo, Kaitlyn Toole, Ruth Kips and Emma Young, *Nuclear Forensics: Scientific Analysis Supporting Law Enforcement and Nuclear Security Investigations,* Analytical Chemistry, v. 88, n. 3 (2016), 1496.

17) Lyudmila Zaitseva and Friedrich Steinhäusler, *Nuclear Trafficking Issues in the Black Sea Region*, EU Non-Proliferation Consortium, Non-Proliferation Papers, n. 39 (2014).

18) Kenton J. Moody, Patrick M. Grant, Ian D. Hutcheon, *Nuclear Forensic Analysis, 2nd Edition* (Boca Raton, London, New York: CRC Press - Taylor and Francis Group, 2015), p. 417.

19) Sidney Niemeyer and Ian D. Hutcheon, 'Forensic Analysis of a Smuggled HEU Sample Interdicted in Bulgaria' in *International Conference on Advances in Destructive and Non-Destructive Analysis for Environmental Monitoring and Nuclear Forensics* (Vienna: International Atomic Energy Agency, 2002).

20) S. Baude, B. Chartier, D. Kimmel, F. Mariotte, D. Masse, H. Peron, D. Tilly 'The French Response in Cases of Illicit Nuclear Trafficking Lessons Learned from a Real Case', in *Illicit Nuclear Trafficking: Collective Experience and the Way Forward* (Vienna: International Atomic Energy Agency, 2008), 363.

21) S. Baude, ‘HEU Seized in July 2001 in Paris Analytical Investigations Performed on the Material’, in *Illicit Nuclear Trafficking: Collective Experience and the Way Forward* (Vienna: International Atomic Energy Agency, 2008), 397.

22) Zsolt Varga, Maria Wallenius, Adrian Nicholl, Klaus Mayer, Ionel Balan and Vasile Benea, ‘Measurement of Production Date (Age) of Nanogram Amount of Uranium’ *Journal of Radioanalytical and Nuclear Chemistry,* v. 322 (2019): 1585

23) International Atomic Energy Agency, *IAEA Safeguards Glossary, 2001 Edition*, International Nuclear Verification Series No. 3, (Vienna: International Atomic Energy Agency, 2002).

24) International Atomic Energy Agency, *Arrangements for the Termination of a Nuclear or Radiological Emergency*, IAEA Safety Standards, General Safety Guide No. GSC-11, (Vienna: International Atomic Energy Agency, 2018).

25) International Atomic Energy Agency, *Categorization of Radioactive Sources*, IAEA Safety Guide No. RS-G-1.9, (Vienna: International Atomic Energy Agency, 2005).

26) Tom Bielefeld, ‘Mexico’s Stolen Radioactive Source: It Could Happen Here’, *Bulletin of the Atomic Scientists,* 2014, 01, (2014).

27) International Atomic Energy Agency, *Security of Radioactive Material in Transport*, IAEA Nuclear Security Series No. 9-G (Rev.1) Implementing Guide (Vienna: International Atomic Energy Agency, 2020).

28) International Atomic Energy Agency, *Sealed Radioactive Sources,* IAEA/PI/A.98/13-32051 (Vienna: International Atomic Energy Agency, 2013).

29) International Atomic Energy Agency, *Control of Orphan Sources and Other Radioactive Material in the Metal Recycling and Production Industries,* IAEA Safety Standards Specific Safety Guide No. SSG-17 (Vienna: International Atomic Energy Agency, 2012).

30) Klaus Mayer, Maria Wallenius, Zsolt Varga, Magnus Hedberg and Nicole Erdmann ‘Inorganic Mass Spectrometry as a Tool of Destructive Nuclear Forensic Analysis’ in *The New Nuclear Forensics*, edited by Vitaly Fedchenko (Oxford: Oxford University Press, 2015).

31) Mark Elert, *Import of Metal Scrap – Risks Associated with Radioactivity*, KEMAKTA AR 92-22 (Stockholm, Sweden: Kemakta Konsult AB, 1992).

32) Michael Krachler, Maria Wallenius, Adrian Nicholl, and Klaus Mayer, *Spatially-Resolved Uranium Isotopic Analysis of Contaminated Scrap Metals Using Laser Ablation Multi-Collector ICP-MS*, Royal Society of Chemistry Advances, 10, (2020) 16629.

33) Andrei I. Apostol, ‘Nuclear Forensics: 10 Years A Full Time Job’ in *ITWG Update*, *No. 19*, edited by Vitaly Fedchenko (International Technical Working Group, 2021).