

# **Technical Meeting on Nuclear Forensics: From National Foundations to Global Impact**

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## **Book of Abstracts**



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## Poster Session 1 / 1

## Iterative method combined with HRGS for physical characterization of uranium materials in the frame of nuclear forensics investigations

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High-resolution gamma spectrometry (HRGS) is a non-destructive analysis technique, that plays a dual role at the first stages of nuclear forensic examination: fast in-field categorization of radiological or nuclear material in order to identify the safety risk to first responders and to the public and more comprehensive characterization of the material in the laboratory to accurately determine the isotopic composition.

Let us imagine that the nuclear forensic scientist needs to investigate seized nuclear material, categorized as natural uranium (NU), in a sealed container. With equal probability, the container may store a piece of metal uranium or powder of uranium ore concentrate (UOC) or uranium hexafluoride (UF<sub>6</sub>) or other uranium compounds. To perform a high-precision destructive analysis, a scientist must open the container, but the safety of the work will be greatly facilitated if the scientist could accurately estimate the risks posed by the material contained within. In this case, material's physical characterization in order to establish its shape can help.

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\begin{figure}[htbp]
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\includegraphics[width=3.2in]{figure_1.eps}
\caption{Material physical categorization based on value of matrix density and uranium mass fraction\label{F1}}
\end{figure}
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The characteristics of uranium materials of the nuclear fuel cycle vary over a wide range of values. The uranium mass fraction (uranium assay),  $\omega$ , and the matrix density,  $\rho$ , are the physical characteristics of the material, the knowledge of which makes it possible to assess its physical form and, as a consequence, make a decision on the necessary safety measures in the process of its further characterization. Once the quantities,  $\omega$  and  $\rho$ , of an unidentified material are determined, its shape can be deduced (see the diagram in Fig.1).

The proposed approach rests on processing HRGS experimental results with the iterative quasi-Newton Broyden method for finding roots of two equations in variables  $\rho$  and  $\omega$ .

For investigation we used set of uranium materials in various shapes (powders, granules, metals and alloys), see Fig. 2 with enrichment from 0.4 to 19.7%. In addition, unsealed samples of pure uranium metal (see Fig. 3) and fuel elements in the form of microspheres based on UO<sub>2</sub> (see Fig. 4 and 5) were studied.

Using HRGS characterization supplemented with our iterative algorithm, the powders of U<sub>3</sub>O<sub>8</sub> with uranium mass fraction of about 84% can be distinguished from the powders of UO<sub>2</sub> with uranium mass fraction of about 87%, as well as uranium products in form of liquids or loose powders with matrix density of 0.5–2.0 g/cm<sup>3</sup> can be distinguished from uranium products in form of compacted fuel elements with matrix density of 6.0–10.0 g/cm<sup>3</sup> or from pure metal uranium and uranium alloys with matrix density of 14.0–19.0 g/cm<sup>3</sup>.

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\begin{figure}[htbp]
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\caption{A set of uranium materials in various shapes.\label{F2}}
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\caption{Unsealed sample of pure uranium metal.\label{F3}}
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\caption{Image of coated by SiC fuel microsphere slice obtained by Leica M165 optical microscope\label{F4}}
\end{figure}
\begin{figure}[htbp]
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\caption{Fuel microspheres based on UO2 (general view).\label{F5}}
\end{figure}

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The values of  $\omega$  and  $\rho$  obtained by HRGS are confirmed by independent analytical methods. Uranium mass fraction in the fuel microspheres based on  $UO_2$ , estimated as 88.02% by HRGS is consistent with the results of isotope dilution mass spectrometry  $87.76 \pm 0.64\%$ . Densities of two different uranium metal samples, estimated as  $18.42 \text{ g/cm}^3$  and  $19.33 \text{ g/cm}^3$  by HRGS is consistent with the results of gas pycnometry technique  $18.24 \pm 0.55 \text{ g/cm}^3$  and  $18.86 \pm 0.59 \text{ g/cm}^3$ , respectively.

### Oral Session #1 – Legal Framework and the CPPNM/A / 3

## REGULATORY AUTHORITY ACTIVITIES IN SUPPORT TO NUCLEAR FORENSICS

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#### 1. INTRODUCTION

The nuclear forensics is the process of comparing sample characteristics with existing information about the types of material the origin and methods of production of nuclear and other radioactive material or previous cases associated with similar material. Thus, nuclear forensics regarded as a new area of integrated science research that allows not only to identify radioactive material (withdrawn from illicit trafficking or resulting from a nuclear accident), but also trace the entire chain of related events - from source origin until to detection. Nuclear forensics is the examination of nuclear and other radioactive materials using analytical techniques to determine the origin and history of this material in the context of law enforcement investigations or the assessment of nuclear security vulnerabilities. In this activity, consolidation and assistance of all organizations that can be involved in nuclear forensics are necessary.

#### 2. PRACTICAL ACTIVITIES

On April 24, 2008, the President of the Republic of Azerbaijan signed a decree on creation of an independent regulatory authority, State Agency on Nuclear and Radiological Activity Regulations (SANRAR) under the Ministry of Emergency Situations, which gives the regulatory function in considerable extent to the sole body. With establishment of Regulatory Authority all the powers and duties related to the provision of supervision and control over nuclear and radiological activities, accounting and control of nuclear and other radioactive materials, as well as authority for authorization in this area were transferred to SANRAR. Since the creation of new Regulatory Authority, the analysis of all obligations arising from the international agreements and treaties, as well as the improvement of the implementation of assigned functions, have been one of the main priorities of SANRAR activities. To date, SANRAR plays an important role in fulfilling of obligations stipulated by the relevant international agreements, taking the necessary measures



to improve of existed infrastructure aimed to strengthening of the legislative and technical bases, as well as to improving of the used communication and information technologies. The practice of the regulatory activities in the Republic of Azerbaijan are includes the authorization, inventory, accounting, performance of the relevant obligations under the safeguards agreement, inspections, export-import control, expert assessment, enforcement, etc. The presentation will describes the regulatory aspects in the field of regulation of nuclear and radiological activities in relation to the provision of necessary information and technical support for nuclear forensics, as well as the created associated infrastructure. Technological capabilities, disadvantages and ways of improvement will also be described in presentation.

### 3. FINDINGS OR RESULTS

Decision-making capabilities, established organizational structure and financing mechanisms allow the fulfillment of the main functions of the regulatory authority at present. At the same time, there are challenges related to the legislative base and staffing of regulatory authority with the highly qualified staff in connection with the planning of expansion of activities in the field of peaceful nuclear technologies and for improving their capabilities for successful implementation an appropriate role in support to nuclear forensics activities.

### 4. CONCLUSION

The decision on development of nuclear technologies for peaceful purposes and planning of the construction of research reactor in the future requires improving the structure and capabilities of the Regulatory Authority, as well as improving the legislative framework for the possibility of comprehensive execution of regulatory functions. The presentation will summarize achieved progress and provide proposals to improvement of existing infrastructure in considerable to extent to the necessary requirements.

## Poster Session 2 / 4

### **Investigation of the lanthanites pattern for uranium attribution in nuclear forensics environment**

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Illicit trafficking of nuclear material has been a subject of concern since the first seizures of nuclear material were reported to the International Atomic Energy Agency in the beginning of the 1990s. In South Africa cases of illegal trafficking of nuclear materials has been reported to the South African Police Services (SAPS). The aim of this study was to determine whether the lanthanides patterns measured in a particular sample can be used to attribute the uranium sample to the production or reprocessing plant. In this work, twenty samples selected for investigation originate from South Africa and Namibia uranium mines. Measurements were carried out using an inductively coupled plasma mass spectrometer (ICP-MS) PerkinElmer NexION 2000. Both measured results from different mines show significant variation within mine and thus provide valuable information about the geochemical formation and origin. This finding can be used to link the seized sample to particular reprocessing plant making possible for the authorities to narrow their search to the origin of the sample.

## Oral Session #2 – Capability Development and Sustainability / 7

### **Current Status and Future Prospects on Nuclear Forensics Capability Building and Technology Development by the Integrated**

## Support Center for Nuclear Non-proliferation and Nuclear Security at the Japan Atomic Energy Agency

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Any nuclear security event involving nuclear and other radioactive materials outside of regulatory control (MORC) has the potential to have severe consequences for public health, the environment, the economy and society. Each state has a responsibility to develop national nuclear security measures to respond to such an event, and this includes a nuclear forensics capability. In Japan, national nuclear forensics capability building efforts mainly based on research and development (R&D) have been conducted since 2010, in accordance with national statement of Japan at the 1st Nuclear Security Summit. Most of that work is undertaken at the Integrated Support Center for Nuclear Non-proliferation and Nuclear Security (ISCN) of the JAEA in close cooperation with other competent authorities. The ISCN has made increased contributions to the enhancement of international nuclear security by establishing technical capabilities in nuclear forensics and sharing the achievements with the international community.

The ISCN has mainly engaged in R&Ds for establishing and strengthening nuclear forensics technical capability. As for the laboratory capability, several new pieces of analytical equipment have been introduced for nuclear forensics R&D purposes. High-precise analytical techniques that has been proven in the past nuclear forensics cases have been established, and several new techniques that can contribute to the analysis of nuclear forensics signatures have been developed in the laboratory. The ISCN has been also developed a proto-type nuclear forensics library based on the nuclear material data and experience of past research of nuclear fuel cycle in JAEA. These technical capability developments have been conducted based on the international cooperation with a variety of partners such as the U.S. Department of Energy and EC Joint Research Center, and participation in exercises organized by ITWG.

Recent R&Ds activities by the ISCN have been mainly based on the needs of domestic competent authorities, such as first responders and investigators, and aim to develop technologies for the implementation of nuclear forensics covering the entire spectrum of forensics processes from crime scene investigation to laboratory analysis and interpretation. One important key issue is the enhancement of technical capability for post-dispersion nuclear forensics. For instance, the ISCN has carried out the development of mobile radiation measurement equipment coupled with the low-cost, small radiation detectors that use machine-learning algorithms, which enables quick and autonomous radioisotope identification to support first responders in crime scene or post-dispersion crime scene. Signature analysis of samples collected at a post-dispersion crime scene are also among the important technical issues studied at the ISCN. The application of emerging technologies to nuclear forensics has also been studied. This includes the application of deep learning models analyzing a new signature to nuclear forensics interpretation to enable more confident and objective sample comparison analysis, and the development of instant contamination imaging technology contributing appropriate examination plan development on the seized samples in collaboration with conventional forensics.

Many analytical techniques have been developed and the capability to analyze nuclear and other radioactive materials for nuclear forensics purposes has been matured considerably over the past decade. The challenges of post-dispersion samples, collaboration with conventional forensics and the development of new signatures will be more important in the near future. Therefore, the ISCN will promote the R&Ds to further enhance the technical capabilities solving these issues. In addition, the ISCN is also promoting to expand the nuclear forensics research into universities and other research institutes in Japan. This is expected to contribute to the establishment of a domestic nuclear forensics network that enables to respond quickly and flexibly to the MORC incidents, and to the maturation of nuclear forensics as a new academic field.

**Oral Session #5 – Analytical Methods for Analysing Radiological and Nuclear Evidence / 8**

## **Analysis of UOC for nuclear forensics fingerprinting using Scanning Electron Microscope**

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Nuclear forensics involves the analysis of nuclear material for possible provenance determination using various analytical tools that are available for such analysis. In this study, Scanning Electron Microscopy (SEM) combined with Electron Dispersion Spectrometer (EDS), were used to determine the signatures of uranium ore concentrates (UOC) samples for nuclear forensic applications. SEM and SEM / EDS provided substantial information on this UOC's morphology and elemental composition. Distinct qualitative and quantitative difference are present for the different UOC's. The UOC's surface consists of agglomeration made up of homogenous spherical particles, irregular shaped particles and plate like bulky particles. Average particle size ranged between 0.1 – 0.2 µm. EDS analysis of all the samples showed they contained a consistent 70 weight % of uranium and a stoichiometric formula closest to the molecule of UO<sub>4</sub>. This technique can thus be used to distinguishing and fingerprinting UOC's originating from different mines in South Africa.

**Poster Session 3 / 9**

## **Nuclear Forensic Analysis Laboratory Capability in the Integrated Support Center for Nuclear Non-proliferation and Nuclear Security at the Japan Atomic Energy Agency**

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Nuclear and other radioactive materials outside of regulatory control (MORC) can trigger nuclear security events with severe environmental and economic consequences. In order to deal with such threats, it is necessary to strengthen international nuclear security measures, including nuclear forensics. Based on the national statement at the first Nuclear Security Summit, Japan has been conducting research and development on nuclear forensics and improving its capabilities since 2011. The development of nuclear forensics technology in Japan is led by the Integrated Support Center for Nuclear Non-proliferation and Nuclear Security (ISCN) of the Japan Atomic Energy agency (JAEA) and is being carried out under domestic and international cooperation. By actively disseminating the results of its technology development to the domestic and international communities, ISCN is contributing to the improvement of nuclear forensics capabilities not only in Japan but also internationally. The present paper has discussed the status of the nuclear forensic laboratory capability in the ISCN.

ISCN has been mainly focusing on the development of nuclear forensic analysis technology targeting uranium samples. ISCN possesses a variety of analytical instruments to analyze the physical and chemical characteristics (i.e., nuclear forensics signature) of uranium samples. For example, electron microscopes and mass spectrometers such as SEM and TIMS have been introduced in the laboratory,

and high-precision analytical capabilities have been developed using them. In morphology analysis, the consistency of samples can be analyzed by evaluating the particle size and shape distributions based on the SEM images. ISCN has also developed the procedure for uranium isotope ratio analysis and age dating by using TIMS that can contribute to the determination of the origins and history of the uranium samples. These laboratory capability of ISCN have been validated by the joint analysis project under bilateral and multi-lateral cooperation, and by participation in collaborative material exercises (CMX) organized by the ITWG. The ISCN has recently introduced several instruments to enhance the laboratory capability, such as inductively coupled plasma mass spectrometer (ICP-MS) and X-ray fluorescence spectrometer (XRF) for trace element analysis. By using both of the ICP-MS and XRF, it becomes possible to improve flexibility of analysis that meet the needs from criminal investigators. For example, the ICP-MS is capable of highly accurate analysis, but it requires complex and time-consuming sample pretreatment. On the other hand, the XRF is a non-destructive assay method has the great advantage in measurement time, but less accurate compared to the ICP-MS. It is important to use both instruments properly based on the investigators' needs and the combination of the instruments would contribute to optimize analytical process for nuclear forensics purposes.

The ISCN has recently re-evaluated the laboratory capability with the new instruments using certified reference materials. This paper also discusses the results of that reassessment. As a future activities ISCN is going to participate next CMX. This would be helpful to evaluate our capability with new laboratory configuration. It is also planned to initiate the development of analytical techniques for new signatures with international partners.

#### Oral Session #8 – Laboratory Capabilities / 10

### Research, Education and Training activities at Nuclear Physics Lab, AUTH

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The Nuclear Physics Lab, at School of Physics, at Aristotle University of Thessaloniki (NPL-AUTH), has more than 40 years of research activity in the field of Radiation Physics and Nuclear Physics Applications.

The members of the NPL-AUTH are radiation Protection experts and nuclear security event responders ready to response to incidents involving nuclear and other radioactive materials out of regulatory control. Environmental radioactivity, Chernobyl and Fukushima accidents, radioactive aerosols, waste characterization, orphan sources in scrap metal, retrospective doses, sterilization dosimetry in food and drugs are some of the group research activities.

The members of the NPL-AUTH are specialist in gamma-spectrometry and there are three Ge detectors in the Lab calibrated for various geometries and matrixes. Furthermore, infrastructure for radioactive aerosol measurements and radioactive aerosol size characterization is available at the lab, as well as two (2) Harshaw Thermoluminescence (TL) readers, while an XRF, an XRD and a scanning electron microscope with Energy Dispersive X-Ray Analyzer (SEM/EDS) is available at the School of Physics, AUTH for a common use.

The NPL-AUTH belongs to the ALMERA Network and participates every year in the IAEA Proficiency Inter-calibration tests. The NPL-AUTH participates in various IAEA CRP projects and in one RER project.

Regarding the educational activities, the members of Nuclear Physics Lab, participate in many compulsory and elective courses at School of Physics, dealing with basic Nuclear Physics and Nuclear Physics Applications in undergraduate and postgraduate programs. In addition, through our teaching duties we evolved to an efficient communicator able to distill technical information into easy-to-understand advice and explanations. We make full use of e-learning tools for an effective distance-learning process and we have great experience in preparing training material and lessons for academic teaching, professionals and workshops, all completed with resounding success.

Alongside teaching undergraduate and graduate level students and mentoring PhD candidates, we maintain an intense focus on research by leading and participating in many research projects. We attracted international recognition to our work and forged lasting relationships with the international scientific community. We have published more than 500 publications in peer-reviewed International Journals and International Scientific Conference in the field of Environmental Radioactivity, Nuclear

Physics Applications and in Dosimetry. There are more than 8000 citations in our papers. Regarding scientific Conferences, we hosted during 21-25 September 2015 the Environmental Radioactivity International Conference ENVIRA2015 ([www.envira2015.gr](http://www.envira2015.gr)), and during 6-10 December 2021 the online ENVIRA 2021 (<https://envira2021.gr/>), where in both conference the Conference Chair is a NPL-AUTH member. Furthermore, we will co-host the RAP2022 International Conference that will take place between 6-10 June 2022 in Thessaloniki, Greece and we will host a RCM meeting in the frame of J0214 CRP Project, during 15-18 February 2022. Participating in forensic activities will enable the NPL-AUTH and Greece to develop and sustain national capabilities in nuclear forensics. The NPL-AUTH Lab has the infrastructure, the experience and knowledge to support any research, educational and training activity in this field.

## Poster Session 1 / 11

### Characterization of uranium bearing material using HPGe gamma detector for nuclear forensics purposes

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Nuclear forensics is a method that uses nuclear material to uncover information. The nature of the method of manufacturing is expressed in the material's elemental and isotopic structure as well as its microscopic and macroscopic appearance. Notwithstanding the presence of national nuclear security frameworks, there keeps on being instances of material out of regulatory control, regardless of whether accidentally, for example, through misfortune, or purposefully because of a criminal intention, for example, robbery and theft. Every nation which is a Member State of the IAEA must build up the capacity to forestall, distinguish, and react to any event that includes trafficking of radioactive material that has nuclear security concerns. Thus, nuclear forensics, as a Tool for Nuclear Security has proven relevant in the identification of the products, how, when, and where the materials were produced and how they were designed to be used legally. Non-destructive analytical techniques are used to characterize and identify specimens as part of the verification of nuclear materials and the detection of undeclared nuclear materials in national and international safeguards. At the first two stages, gamma spectrometry serves a dual purpose: quick in-field categorization of radiological or nuclear material to identify the safety risk to first responders and the general public, and more thorough characterization of the material in the laboratory to accurately determine the isotopic composition. One of the challenges in this field is to clarify distinctions between origins, detect clusters and patterns, and eventually attribute samples to or differentiate from known sources using isotope ratios in uranium-bearing material samples with appropriate accuracy and measurement errors. The objective of this study was to investigate the use of the radiometric spectrometry technique to accurately characterize the uranium-bearing materials, using direct detection of high purity gamma spectrometry (HPGe) for gamma-rays from <sup>235</sup>U and <sup>238</sup>U. Isotopic composition and total U-content of UOC and uranium ore were determined. The results show that U & UOC isotopic ratios and activity of these nuclear materials from two uranium mines differed significantly with no correlation at all.

## Oral Session #7 – Signature Research on Isotopic Signatures and Age-Dating / 12

### The oxygen isotopes composition of uranium oxides in the nuclear fuel cycle as a new signature for process attribution

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Dear All,

Our work aims to expand the traditional nuclear forensics characterization techniques by measuring the oxygen isotopic composition of U<sub>3</sub>O<sub>8</sub> to explore new isotope signatures for attribution. We investigated the effect of different syntheses conditions such as calcination time, temperature, and cooling rates, on the final oxygen isotopic composition of U<sub>3</sub>O<sub>8</sub>, produced from Uranium metal, Uranyl Nitrate Hydrate, and Uranium Trioxide as starting materials.

It will be our honor to present this work at this technical meeting on Nuclear Forensics.

Regards,  
Maor Assulin

### Oral Session #3 – Radiological Crime Scene Management / 13

## Radiation Protection Concerns for Radiological Crime Scene Management

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A crime scene is any physical scene, anywhere, that may provide potential evidence to an investigator. It may include a person's body, any building, vehicles, places in the open air, or objects found at those locations. "Crime scene examination" refers to an examination where forensic or scientific techniques are used to preserve and gather physical evidence of a crime.

The crime scene with radioactive or nuclear material can be understood as the crime scene where, in addition to the necessary measures for the preservation of evidence, it is necessary to use all radiation protection techniques to ensure the personal safety of experts and others involved in the evidence collection and preservation.

Several publications deal with the most different types of radiological protection:

- Occupational Radiation Protection General Safety Guide IAEA Safety Standards Series No. GSG-7, 2018;
- Occupational Radiation Protection: Protecting Workers Against Exposure to Ionizing Radiation Proceedings of an International Conference Held in Geneva, Switzerland, 26–30 August 2002;
- Occupational Radiation Protection in the Uranium Mining and Processing Industry, Safety Reports Series No. 100, 2020;
- Radiation Protection and Safety in Veterinary Medicine, Safety Reports Series No. 104, 2021;
- Protection against Exposure Due to Radon Indoors and Gamma Radiation from Construction Materials – Methods of Prevention and Mitigation, IAEA TECDOC No. 1951, 2021;
- Radiation Protection and Safety in Medical Uses of Ionizing Radiation, Specific Safety Guide, IAEA Safety Standards Series No. SSG-46, 2018;
- Radiation Protection of the Public and the Environment, General Safety Guide, IAEA Safety Standards Series No. GSG-8, 2018;
- Radiation Protection of Itinerant Workers, Safety Reports Series No. 84, 2015;

Each IAEA member state defines, based on its criteria, the person who can access a crime scene containing radiological and nuclear material. Whether from the police, military, secret service agents, firefighters, special groups of law enforcement agents, experts from regulatory authorities, scientists and professors from universities and research centers, and even prosecutors and other technicians and specialists at their own discretion.

Those who work at crime scenes with radiological or nuclear material (despite not always being occupationally exposed workers) may thus be considered temporarily during action in the crime area

with radiological or nuclear material.

There is no specific IAEA guide on how to protect these agents in RSCM activities. The Occupational Radiation Protection General Safety Guide IAEA Safety Standards Series No. GSG-7, 2018 defines a group as the “emergency workers.” This group of emergency workers can be further divided into three categories (I, II, and III – regarding different kinds of actions), but this is for emergency workers.

Eventually, the document Radiation Protection of Itinerant Workers Safety Reports Series No. 84 of 2015 could be used as a starting point for regulation or guidance on how to effect radiological protection for those involved in the management and handling of crime scenes with radiological and nuclear materials. The definition of Itinerant Workers is an occupationally exposed worker, who work in supervised and (or) controlled areas at one or more locations and are not employees of the management of the facility where they are working.

Along with the three fundamental principles of radiological protection (Justification, Optimization, and Limitation of individual doses), we could add the Prevention of accidents in workplaces, with risks considered and analyzed in the design of facilities and equipment and in work procedures involving the use of radiation sources or radioactive material to minimize the probability occurrence of accidents.

However, in this specific case, it is not possible to talk about a facility design or even a work plan, given the variety of situations where a crime scene can occur from smuggling material in postal, customs, or border facilities to theft in nuclear or radioactive facilities to even places subject to exposure from a dirty bomb or RDD.

To general recommendations such as

- Use adequate protection
- Avoid undue exposure to radiation
- Minimize exposures
- Avoid the unnecessary presence of people in the scene
- Correct handling of the Basic Principles of Time, Distance, and Shielding

More specific recommendations are added, such as

- Training and dissemination of concepts of radioprotection and work with radiation sources, mainly for professionals without training in the area, mainly military, police, law enforcement agents, health care personal
- Take all necessary preventive measures to avoid accidents that lead to its contamination as well as the contamination of evidence and evidence
- Maintenance of the evidence and evidence chain of custody
- Always use the individual monitor during working hours.
- Creation of dose databases of professionals involved in crime scene assistance.

IAEA Nuclear Security Series No. 22–G Implementing Guide ( under revision) is a 2014 document on the process used to ensure safe, secure, effective, and efficient operations at a crime scene where nuclear or other radioactive materials are known or suspected to be present. This publication focuses on the framework and functional elements for managing a radiological crime scene distinct from any other crime scene. The main suggestion of this work is the creation of a specific guide of radiation protection for this RSCM.

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## **IMPLEMENTATION OF THE SELF-ASSESSMENT TOOLS FOR BUILD UP A NATIONAL NUCLEAR FORENSICS CAPABILITY**

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1. INTRODUCTION Thailand was established nuclear forensics in 2013. The project began with the Thai government budget and European Union Chemical, Biological, Radiological and Nuclear

(CBRN) Risk Mitigation Centres of Excellence (CoE) supported the funding of the Network of Excellence for Nuclear Forensics in the South East Asia Region (Project 30) in 2013–14. The objective was to strengthen regional public security by upgrading nuclear forensics capabilities, technologies, and methodologies for assessing nuclear and other radioactive materials. In order to build up the national capabilities, the Office of Atoms for Peace (OAP) was designated to identify the strength and weaknesses of current facilities. The Nuclear Forensics Self-Assessment Tool (SAT) developed by the Global Initiative to Combat Nuclear Terrorism (GICNT) was utilized to identify existing capabilities. Thailand has created a national roadmap for the development of nuclear forensic science in the country, focused on increasing the capabilities and technical expertise of a national nuclear forensics laboratory.

2. UTILIZATIONS OF THE SELF-ASSESSMENT TOOLS The OAP translated SAT into the Thai language, including selected relevant stakeholders. Three sets of Google Forms were created to meet the requirement for assessment of the competent authorities. The stakeholders were the frontline officers, forensics organizations, regulatory bodies, academic lecturers, and analytical laboratories scientists. The SAT was distributed to the agency's contact point and directly to the organization members. The one hundred persons of the competent authorities acquired the tool, and seventy-two of them responded to the form due to the time constraint for allocating the SAT. Subsequently, OAP identified the strength and deficiencies according to the feedback SAT. The networks development, SOP development, ISO/IEC 17025 accreditation needed to be prioritized to overcome the gaps. These issues were included in sustainable nuclear forensics activities to be the five-year plan of the national nuclear security.
3. IMPLEMENTATION OF THE FEEDBACK FROM THE SELF-ASSESSMENT TOOLS The network communication and SOP were developed by discussing with the competent authority, also plan to include in the Nuclear Security Regime by 2022. In addition, OAP has developed the Nuclear Forensics Laboratory to acquire the ISO/IEC 17025. The first scope for supporting the Nuclear Energy for Peace Act was the qualification of uranium and thorium in a geological sample using gamma spectrometry, certified in June 2021.
4. CONCLUSIONS The SAT was the main subject for assisting the country to identify its existing capabilities and disclose deficiencies. The tool incorporates the whole process, which is not only forensic analysis but also crime scene management. However, some specific areas would be rather complicated for some competent authorities to respond to the questions. Because Thailand does not have inclusive nuclear activities, the SAT was necessary to adapt to meet the requirement of the stakeholders to understand. The OAP had selected only the related nuclear activity and provided more elaborate details so that almost competent authorities would identify strengths and deficiencies effectively. A few topics were specific to reveal the actual circumstances, especially the competent authorities are not in a position of the practitioner-level. Some questions need to respond from the policy level since they can establish a long-term plan for implementing nuclear forensics efficiency. Consequently, the communication between the pilot organization and the stakeholders is a significant issue to fruitful the SAT as an element for national planning to achieve the goals for capacity building of nuclear forensics.

### Poster Session 3 / 15

## CONDUCTING VIRTUAL EXERCISES FOR NUCLEAR FORENSICS CAPACITY BUILDING DURING THE PANDEMIC

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1. **INTRODUCTION** Thailand arranged the ASEAN Regional Forum on Nuclear Forensics in December 2011, Then, the Office of Atoms for Peace (OAP) planned to establish a Nuclear Forensics Center. Since 2013, nuclear forensics in Thailand was established under the Seoul Nuclear Security Summit. The Prime Minister has declared to support global nuclear security as well as nuclear terrorism mitigation. OAP has been designated to build up nuclear forensics capabilities, which have three main functions to enhance the performance; network strengthening, signature analysis, and database development. Those are necessary to prioritize. The national training program is a key element to support the network development in communication among the competent authorities. Due to the pandemic, Thailand had to adapt the training course to the virtual engagement to keep the momentum of strengthening the networks during 2020-2021. The Standard Operating Procedure (SOP) evaluation and the software assessment for nuclear and radioactive materials were discussed through the virtual exercises to fulfill the requirement in practical use, which was complied with law and regulations.
2. **CONDUCTING THE VIRTUAL EXERCISES DURING PANDAMIC** The SOP was developed by discussing with the competent authorities and finished in 2017. The role and responsibility of the nuclear forensics networks were defined regarding the law and regulations from each procedure. In order to identify the strength and weakness of the SOP, the virtual exercises were conducted through interactive scenarios in 2020. The competent authorities assisted the OAP in completing the SOP. In addition, Thailand has created the software for assessment nuclear forensics signatures at a crime scene in phase I. The virtual exercises were also established to estimate the meet all requirements for practical use of the frontline officers. The software has arrayed in three authentications access, composed of frontline officers, regulatory agencies, and the nuclear forensics authorities.
3. **THE VIRTUAL EXERCISE OUTCOMES** The feedback from the competent authorities is valuable in terms of the development of the SOP and software for assessment, sixty participants from the frontline officers were responding to the evaluation forms. The SOP was adopted accompanied by national law and regulations. The finished SOP will be planned to include in the nuclear security regime in 2022. According to the software for assessment at the crime scene, more than two hundred participants attended the workshop and provided helpful comments. The software was planned to upgrade in phase II and will be completed in 2022.
4. **CONCLUSION** The virtual exercise is a vital factor for communication under the pandemic circumstance with the advantages of no limitation with attendees and distance to maintain the networks strengthen. However, the actual workshop is still necessary to practice with the relevant organizations to raise their awareness for combatting nuclear threats. Furthermore, the networks will have an opportunity to use the detection equipment to detect the nuclear and radioactive materials in the actual situation, including familiarity with the software for assessing nuclear forensics signatures at a crime scene too.

**Poster Session 3 / 16**

## **Forensics Detection of Nuclear Materials by Machine Learning Microphotonic Techniques**

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Nuclear security is one of the greatest challenges facing the world today. Detecting criminal/unauthorized acts involving nuclear and other radioactive material out of regulatory control is a priority in nuclear security. In nuclear forensics detection of nuclear materials to determine whether the sample is nuclear, its isotopics and corresponding enrichment are the key aspects to collect information

about the samples and other (suspicious) trace evidences; identify sample type(s); and categorize them normally using traditional radiation detectors and spectrometers. However these techniques are neither the only ones suitable for the task nor do they lack limitations especially with regard to rapid analysis. For nuclear forensics to be an effective “deterrence” against nuclear security threats, techniques for direct rapid analysis of nuclear and radioactive materials (NRM) are required.

A synergy is achieved by combining machine learning and microphotonics based techniques namely laser based spectrometry and spectral imaging to elucidate more comprehensively the isotopic, molecular and elemental composition, as well as microstructure of nuclear materials (each step in the fuel cycle creates and/or modifies these nuclear forensic (NF) signatures).

We will in this talk describe our work (photonics-based spectral imaging and microanalytical method developments for nuclear forensics in support of nuclear security) where we exploit laser induced breakdown spectroscopy (LIBS) spectral/imaging and confocal laser Raman spectromicroscopy towards this goal. The work focuses on the methods that answer questions that may be asked when nuclear material is seized by Law Enforcement. The techniques have further utility in responding to anthropogenic environmental releases of nuclear and radioactive materials and illicit trafficking activities of such materials in our region, as well as analyzing activities related to radiological dispersal devices (RDD) and improvised nuclear devices (IND).

While LIBS reveals the atomic (and sometimes molecular and isotopic) signatures of micro-plasma obtained from the ablated samples, laser Raman microspectroscopy reveals the molecular configuration as well as structure and morphology of the materials. Machine learning is used in combination to extract subtle information from the complex multivariate spectra/images and to perform data dimensionality reduction as well as exploratory modeling. Key advantages of this approach are: small samples (µg) can be evaluated with minimal or no sample preparation; and samples can be remotely analyzed rapidly (µs-seconds – ideal for radioactive samples). Especially microanalytical capability for micro-size samples is a powerful tool for monitoring undeclared nuclear activity, verifying nuclear safeguards, responding to nuclear anthropogenic releases, and analyzing materials from radiological crime scenes.

We have used LIBS coupled with machine learning to determine trace Y, Rb, Zr, Sr and Te in nuclear glass. These elements often coexist with uranium in post irradiation nuclear wastes. The biggest attraction of the developed analytical approach is that the protocol may be integrated into a suitable software interface and installed in a handheld LIBS system to provide on-site, real-time, stand-off and direct rapid analysis of fission products (FP) in post irradiated materials of nuclear origin and detonation events since only tiny glass debris (1 mm) and tiny ( $\leq 2 \mu\text{l}$ ) can provide reliable nuclear forensic signatures. We also exploited machine learning enabled LIBS in air and at atmospheric pressure utilizing weak uranium lines to predicted uranium concentration in uranium-bearing mineral ores between 103 - 837 ppm. The developed method in conjunction with principal component analysis (PCA) successfully assigned the samples to their origin. Size - resolved ( $\varnothing$ : 2.5  $\mu\text{m}$ , 4.5  $\mu\text{m}$ ) analysis of individual aerosols from a mimic uranium mine atmosphere has been undertaken in the context of in-field nuclear forensics, using laser Raman spectromicroscopy (LRS), method that is suitable for forensic analysis of uranium-bearing nuclear materials and hot aerosols in the atmosphere. The concentration of uranium ranged 50-200 ppb, being more enriched in the larger compared to the smaller size fraction.

#### Oral Session #1 – Legal Framework and the CPPNM/A / 17

### **The Australian Nuclear Science and Technology Organization (ANSTO) and the Philippine Nuclear Research Institute (PNRI) Collaboration on Nuclear Forensics and Border Protection**

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The Australian Nuclear Science and Technology Organization (ANSTO) and the Philippine Nuclear Research Institute (PNRI) signed a Memorandum of Understanding (MOU) last January 2021. The partnership of ANSTO and PNRI covers a wide range of applications, such as Nuclear Forensics, Emergency Preparedness, Border Security Protection, Environmental Protection, Radiation Safety, and Advanced Materials and Technologies using neutron scattering and synchrotron radiation-based measurements. Both agencies celebrated the signing of the MOU with the Department of Science and Technology's (DOST) Secretary Dr. Fortunato "Boy" T. de la Peña highlighting the signing of the MOU in the DOST Weekly Reports and Nuclear Australia, Quarterly Newsletter of the Australian Nuclear Association, featuring an article in the March 2021 issue.

One of the key areas of collaboration that has been highlighted in the MOU is in Nuclear Forensics, a field that is relatively new to the PNRI. But with the capability in nuclear technology of PNRI, and the partnership of ANSTO in terms of training and capacity-building, PNRI will be able to integrate into this field. Through the introduction of ANSTO, PNRI is now a member of the International Technical Working Group (ITWG) on Nuclear Forensics, led by the USA and Europe, and supported by the International Atomic Energy Agency (IAEA). In the 25th ITWG-Nuclear Forensics Conference last June 17, 2021, which was attended by participants from America to Asia, PNRI made a virtual presentation on the Nuclear Forensics Initiative in the Philippines. The ITWG-Nuclear Forensics community welcomed warmly the membership of the Philippines as Thailand and the Philippines are the only countries in the Southeast Asian region who are members of the international group.

As part of the activities of the ITWG-Nuclear Forensics, an international exercise on nuclear forensics capabilities called Collaborative Materials Exercise (CMX) is done wherein participating laboratories all over the world do a "mock" nuclear forensics exercise. For this year, the PNRI will join the CMX exercise in September 2021 and ANSTO, being senior members of the ITWG-Nuclear Forensics has extended their experience and are guiding the PNRI team who will be conducting the nuclear forensics investigation for the 1st time. Preparation of the workplan, processes and documentation were prepared by the PNRI CMX Team with consultation meetings with the ANSTO Team.

To fund the research initiatives and activities of PNRI in the area of Nuclear Forensics and Border Protection, the program "Nuclear Forensics for Border Protection and National Security" was included in its Long-Term Nuclear Research and Development Plan. Four (4) projects were included in the program, namely:

Project 1: Ammonium Nitrate (AN) and Explosives Characterization as a Link to the Source of Origin in PH and the SEA region

Project 2: Nuclear Forensics Capacity building for the Department of National Defense and the Philippine National Police

Project 3: Analysis on Radioactive Tracers on the West Philippine Sea

Project 4: X-ray Imaging and Nuclear Forensics for the detection of contraband at the Philippine Ports

All these projects are envisioned to be in close collaboration with ANSTO.

With ANSTO as the lead agency, and PNRI its partner, a Regional Cooperative Agreement (RCA) pre-concept proposal for the 2024-25 project cycle was submitted last August 2021 with the title, "Leveraging Nuclear Science and Technology to Strengthen Border and Nuclear Security". ANSTO (Australia) and PNRI (Philippines) were to jointly promote and coordinate this regional project to foster collaboration with interested RCA Government Parties to strengthen regional capability in areas including border security and nuclear forensics. Although the pre-concept proposal did not push through, other proposals are being crafted to strengthen and continue the ANSTO and PNRI collaboration.

### Poster Session 3 / 18

## DEVELOPMENT OF CAPABILITY ON NUCLEAR FORENSICS SIGNATURE INVESTIGATION TO ESTABLISH A NATIONAL NUCLEAR FORENSICS LIBRARY

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#### 1. INTRODUCTION

In 2013, with the support of the Royal Thai Government, the European Commission, and the IAEA, Thailand Nuclear Forensics Laboratory was established officially at the Office of Atoms for Peace. For almost ten years, Thailand has kept the momentum to enhance the national capability to fight against illicit trafficking on nuclear and other radioactive materials out of regulatory control in Thailand and Region. Thailand's Nuclear forensics laboratory staff were trained under the IAEA Human Resource Development Program. At the national level, the nuclear forensics network was strengthening in paralleled, raising awareness on the existing nuclear terrorism. The ability to reliably determine the origin and history of nuclear and other radioactive materials depends upon access to relevant sample materials, the identification of priority signatures, high confidence measurements or predictions, and the ability to perform inter-comparisons between data collected on an unknown sample and knowledge or database of known material to potentially including a national nuclear forensics library [1]. To raise the confidentiality in the results from the nuclear forensics investigation, the laboratory has built up the capability to measure the nuclear forensics signature for the establishment of the national nuclear forensics library.

#### 2. WORK CONDUCTED

As we know, nuclear forensics identification provides information on the origin and history of nuclear and other radioactive materials out of regulatory control concerning the nuclear security event, leading to the prosecution under legal proceedings. Therefore, the increasing confidence of nuclear forensics signatures identification is significant in nuclear forensics investigation. It is important to develop the signature analysis methodology in-depth to ensure the accuracy and reliability of results. Based on the existing instrument and facilities in Thailand nuclear forensics laboratory, four techniques, Gamma spectrometry, X-ray Diffraction(XRD), Scanning Electron Microscope equipped with Energy Dispersive X-ray spectroscopy (SEM-EDX), and Inductively coupled plasma mass spectrometry (ICP-MS) were performed to develop the capacity of measurement the nuclear forensics signature. Due to the limitation of existing samples in the country, Certified Reference Material (CRM), standard point sources, monazite ore, and rare-earth were used to build up capability on nuclear forensics signature investigation. The analysis follows the recommendation on the Implementing Guide entitled Nuclear Forensics in Support of Investigations [2]. Gamma spectrometry provides isotropic analysis information, then, Chemical forms were identified by XRD. After that SEM-EDX technique was performed to identify particles, morphologies, and element distribution and also track the U/Th particles. Finally, the rare-earth elements and trace elements were determined by ICP-MS.

In the beginning, the national nuclear forensic library was established based on existing of nuclear and other radioactive material under regulatory control in the national database and the results from the nuclear forensics laboratory, to confirm the existence of the nuclear and other radioactive material in the country. After that, the results from the nuclear forensics laboratory investigation of nuclear and other radioactive material out of regulatory control were included.

#### 3. CONCLUSIONS

To develop the national nuclear forensics library, Thailand's nuclear forensics laboratory develops capability on nuclear forensics signature investigation based on existing equipment and facilities. The nuclear forensics analytical results using the CRM contribute to increasing the investigation confidentiality. The first national nuclear forensics library was established which consisted of the national existing nuclear and other radioactive material under regulatory control and the results from the nuclear forensics laboratory investigation.

#### 4. REFERENCE

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- [2] International Atomic Energy Agency, Nuclear Forensics in Support of Investigations: Implementation Guide, NSS. NO.2-G, International Atomic Energy Agency, Vienna, 2015.

**Oral Session #8 – Laboratory Capabilities / 19**

## **Thailand's Challenges of Becoming an ISO/IEC 17025 Accredited Nuclear Forensics Laboratory: A Scope of Qualitative Analysis of Uranium and Thorium in a Geological Sample Using Gamma-Ray Spectroscopy**

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The Nuclear Forensics Laboratory in Thailand was established in July 2014 at the Office of Atoms for Peace (OAP) through the support and collaborations of domestic and international partners. Thailand has implemented nuclear forensics to prevent and respond to nuclear security events and illicit trafficking of nuclear and radioactive materials in the country and the ASEAN region. OAP has developed the country's nuclear forensic framework by parallel implementing the main four parts: radiological crime scene management, laboratory analysis, nuclear forensics library development, and domestic nuclear security network. According to the precise framework, OAP has accomplished laying a foundation of nuclear forensics in Thailand.

Nevertheless, one of the most challenges in nuclear forensics is the capability of laboratory analyses. Due to an unpredictable of a wide range of samples and forms. Therefore, OAP deliberated to use an International Organization for Standardization ISO/IEC 17025 standard (General requirement for the competence of testing and calibration laboratories) as a tool to support and provide more confidence in the laboratory analyses. In addition, the ISO standard should assist the laboratory management to get into a system process. It is known that gamma-ray spectroscopy is a useful technique for non-destructive analysis in a nuclear forensics laboratory. It is capable of an isotopic determination for both nuclear and radioactive materials and it can achieve an early investigation. Then, OAP decided to start ISO/IEC 17025 accreditation with a qualitative analysis by using gamma-ray spectroscopy. After undergoing many steps, since June 2021 Thailand's Nuclear Forensics Laboratory is an ISO/IEC 17025 accredited nuclear forensics laboratory for qualitative analysis of uranium and thorium in a geological sample using gamma-ray spectroscopy. This successful achievement will lead to a scope expansion in the ISO system with other instruments in the laboratory. Benefits and challenges during steps of preparation and after ISO/IEC 17025 accreditation will be provided in this paper.

**Oral Session #6 – Novel Techniques Applied to Nuclear Forensic Examinations / 20**

## **Development of a FIB-SEM-ToF-SIMS microanalysis method for potential applications in the investigation of nuclear forensics samples and safeguards**

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We describe the development of a comprehensive microanalysis method using focussed ion beam-scanning electron microscopy (FIB-SEM) in combination with time-of-flight-secondary ion mass spectroscopy (ToF-SIMS) for potential applications in the investigation of nuclear forensic samples and safeguards. Possible materials for examination include bulk uranium and powder specimens with varying isotopic enrichments. The integration of FIB-SEM and ToF-SIMS instrumentation into a single microanalytical platform is still an emerging capability. Its use for the precise and accurate isotopic characterisation of discrete particles or grains within nuclear materials is an area we are just starting to explore at ANSTO. Analysis of these isotopic signatures is useful in deconvoluting the history of how nuclear materials are formed and it can also provide further insight during the investigation of unknown samples.

SIMS has been extensively used as a technique for the surface elemental mapping and depth profiling of materials at the nano-scale, with detection sensitivity from the ppm to ppb range. Recent developments in SEM and related FIB

instrumentation have led to advanced capabilities for performing in-situ SIMS measurements using a dual-beam FIB-SEM system. Microanalysis by FIB-SEM with ToF-SIMS involves using the ion beam to remove material at the sample surface by mechanical sputtering and ionisation. Secondary ions (SI) from the sputtered material are extracted and then pulsed through a ToF-SIMS analyser where they are separated according to their mass/Q ratio and the measured time of flight to the detector (Figure 1a). In comparison to energy dispersive X-ray spectroscopy (EDS), which is the more commonly used microanalysis technique in the SEM, FIB- based ToF-SIMS has a lower interaction volume allowing for high surface sensitivity and greater spatial resolution for isotope mapping (Figure 1b).

At ANSTO, a Xe plasma TESCAN FERA FIB-SEM is equipped with an EDS and a ToF-SIMS detector. Experiments have been carried out on a range of inactive materials that include bulk, thin film and powder samples. A typical workflow begins with high-resolution SEM imaging, followed by EDS and finally ToF-SIMS analysis of specific regions of interest for each material (Figure 2). ToF-SIMS spectra obtained in the negative and positive ion modes may reveal distinct compositional and isotopic variations in very small volumes (within nm<sup>3</sup> to μm<sup>3</sup> range). Comparison to EDS spectra can be used to confirm the elemental composition and the possible detection of contaminants in the material. From our initial measurements using inactive materials, we believe that the method can be successfully applied for the routine investigation of individual particles within a broad range of nuclear forensic and safeguards applications.

#### Oral Session #4 – International Cooperation and Exercises / 21

### NUCLEAR FORENSICS IN THE REPUBLIC OF KAZAKHSTAN AND ROLE OF INTERNATIONAL COOPERATION IN ITS DEVELOPMENT

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There are a large number of radiation-hazardous objects of different types located on the territory of the Republic of Kazakhstan: the sites of nuclear testing, research activities and nuclear power plants, the uranium- and oil- mining and processing plants. The large volumes involved with the shipments and transfer of nuclear and radioactive materials in Kazakhstan cause serious risks of illicit trafficking and nuclear smuggling. At the same time, it is necessary to take into account the geo-political position of Kazakhstan, the country which is located in the very center of Eurasia close to such countries as China, Russia, Kyrgyzstan, Uzbekistan, Iran, Afghanistan, etc. Kazakhstan considers the issues of combating illicit trafficking of nuclear and radioactive materials, including nuclear forensics, to be of great importance.

The activities of nuclear forensics are mainly performed in the Institute of Nuclear Physics.

One of the main activities of the Institute of Nuclear Physics is the development and application of

nuclear-physical methods for the analysis of environmental materials including mineral raw materials. Currently, a range of nuclear-physical methods of analysis is available in the Institute of Nuclear Physics, established on the existing spectrometric and analytical equipment, including methods for elemental analysis such as neutron activation analysis, X-ray fluorescence analysis, inductively-coupled plasma mass spectrometric analysis and other instrumental and radiochemical methods of analysis for radionuclides and isotopic composition.

The Institute of Nuclear Physics has designed and developed a special hardware-and-methodological system for the forensics examination of various nuclear and radioactive materials and products. The wide range of available equipment and procedures is applied to the study of the materials/products received for examination.

The Institute is licensed for handling ionizing radiation sources, radioactive substances and waste, and for providing the services in nuclear energy application, including the determination of radionuclides in materials and in the environment; the Institute operates the necessary technical equipment for the wide range of analytical research activities in applied nuclear physics, including specialized laboratories and qualified staff of former “weapon” specialists with experience in similar work.

The quality of analyses is confirmed by successful participation in many international inter-laboratory comparisons and professional tests organized by the IAEA.

Lately, the international cooperation of INP is dynamically developing in nuclear forensics.

The representatives of our Institute have participated in several meetings of the International Technical Working Group (ITWG).

We have participated in various exercises “Csodaszarvas: Mystic Deer”, “Galaxy Serpent”, CMX-5, CMX-6 and planning to participate in CMX-7.

Many workshops, trainings and conferences have been held in Kazakhstan, which were supported by DOE with participation of specialists from several US National Laboratories.

The Inter-laboratory comparison study of nuclear materials from the Lawrence Livermore National Laboratory, US DOE has been completed. Now the partner project of the International Science and Technology Center “Advancement of material-technical and regulation-methodological framework for Nuclear and Radiologic Materials Forensics in the Republic of Kazakhstan” is being implemented jointly with LLNL.

And, of course, one cannot fail to mention the cooperation within the framework of the IAEA programs, in which events such as training at the Hammer training center (USA), training of two of our employees in the laboratory for the analysis of microparticles (Moscow, Russia), and a visit to the Hungarian laboratory nuclear forensics, were conducted. Of particular note is a series of IAEA regional seminars and exercises held at the Microparticle Analysis Laboratory (Moscow, Russia, 2017, 2019 and 2021) and the Institute of Nuclear Physics (Almaty, Kazakhstan, 2019)

### Oral Session #3 – Radiological Crime Scene Management / 22

## Sustaining the US Conventional Forensic Examination on RN Contaminated Evidence Capability

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On June 3, 2010, the FBI Laboratory opened its newest partner facility dedicated to the examination of evidence contaminated with or containing radioactive and nuclear materials at the Savannah River National Laboratory (SRNL). The FBI facility, known as the Radiological Evidence Examination Facility (REEF), opened with great fanfare, with local, state, and federal politicians giving speeches and even a ceremonial ribbon cutting. Eleven years later, with the opening far in the past, the REEF remains the FBI’s premier facility for examining RN (radioactive/nuclear) evidence and evidence contaminated with RN materials. The resilience of the REEF to continue to be operational and withstand regulatory hurdles, budgetary swings, and US policy changes is a result of active sustainment planning by the FBI and SRNL.

Sustainment was built into the REEF’s initial plans. When initial funds were being authorized in 2005, decisions, which seemed simple at the time, continue to play significant roles in the operations at the REEF. Questions such as “What kind of RN materials do we expect?”, “How much evidence do we think we’ll need to process?”, “What kind of examinations are needed?”, and “How large do we expect the evidence to be?” set forth initial construction requirements which to this day bound what

items can be sent to the REEF for examination.

The REEF was built in a phased approach, with the opening of a single RN capable suite in 2007 called the Radioactive Evidence Analysis Laboratory (REALS). This small two (2) room suite was a testbed for what the REEF would eventually become, a 12 room fully capable conventional forensic examination facility within a secure RN facility. The REALS allowed FBI and SRNL facility engineers to work together, learning each other's policies, rules, and regulations, the same ones necessary to create the REEF, but on a much smaller scale. It also allowed the FBI Laboratory's forensic examiners to gather their answers to the above questions using their years of experience processing evidence from crime scenes across the world. All of this was tempered by the underlying thought of "How is this capability going to be maintained in between cases?"

The REEF capability continues to be maintained by a robust exercise and drill program. Yearly, FBI examiners participate in exercises at the REEF where they work mock evidence, using the procedures and practices as if the evidence were contaminated with RN materials. Since 2017, these exercises have incorporated the use of F-18, a short-lived medical isotope. The mock evidence prior to examination is contaminated with the F-18, thereby giving the FBI examiners and the SRNL personnel supporting REEF operations the opportunity to work with real radioactive contamination, test new processes, and become more comfortable working within engineering controls.

Sustainability is and continues to be a key component to the success of the REEF at SRNL.

## Oral Session #2 – Capability Development and Sustainability / 23

### Nuclear Forensics Expert Pipeline Sustainability as a Key Component of Nuclear Security

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The nature of nuclear forensic science as a rarely necessary but crucial component of national security presents many challenges in implementing and sustaining a national nuclear forensics program. One such challenge is recruiting and maintaining scientists with the specific skillsets needed for nuclear forensic analysis, particularly when such analysis is rarely performed or requested by prosecutors. As a result, the U.S. Department of Energy's National Nuclear Security Administration Office of Nuclear Smuggling Detection & Deterrence (NSDD) is increasingly working to address forensic expertise attrition through its capacity building program of assistance. NSDD largely aims to assist partner countries in increasing their capacities to detect, disrupt, and investigate radiological or nuclear (R/N) material found out of regulatory control. The program accomplishes these objectives through a variety of capacity building activities and provision of equipment spanning the spectrum of detecting R/N material to the discussion of technical forensic methods used in support of investigations. To specifically address personnel shortages in nuclear forensics investigation support, NSDD is preparing a new initiative aimed at addressing possible nuclear forensic expert attrition in a subset of countries. The initiative will fund four students to obtain Master's degrees in nuclear forensics-related disciplines in Moldova, Tajikistan, Armenia, and Serbia, and incorporate both in-country and U.S.-based expert mentorship throughout the educational program. Students will be contractually obligated to work within their country of citizenship for a period of two years following the completion of their studies to bolster the technical nuclear forensics structure within their country. NSDD will present on this initiative, highlighting the need for expert recruitment in the field of nuclear forensics.

Through this work, NSDD seeks to improve global resources to investigate and prosecute perpetrators of nuclear smuggling and to strengthen nuclear security culture through expertise sustainment.

## Oral Session #3 – Radiological Crime Scene Management / 25



**Mr**

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Abstract - NUCLEAR FORENSIC SCIENCE SUPPORT TO A NUCLEAR SECURITY EVENT

**Oral Session #3 – Radiological Crime Scene Management / 26**

## **Advancement in Australian CBRN training facilities giving front-line responders a realistic awareness of working in the ‘hot zone’**

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With the current credible terrorism threat level at ‘probable’, Australian state and federal police agencies continue to prioritise CBRNe (Chemical, Biological, Radiological, Nuclear, and high yield Explosives) interagency training for frontline responders. The ANSTO Nuclear Forensics team have provided the RN component of the interagency workshops for Australian law enforcement over the last decade by delivering hands-on demonstrations of current radiation field detection technology and radiological crime scene processes, in realistic ‘hot-zone’ scenarios. Partnering with our law enforcement agencies to deliver this essential service allows emergency teams to keep up to date with their knowledge of radiological detection equipment and their familiarity with best practice in the management of a radiological crime scene, furthering their ability to prepare strategic responses to radiological threats.

With the recent addition of a new, world-class fire and rescue training facility located on the outskirts of Sydney, emergency responders have been provided with the opportunity to run large-scale multi-agency exercises. The facility was completed in 2019 to become the new Fire and Rescue NSW Emergency Services Academy headquarters, with access being extended to other state and federal emergency services for training purposes. The facility includes staged scenes such as rubble following an earthquake, the aftermath of a car crash, a petrol station, an underground railway line, a railway station and a five-storey building with combustible cladding, allowing many CBRN scenarios to be simulated.

In 2020 and 2021, the NSW Police Forensic Evidence and Technical Services Command (FETSC) organised week long CBRN workshops at the academy. The workshops were designed as a development opportunity for staff, team leaders and other technicians working within the forensic framework to exercise response to large scale hazardous scenes. The course included external cooperation from multiple agencies such as Forensic and Analytical Science Service (FASS), NSW Department of Health, NSW Ambulance, Public Order and Riot Squad (PORS), Fire and Rescue NSW, and ANSTO. Three days of the workshop included practical exercises involving separate chemical, biological, and radiological scenes, followed by a full day scenario involving multiple hazards and life threatening situations.

ANSTO’s involvement in the FETSC CBRN training course was giving instruction on best practices for radiological crime scene management, through both theoretical and practical modalities. ANSTO provided a presentation outlining general radiation awareness, an introduction to nuclear forensic science and radiological crime scene operations as well as a practical demonstration on the use of personal dosimeters, radiation detectors, shielding and transport equipment. For the hazardous scenes involving all CBRN components, ANSTO staff were the RN subject matter experts (SME) and provided a 0.37Bq Cesium-137 source to simulate a live threat. Fluorine-18 was also utilised to provide real-world experience operating in a contaminated environment.

With our experience in safely transporting and handling industrial sealed sources and our access to a supply of radioactive material for use in mock crime scenes, we ensured that trainees received first-hand experience using their own specialised detectors in a simulated 'hot zone'. ANSTO's expert guidance and supervision, created a safe environment for our partner agencies to experience and learn about detection of a real radioactive source while leveraging infrastructure developed for fire and rescue training.

## Oral Session #7 – Signature Research on Isotopic Signatures and Age-Dating / 27

### Measurement and validation of the isotopic composition in inhomogeneous samples by LA-MC-ICP-MS

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#### Introduction

Uranium-bearing materials are strictly controlled by the international nuclear safeguards system. However, if such materials get out of regulatory control and subsequently confiscated, a comprehensive characterization is required to assess the hazard, to determine the intended use and the potential origin. Nuclear forensics focuses on the analysis of these intercepted nuclear or other radioactive materials to provide information for the investigating authority to avoid the diversion and subsequent malicious use [1-3].

Most of the nuclear forensic samples contain visible or non-visible inhomogeneity, which can be exploited for potential nuclear forensic conclusions. One of the available micro-analytical techniques for such measurements is laser ablation inductively coupled plasma mass spectrometry with simultaneous detection (LA-ICP-MS) [4,5]. As LA uses a focused laser beam scaled down to a few micrometers, LA-ICP-MS analysis can reveal sample inhomogeneity in the material in question and is able to measure the spatial distribution of the isotopic composition. However, the precise measurement can be hindered by instrumental parameters (e.g. laser beam size or scan speed) or by the sample characteristics (e.g. grain size or differences in U isotope enrichment) [6].

#### Present Work

The present work examines the nuclear forensic value of U isotopic inhomogeneity for by LA-ICP-MS measurements. To study the important parameters, a synthetic sample was prepared by the mixing of two solid certified standard materials (SRM U-010 and SRM U-030) to mimic an inhomogeneous U sample. By a rough LA measurement using a line scan or a 2-D area (map) on the sample surface, the points-of-interest in the inhomogeneous sample can be found and chosen (localization). After finding the required sample position, a more precise measurement can be performed on the selected small locations. The procedure allows the accurate analysis of the isotopic composition at the relevant spots and the proper identification of the end-products (i.e. the constituting starting materials). The developed LA-ICP-MS method was also compared with the large-geometry secondary ion mass spectrometry.

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## Poster Session 2 / 28

# Preparation and validation of Pu age dating materials for nuclear forensics

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## Introduction

Nuclear materials are subject to strict verification by the international nuclear safeguards. However, the IAEA's Incident and Trafficking Database records year by year cases where such a material is found out of regulatory control. Such an interception is typically followed by a comprehensive analysis of the material in order to provide hints on its intended use and potential origin. Nuclear forensics analysis provides information to the investigating authority to prevent malicious use, prosecute the perpetrators and to avoid further diversion [1-3].

Among the significant signatures in nuclear forensics, age dating (also known as the determination of the production date), was found to be a very valuable sample characteristic. The method is based on the radioactive decay of the parent radionuclide into the daughter nuclide: the measured parent-daughter ratio (also known as radio-chronometer) indicates the date of a specific production step (e.g. parent/daughter chemical separation step, metal casting), and it is colloquially referred to as the "age" of the material. This is a self-explanatory signature, i.e. it does not require a comparative dataset for the evaluation. The method is quite well established for uranium-bearing samples, but it has been less studied for plutonium materials [4] and would particularly benefit from a sound metrological basis.

## Present Work

The present work describes a method for the preparation of plutonium age dating reference materials (similarly to the uranium IRMM-1000 certified reference material) and its independent validation by an international co-operation. The prepared samples can be used to validate experimental protocols for determining the production date of plutonium via the  $^{234}\text{U}/^{238}\text{Pu}$ ,  $^{235}\text{U}/^{239}\text{Pu}$ ,  $^{236}\text{U}/^{240}\text{Pu}$ , and  $^{241}\text{Am}/^{241}\text{Pu}$  chronometers. Reactor-grade plutonium was used as starting material and chemically purified using a dedicated method to ensure high Pu recovery, while maximizing U and Am separation efficiencies. The U and Am separation factors were determined by the addition of high amounts of  $^{233}\text{U}$  and  $^{243}\text{Am}$  spikes and their re-measurement in the final product. The prepared material is intended for quality control and assessment of method performance in nuclear forensics and safeguards [5].

In order to establish the proof-of-concept for a methodology for developing a Pu age dating reference material and to confirm our age dating results, several subsamples with various Pu concentrations and physical forms (solid and liquid) were sent to expert nuclear forensic laboratories (Lawrence Livermore National Laboratory and Los Alamos National Laboratory in the USA, AWE in the United Kingdom, CEA in France, FOI in Sweden and International Atomic Energy Agency laboratory in

Seibersdorf). The co-operation allows detecting any problems and issues related to the preparation and tailoring the Pu age dating reference material to the laboratory needs.

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#### Poster Session 4 / 29

### IMPLEMENTATION OF TRADITIONAL FORENSICS METHODS AND PROCEDURES WITHIN THE NUCLEAR FORENSICS LABORATORY OF ROMANIA

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When conducting a criminal investigation in which nuclear or other radioactive material is present, an important role will be played by the application of traditional forensic methods in analyzing the evidences contaminated with radionuclides.

Fingerprints found on the contaminated evidence can lead to the identification of the perpetrators by using the judicial or law enforcement national fingerprint databases. An effective tool in supporting criminal investigations by providing information on the fingerprints consists in cyanoacrylate vapor method.

In order to implement the traditional method of developing latent fingerprints on non-porous surfaces at the Romanian National Nuclear Forensics Laboratory (NNFL-RO), the Lumicyano substance was evaporated inside a two-chamber Plexiglas system equipped with gloves, electric hot plate, humidifier and defumigator. Special humidity conditions were established for this method.

This paper describes the experimental setup, the lesson-learned and good practices on implementing this method on non-porous surfaces using Lumicyano.

## ONIX: An open-source depletion code with nuclear forensic applications

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Nuclear forensic interpretation is an unavoidable stage of the nuclear forensic process: it transforms the analytical results obtained at the stage of characterization into meaningful information useful for formulation of the nuclear forensic findings. As has been demonstrated by the ITWG's exercises CMX-1 and Galaxy Serpent 1 and 4, a capability to understand and use nuclear forensics signatures pertinent to nuclear material irradiated in a reactor is an important part of nuclear forensic interpretation process. It is also often required for operating a national nuclear forensics library [1].

Nuclear depletion codes assist nuclear forensic interpretation by modelling the isotopic evolution of nuclear and non-nuclear materials through neutron irradiation and natural decay. Unfortunately, their proprietary nature and various export control regulations restrict the distribution of these codes, reduce the degree of transparency, and complicate the collaboration between groups from different countries which may be required in case of investigating a cross-border nuclear smuggling incident. The success of these collaborative efforts depends enormously on trust between the different parties involved and the transparency of the technologies used [2].

ONIX (for Open IsotopiX) overcomes these limitations by offering a fully open-source depletion software that can model neutron irradiation and natural decays for any materials. Coupling to the open-source Monte Carlo neutron transport code OpenMC allows ONIX to model systems as complex as nuclear reactors [3]. The code uses state-of-the-art algorithms and nuclear data sets. ONIX has been validated and verified through comparison with multiple numerical and experimental benchmarks [4].

ONIX includes the NAX module (for Nuclear Archaeology and forensiX) which has been specifically designed to be a practical numerical tool for technical tasks relevant to nuclear forensics. The NAX module has the following key functionalities:

- Deplete any type of materials from nuclear fuel to structural materials in a nuclear reactor;
- Simulate the depletion of materials with complex irradiation history, e.g. structural materials in reactors spanning multiple fuel cycles;
- Find the best isotopic ratios that can be measured for fluence estimation for a nuclear reactor design and operation history defined by the user;
- Visualize individual nuclides production and destruction paths at any stage of irradiation or decay.

ONIX is currently used by several researchers in various institutions [5], [6], [7], [8]. This paper will present ONIX to the nuclear forensics community and demonstrate different ways to apply it to nuclear forensic science.

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## **Building and Maintaining National and International Partnerships through Virtual Engagements in Investigation Support**

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The mission of the U.S. Department of Energy's National Nuclear Security Administration (DOE/NNSA) Office of Nuclear Smuggling Detection and Deterrence (NSDD) is to strengthen the capabilities of partner countries to detect, disrupt, and investigate the smuggling of nuclear and radioactive material. NSDD works with over 80 national and international partners to achieve its mission. The ongoing COVID-19 pandemic has adversely impacted the ability to carry out in-person engagements and interactions. To address this challenge, former in-person engagements such as Scenario-Based Policy Discussions and scoping visits for building and maintaining partnerships were modified to virtual engagements to continue building partner countries' capabilities to respond to nuclear security events. These unique virtual engagements focus on developing or exercising collaboration between different stakeholders in a partner country to encourage information sharing and to clarify the role of each stakeholder in supporting investigations.

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## **The Pivot to Virtual Engagement in International Nuclear Forensics**

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In response to unprecedented global challenges including the 2020 coronavirus pandemic, nuclear forensics international engagements pivoted to virtual meeting formats and emerged with stronger, resilient and lasting partnerships. The need for international collaboration in nuclear security has never been greater; the security of the global nuclear fuel cycle involves extraction, refining and processing sites often far removed from reactors and industrial sites that utilize nuclear and other radioactive material for diverse applications. Reliance on nuclear and other radioactive materials continues unabated with the accompanying potential for low probability, high consequence malicious use by terrorists. The International Atomic Energy Agency's (IAEA) Incident and Trafficking Database reported 140 confirmed incidents of nuclear and other radioactive material out of regulatory control in 2019 and 116 confirmed incidents in 2020.

The scope of the change in engagement driven by the pandemic affected nuclear forensics implementation at all levels: institutionally, nationally, regionally and internationally. The objectives of nuclear forensics outreach remain as before: to identify, develop and socialize best practices in the

field of nuclear forensics. The challenge is to ensure the resilience of virtual engagements to allow for the full spectrum of coordination, research, training, peer-reviews, and outreach to other communities – to include law enforcement –while maintaining timeliness as well as the highest confidence in the provision of this information.

Using web-based tools including video conferencing, webinars, podcasts, on-line e-learning, as well as electronic dissemination of newsletters and updates, the nuclear forensics community adapted rapidly. Concerted effort was made throughout to understand the needs and preferences of end-users for receiving nuclear forensics assistance; questionnaires and anonymous polling were effective to focus virtual messaging and outreach.

Several examples are highlighted. Within working nuclear forensic laboratories, periodic (monthly) technical and programmatic meetings were scheduled to promote cohesion and coordination in operations as physical accesses were limited. In place of in-person research meetings, different technical topics were presented virtually each month by principal investigators augmented by brief status reports from scientists and examiners. Inside the academic setting, in-person instruction was curtailed, research activities modified, and replaced by an array of virtual information streams to include nuclear security seminar series and podcasts. International assistance adapted; the IAEA embarked on a series of webinars in 2021 to include operational perspectives connecting radiological crime management and nuclear forensics as well as offering like e-learning modules. International nuclear forensics conferences also shifted formats without sacrificing technical content; the NuFor meeting organized in the United Kingdom focusing on emerging science and early career investigators was conducted entirely virtually in 2020 and was convened in a hybrid (in-person and virtual) format in 2021.

To best provision international assistance, the Nuclear Forensics International Technical Working Group (ITWG) polled its membership and, not unexpectedly, results indicated a strong preference for regularly scheduled (e.g. monthly) webinars. This virtual series commenced in October 2020. A year later, eight webinars have been conducted including a presentation on the development of uranium ore concentrate reference materials, status reports on prior and current ITWG collaborative material analytical exercises (CMX) as well as outcomes from virtual exercises involving national nuclear forensic libraries (e.g. Galaxy Serpent). Additional subjects included the recovery of evidence contaminated by radionuclides, laboratory protocols for analysis of solids and powders, as well as radioactive source identification. Beyond the webinar series, a highlight for ITWG virtual engagement was the working group's first all virtual annual meeting convened over a four-day interval in June 2021. This meeting featured technical presentations from the ITWG Nuclear Forensics Laboratories (INFL) to include updates from nuclear forensic laboratories globally. Plenary sessions featured a 25-year retrospective on the establishment and technical evolution of the ITWG by its founding members, updates from the ITWG's international partners (IAEA, Global Initiative to Combat Nuclear Terrorism) as well as standing ITWG task group reports. Over 130 experts participated from more than 30 states and international organizations.

A mix of in-person, virtual and hybrid engagements will certainly become the 'new normal' for international engagements in nuclear forensics. Recent success in virtual engagements will further strengthen international nuclear forensics in the years ahead.

#### Oral Session #4 – International Cooperation and Exercises / 33

### The Joint Romania – U.S. Nuclear Forensics Examination of Legacy High Enriched Uranium (HEU) Materials

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The Horia Hulubei National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH) in Romania is participating in a joint sample analysis with Lawrence Livermore National Laboratory (LLNL) and Los Alamos National Laboratory (LANL) on a set of legacy high enriched uranium (HEU) materials that have been under safeguards at the IFIN-HH for many decades.

Initiated in 2019 as part of larger collaboration between the U.S. Department of Energy and the Romanian government, this project aims to facilitate a closer cooperation between the U.S. laboratories and IFIN-HH in the field of nuclear forensics, and create opportunities for further projects of mutual interest on both the national and regional levels. The pilot-project titled “Nuclear Forensic Characterization of Legacy Uranium Samples” involves the analysis of a set HEU metal samples with a nominal enrichment of ~93 wt.% U-235, as well the exchange of technical procedures and staff during lab demonstrations and data review meetings. For this project, a total number of five metal samples of interest were selected at IFIN-HH: ten swipe samples (two from each sample) were prepared and shipped to LANL and LLNL, and two solid samples were subsampled and are awaiting the shipment approvals.

A comprehensive nuclear forensics examination of the selected uranium metal samples was completed at IFIN-HH in Romania using a wide range of non-destructive and destructive analysis techniques. Age dating of the samples using gamma spectrometry at IFIN-HH showed a production age of around 1969-1970 for all samples, while the uranium isotope ratio measurements by inductively-coupled plasma mass spectrometry (ICP-MS) indicated a significant difference in the U-236 relative concentration. The U-236 concentration in the samples was confirmed by the U.S. labs through the analysis of the particle swipes provided by IFIN-HH using both secondary ion mass spectrometry analysis (SIMS) of individual particles and digestion and ICP-MS analysis of the entire swipe for each sample. The digestion and ICP-MS analysis of the entire swipe for each sample was also used to determine the radiometric ‘age-dates’ of the samples. The data seemed to indicate a difference in processing history between the samples that resulted in an increased U-236 ratio for a subset of the materials. The preliminary results of this study also showed how different nuclear forensic signatures (e.g., sample age, isotopic and chemical characteristics) and analytical approaches (particle analysis vs. bulk) can provide clues on sample origin and history.

#### **Oral Session #1 – Legal Framework and the CPPNM/A / 34**

### **Overview of criminal legislation and nuclear forensics capabilities in Western Balkan countries**

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The Western Balkan (WB) countries are Albania, Bosnia and Herzegovina, Montenegro, North Macedonia and Serbia. All of them except Bosnia and Herzegovina are European Union (EU) membership candidates. Western Balkan countries are a part of the European neighborhood that is also close geographically and in terms of trade and smuggling routes to the Black Sea region, which is widely recognized as an area worst affected by illicit trafficking of nuclear and other radioactive materials. The Western Balkans, forming a land bridge and the shortest transit route between the south-east flank of the EU (Greece, Bulgaria and Romania) and its central European ‘core’ (Hungary, Croatia, Slovenia and Austria), is arguably the part of the European neighborhood that merits significant attention from the EU in terms of response to and prevention of nuclear smuggling.



All the governments in WB region are legally and politically bound to support development of nuclear security, including radiological crime scene management and nuclear forensics capabilities. All mentioned countries have endorsed, signed and ratified multiple pertinent international treaties and other legal mechanisms, including the Treaty on the Non-Proliferation of Nuclear Weapons (NPT), Convention on the Physical Protection of Nuclear Material (CPPNM) and its Amendment, and International Convention for the Suppression of Acts of Nuclear Terrorism (ICSANT). All the WB countries have already started incorporating the legal requirements into their national legislation.

Currently Serbia and other WB countries have legal ban on the use of nuclear power. That ban is expected to be lifted soon and therefore, the nuclear and other radioactive material holdings in the region will increase. Despite the recognized need for nuclear security capabilities development in WB, the gaps in availability of equipment and lack of practical experience and expertise in the area of nuclear forensics and radiological crime scene management persist. For example, in most WB countries, national nuclear security response plans are being developed, but they are still to be adopted and made comprehensive. Language barriers and other factors have contributed to a lack of systematized information about the criminal legislation and capabilities related to nuclear security in general and nuclear forensics in particular. This paper addresses this issue by giving a clear overview of existing legal arrangements and practical capabilities for nuclear forensics and radiological crime scene management in Western Balkan countries. It will also analyze that overview and provide recommendations on potential next steps towards optimal nuclear security capabilities.

#### **Oral Session #5 – Analytical Methods for Analysing Radiological and Nuclear Evidence / 35**

### **NON-DESTRUCTIVE ASSAY OF INDUSTRIAL GAMMA RADIOGRAPHY DEVICES: CASE STUDY**

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This paper describes the characterization of a large number of depleted uranium-based gamma radiography devices of different manufacturers and origins. It presents an application of different types of non-destructive analysis techniques that can provide quick information on the history and origin of the studied samples, thus assisting the criminal investigation and prosecution in cases when such materials are found outside the regulatory control. A general overview of a recent Romanian case in which gamma radiography devices served as evidence, is also provided.

#### **Oral Session #1 – Legal Framework and the CPPNM/A / 36**

### **Use of Nuclear Forensics Capacities to Support National Enforcement of and Demonstrate Compliance with International Nuclear Counter-Terrorism Legal Instruments**

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The development of national nuclear forensic science, commonly referred to as nuclear forensics, capacities offers states a meaningful mechanism to contribute to enforcement of national laws related

to nuclear or other radioactive material out of regulatory control in line with key international nuclear counter-terrorism legal instruments.

While no single encompassing international legal instrument mandates a state's compliance with nuclear security norms or standards, there are a series of six nuclear counter-terrorism international legal instruments that may be associated with the implementation of nuclear forensics, to include, inter alia, the International Convention on the Suppression of Acts of Nuclear Terrorism (ICSANT), the Convention on the Physical Protection of Nuclear Material (CPPNM) and its 2005 Amendment (A/CPPNM), as well as United Nations Security Council Resolutions 1540.

Nuclear forensics provides law enforcement and nuclear security investigators with a technical basis for answering key questions related to the seizure of nuclear and other radioactive material out of regulatory control. Nuclear forensics, like all forms of forensic science, supports to implementation of national laws and demonstrates compliance with international legal instruments through investigative science.

Over the past thirty years, nuclear forensics has grown from a field of research using analytical methods developed for defense, environmental analysis, and nonproliferation missions to a sophisticated analytical capability that allows states to respond to incidents involving nuclear and other radioactive materials that are smuggled, stolen, lost, or abandoned.

Nuclear forensics is unique among other technical disciplines of nuclear security. In support of measures to prevent, detect, and respond to nuclear or other radioactive material out of regulatory control, nuclear forensics provides the technical information necessary to assess questions from law enforcement and nuclear security investigators in the context of national laws and international legal instruments related to nuclear security, as well as information outputs that allow a state to better understand the efficacy of its domestic nuclear security architectures. As understanding of the scope and application of nuclear forensics has grown, it has been regularly applied by law enforcement and prosecutorial bodies to determine if laws pertaining to unauthorized or malicious acts involving nuclear and other radioactive materials have been broken, determine the origin, history, and route of interdicted materials, as well as determine the efficacy of national nuclear security architectures; all of which can be tied to specific provisions contained within international nuclear counter-terrorism legal instruments.

While nuclear security is the responsibility of the state, nuclear forensics fosters international technical coordination and cooperation, and provides a basis for states to support national enforcement of and demonstrate their compliance with international nuclear counter-terrorism legal instruments.

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## Oral Session #2 – Capability Development and Sustainability / 37

### Using a National Nuclear Forensics Library to Address Gaps within a Nuclear Security Infrastructure

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#### Introduction

Seizures of nuclear and other radioactive material out of regulatory control (MORC) have created widespread concern over the possibility of a non-state actor acquiring sufficient nuclear or other radioactive (R/N) material for malicious use. Anytime MORC is recovered, law enforcement and

nuclear security investigators rely on an examination of the forensic characteristics of the material for identification and to assess its provenance.

One mechanism states can adopt that provides a record of nuclear material characteristics and subject matter expertise key to the identification and assessment of MORC is a National Nuclear Forensics Library (NNFL).

#### **What is an NNFL and how is it applied during a nuclear smuggling investigation?**

An NNFL, as an integral element of a state's nuclear security architecture, is a national system for the identification of MORC and assessment of material provenance. It is composed of the technical and administrative information on R/N material produced, used, or stored in the state, and the associated subject matter expertise necessary to use that information to answer investigative questions.

Providing valuable support to investigators of MORC incidents, an NNFL enables investigators to make rapid comparisons of interdicted R/N material with domestic holdings and can help to exclude domestic materials that are inconsistent with measured materials characteristics. A variety of important questions can be addressed with help from an NNFL, including:

- What is the material and what threat does it pose?
- From which part of the fuel cycle was the material derived?
- List item material consistent with the state's holdings?
- Is there more material missing?

#### **How can an NNFL be used in a wider nuclear security context?**

As illicit trafficking of R/N material is largely transnational in nature, determining where R/N material left regulatory control is essential in identifying nuclear security gaps. For example, uranium may be mined and milled at one location, isotopically enriched at a second location, manufactured into fuel pellets at a third location, shipped to a fourth location for assembly, and finally transferred to the site of end use. The material could be diverted from any of these locations or transit routes. If the characteristics of the uranium in question are comparable to domestic inventories, then this data may facilitate the identification of possible diversion points or points-of-entry.

When R/N material is found outside of regulatory control, the capability to determine the origin of material and process history can prove invaluable in identifying and addressing weaknesses in a state's nuclear security infrastructure. It is also potentially important for identifying previously unknown historic security weaknesses that may have resulted in the diversion of other R/N material still circulating outside of regulatory control.

While the utility of an NNFL remains essential throughout law enforcement and nuclear security investigations, it also provides states with strong mechanisms to make important determinations about the health of their domestic nuclear security infrastructure. The information gleaned from use of an NNFL can be leveraged to develop targeted nuclear security remediations for identified gaps.

#### **Conclusion**

The ability to include or exclude likely origins of MORC provides a state with the information it needs to both respond effectively to MORC and determine whether there are current or historic gaps within its operational nuclear security system. As such, an NNFL supports all three nuclear security pillars -prevention, detection, and response - and is a key nuclear security investment by any state that produces, uses, or stores R/N materials.

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Gamma spectrometry plays an important role as a non destructive method in nuclear forensics. In this work an evaluation of the analysis of different uranium containing materials, measured on two High Purity Germanium (HPGe) detector types (planar and coaxial) and analyzed using four software packages is presented. For the purpose of the analysis efficiency transfer software Angle and EFFTRAN were used in comparison to Geant4 and MCNP simulation codes. Both EFFTRAN [1] and Angle [2] present dedicated detection efficiency calculation software, and are based on the efficiency transfer (ET) method. The mechanism of this method is calculating the ratio of the efficiencies for the sample of interest and for the calibration sample and by multiplying it with the measured Full Energy Peak Efficiency (FEPE) of the latter. Monte Carlo simulation, based on Geant4 simulation package [3], has been developed to obtain the response of germanium detectors, with the aim to reproduce experimental spectra of detectors in a wide range of applications in gamma spectroscopy measurements. In the present work, the Geant4 software (version 4.9.5.) was used to obtain the detection efficiencies for different geometries of voluminous sources placed in front of the HPGe detector. As an addition to the Geant4 simulation results, the detection efficiencies were also computed using the MCNP Monte Carlo transport code version 6.2 [4].

Detector 1 is a HPGe low background extended range coaxial detector made by ORTEC with a serial mark GMX-20190. It is an N-type coaxial detector, with a crystal that has a radius of 28.0 mm, a height of 65.2 mm, and an active volume of 160 cm<sup>3</sup>. Being extended range type, it has a beryllium window which is 99.8 % pure and is 0.5 mm thick. The detector has a 32.4 % relative efficiency, an energy resolution (FWHM) of 1.92 keV at 1.33 MeV, and a Peak to Compton ratio of 54.1. Detector 1 is shielded with multi-layer lead, tin, and copper shielding. The shielding is composed of an outer 120 mm layer of refined low background Pb, 3.5 mm of Sn, and an inner layer of 0.5 mm of a high purity Cu.

Detector 2 is a HPGe planar detector, made by CANBERRA with a serial mark GL2020R. It has a 2000 mm<sup>2</sup> active crystal area and 50.5 mm active diameter, with a detection range from 10 keV to 1200 keV. Detector crystal height is 20 mm. The nominal resolution (FWHM) at 122 keV is 680 eV.

Uranium containing samples were measured on both detectors and the obtained spectra were analyzed by EFFTRAN and ANGLE software. The obtained results of the efficiencies and activity concentrations were compared with the simulation results of Geant4 and MCNP. The advantages and disadvantages of the use of different detector types (coaxial and planar) were discussed as well as suitability of the use of mentioned software codes in the analysis.

The results supported the improvement of gamma spectroscopy spectrum analysis methods for the application as a non-destructive tool in nuclear forensics analysis. As an outcome of the project, a close scientific collaboration between research teams from neighbouring states (Serbia and Hungary) was established.

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#### Oral Session #4 – International Cooperation and Exercises / 39

### Celestial Skónis: the 6th Collaborative Materials Exercise of the Nuclear Forensics International Technical Working Group

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The Nuclear Forensics International Technical Working Group (ITWG) recently completed its sixth Collaborative Materials Exercise (CMX-6) – Operation Celestial Skónis. This latest CMX also represented the largest exercise on record in the 25-year history of the ITWG and included participation by 22 laboratories and 15 law enforcement (LE) agencies from 21 countries and one multinational organization. Celestial Skónis was a paired-comparison exercise, in which more than one sample is distributed to participating laboratories for comparison purposes. As is customary with past CMXs, laboratories participating in Celestial Skónis were asked to analyze nuclear and radiological materials as part of a mock nuclear forensic investigation. However, participants of Celestial Skónis were also asked to examine contaminated and radioactive items for conventional forensic evidence (e.g., fingerprints, toolmarks, etc) – an activity that had only been incorporated once before during CMX-2 (2001). A primary outcome of CMXs, participants identify and share best practices in nuclear forensic science during the Data Review Meeting following the exercise (see Figure 1).

Nuclear materials distributed to participants included depleted uranium (DU) metal and minute amounts of DU- and plutonium (Pu)-oxy-fluoride powders. Nonradioactive items comingled/contaminated with these materials were also distributed to support examinations for conventional forensic evidence. A scenario was developed in which authorities discovered radioactive materials (DU metal) out of regulatory control at a metal recycling facility. This discovery led to additional seizures of nuclear materials and contaminated evidence at a nearby metal foundry and storage locker. Forensic examinations of contaminated evidence and analysis of the nuclear materials were requested by LE to connect people, places, things, and events.

Laboratories used a total of 36 nuclear forensic protocols, procedures, and analytic techniques to examine contaminated evidence and analyze materials. Results from these examinations were used to characterize evidence and compare sample characteristics with each other and with a mock library of declared material holdings. Participants submitted preliminary reports after 24 hours, and again after one week, with a final report due to the exercise organizers after two months.

Gamma spectroscopy was an indispensable tool during Celestial Skónis, representing the only “State-of-Practice” technique (defined as a technology used by more than half of the exercise participants) capable of categorizing nuclear forensic evidence nondestructively within 24 hours. Gamma spectroscopy was also valuable at the one week reporting timeline, providing more accurate and precise measurements of several Pu and U isotopes than mass spectrometry over the same time period. However, by the two month period, mass spectrometry regained its standing as the most accurate means for measuring most U and Pu isotope ratios within a bulk sample. This level of precision, while not needed to conclude Exercise Sample (ES)-1 and ES-2 were linked, was eventually needed to confirm with high confidence the consistency of ES-1 and ES-2 to library of holdings.

There were several other noteworthy observations related to nuclear forensic analysis during Celestial Skónis. For instance, particle analysis by secondary ion mass spectrometry (SIMS) was again demonstrated by a small minority of laboratories to be one of the most powerful nuclear forensic tools available today. The ability of this technique to separate comingled sources of radioactive surface contamination during Celestial Skónis and allow laboratories to exploit Pu and U isotopic distributions of individual particles is unmatched by any other analytical technique today. The use of alpha spectroscopy by participants experienced a bit of a resurgence during Celestial Skónis as well, driving by the need to measure <sup>238</sup>Pu and <sup>241</sup>Am after decline in the use of this technique over the past three (U-only) materials exercises.

A special thank you is extended to the laboratories and LE agencies that participated in Celestial Skónis.

## Oral Session #6 – Novel Techniques Applied to Nuclear Forensic Examinations / 40

### Nuclear Forensics Capacity Building: New Technologies, Research & Development and Signature Research in Nuclear Forensics

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### **Introduction**

The Virtual Laboratory on Age Dating for Investigation Support (VLADIS) is a consortium of researchers from Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Australia's Nuclear Science and Technology Organisation (ANSTO), and the National Atomic Energy Commission of Argentina (CNEA). The VLADIS initiative was launched in October 2020 with the goal of creating and maintaining an online platform for practitioners of radiochronometry (age dating) in the field of nuclear forensics and investigation support. This initiative was born out of LLNL and LANL's successful and long-running collaboration with ANSTO through the U.S. Department of Energy's National Nuclear Security Administration (DOE/NNSA) Office of Nuclear Smuggling Detection and Deterrence.

VLADIS aims to facilitate discussion between subject matter experts (SMEs), new staff, and partner agencies around the ever-evolving best practices of radiochronometry within the primarily online environment required by the COVID-19 pandemic. Whereas the group is organized by researchers at LLNL, it is facilitated by nuclear forensics technical analysts from all participating laboratories, including experts in chemistry, mass spectrometry, data analysis, and nuclear forensic signature evaluation. This initiative is not meant to replace in-person visits and technical exchanges between laboratories, rather, VLADIS will complement overall engagement between in-person meetings. The VLADIS initiative is being used as a pilot project for virtual information sharing and could be re-designed for use with other partner countries covering topics beyond radiochronometry. In addition to providing a forum for discussing the technical details of an age dating evaluation, VLADIS is a community where open communication and collaboration is the model, aiding both the science and the scientists.

The Nuclear Forensics International Technical Working Group provides a set of guidelines that outline how to prepare nuclear materials, measure their model production ages, and interpret these ages in the context of a nuclear forensic examination in support of a nuclear security investigation (nfitwg.org). VLADIS seeks to build on this guidance by bringing together international colleagues at various stages in their careers for in-depth discussions on the technical aspects of age dating analysis, thereby establishing best practices for radiochronometry in the community. Monthly meetings, facilitated by nuclear forensics technical analysts from all participating laboratories, are held online at a time convenient for all international participants, which allows all participants to join for real-time discussions. As an example of the initiative's value, Argentina recently participated in an International Atomic Energy Agency Residential Assignment at LLNL in radiochronometry; through VLADIS this partnership has continued and expanded through use of online meeting platforms and our group of gathered experts.

As a supplement to online meetings, the virtual platform 'Slack' is used to facilitate and increase informal communication between the SMEs and users, as well as enable document sharing and archiving. Using these two platforms allows participants to communicate in a variety of ways, from "live" virtual discussions and presentations to posting/answering questions from one another and sharing relevant materials on Slack.

The content of VLADIS is participant-driven, based on individual laboratories' capabilities and specific needs. As such, meeting topics are updated routinely to reflect current challenges, needs, or interests of the group. Relevant literature, externally releasable materials (e.g., white papers), and technical reports are distributed via the Slack channel to foster communication.

**Summary:** VLADIS has bolstered the working relationships between participants and SMEs at LLNL, LANL, CNEA, and ANSTO in the area of radiochronometry. VLADIS can be viewed as a pilot program of active information sharing and collaboration between partner agencies that could be applied to additional partner countries, tailored to their specific capabilities and interests.

## A Cooperation to Improve $^{231}\text{Pa}/^{235}\text{U}$ Age Dating Measurements of Uranium for Nuclear Forensics

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### Introduction

Increased requests for model age determination of nuclear material during nuclear forensic assessments have resulted in broad efforts by nuclear forensic laboratories to establish  $^{231}\text{Pa}/^{235}\text{U}$  radiochronometry capabilities. Ingrowth of  $^{231}\text{Pa}$  from decay of  $^{235}\text{U}$  in chemically purified uranium provides a daughter/parent chronometry pair with which to age date uranium. The application of  $^{231}\text{Pa}/^{235}\text{U}$  (as a second, independent chronometer) in combination with  $^{230}\text{Th}/^{234}\text{U}$  can significantly expand confidence when determining model ages of uranium with unknown origin. However, developing a  $^{231}\text{Pa}/^{235}\text{U}$  radiochronometry capability is challenged by a lack of available certified reference materials for quality control, as well as the short half-life of  $^{233}\text{Pa}$ , which is used as the 'spike' isotope for isotope dilution analysis of  $^{231}\text{Pa}$ . In an effort to address these challenges, the U.S. Department of Energy/National Nuclear Security Administration and the European Atomic Energy Community entered into a Joint Action Sheet for a 'Cooperation on  $^{231}\text{Pa}/^{235}\text{U}$  Age Dating Measurements of Uranium for Nuclear Forensics.' An interlaboratory study between Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), and the European Commission Joint Research Centre Karlsruhe (EC-JRC) was initiated in 2016.

### Technical Work

The technical partnership between LANL, LLNL, and EC-JRC focused on interlaboratory  $^{231}\text{Pa}/^{235}\text{U}$  model age determination in order to assess radiochronometry reproducibility and produce open-source  $^{231}\text{Pa}/^{235}\text{U}$  model ages of a suite of uranium materials for the forensic community. An interlaboratory comparison was designed to jointly develop  $^{231}\text{Pa}/^{235}\text{U}$  chronometry capabilities and measure model ages for three certified reference materials (CRMs): New Brunswick Laboratory CRM U100, CRM U630, and CRM 125-A. The scope of work included an exchange of procedures for the preparation and characterization of a  $^{233}\text{Pa}$  spike and radiochemical purification and mass spectrometry determination of  $^{231}\text{Pa}$  and  $^{235}\text{U}$  in bulk uranium materials. A newly produced  $^{231}\text{Pa}$  Nuclear Forensic Reference Material [1] was distributed between LANL, LLNL, and EC-JRC to support  $^{233}\text{Pa}$  spike calibration. Each laboratory used independent methods to determine  $^{231}\text{Pa}/^{235}\text{U}$  radiochronometric model ages for separate units of CRM U100, CRM U630, and CRM 125-A [2, 3, 4]. The results obtained by the three laboratories are in very good agreement.

### Summary

All laboratories have established  $^{231}\text{Pa}/^{235}\text{U}$  capabilities, and results of the interlaboratory  $^{231}\text{Pa}/^{235}\text{U}$  comparison will be presented here and interpreted in the context of nuclear forensics. The analytical results from this project support ongoing efforts to establish consensus  $^{231}\text{Pa}/^{235}\text{U}$  ages for uranium CRMs for quality control to support nuclear forensic assessments for law enforcement and attribution. In addition to presenting analytical data, we will provide lessons learned and technical recommendations for the forensics community.

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### Poster Session 3 / 42

## Optically stimulated luminescence and thermoluminescence dosimetry for nuclear forensics

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The aim of retrospective dosimetry is the determination of absorbed dose after a certain „radiological event” (e.g. accidents or terrorist attacks), when no dosimeters were used. These measurements are usually performed by applying thermoluminescence (TL) or optically stimulated luminescence (OSL) dosimetry methods.

Retrospective dosimetry is an auxiliary science of Nuclear Forensics, capable from rapid accident dose reconstruction after a possible terrorist event, to tracking of gamma-emitting radiological materials. For decades, retrospective dosimetry has been used for dose estimation of bricks and other radiation-sensitive building materials and household objects. In recent years, TL and OSL measurements of components from electronic devices (mobile phones, pagers, portable computers, music and video playback devices, cameras, digital watches, cars) have been tested. Their surface-mounted components such as resistors, surface-mounted capacitors and diodes, inductors, integrated circuits, smart chip cards including subscriber identification module (SIM) cards, glasses and displays produce material-specific TL and OSL responses, from which the dose absorbed by the given component can be estimated.

OSL dosimetry research was launched at the Nuclear Security Department from 2021 and based on the Belgian Nuclear Research Centre's (SCK•CEN) sample preparation, OSL data evaluation and OSL protocol.

The idea of this paper was to test possibilities of Monte Carlo simulations for the application in the retrospective dosimetry. The obtained experimental data were used to test the simulation code written in Geant4.

If the obtained simulation results are in good agreement with experimental data, than the similar code with some adjustments may be used in future investigations to test the possibilities of using other materials as a retrospective dosimeters. In that case, simulation results will be a part of initial research as a confirmation that experimental work is sustainable.

keywords: retrospective dosimetry, thermoluminescence, optically stimulated luminescence, nuclear forensics

### Oral Session #3 – Radiological Crime Scene Management / 43

## Hungarian Procedure for Radiological Crime Scene Management



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Nuclear or other radioactive materials can be a target of criminal acts and terror attacks, as these materials can be effective tools used in a variety of radiological weapons as in radiological dispersal devices (RDD) or in radiological exposure devices (RED). These weapons can be effective for panic generation and causing economical damage at a state. Illicit trafficking and smuggling of nuclear or other radioactive materials is a real existing problem worldwide. The importance of radiological threat can be recognized through that the major international organizations such as the International Atomic Energy Agency (IAEA) or the Global Initiative to Combat Nuclear Terrorism (GICNT) place great emphasis on this area and seek to raise awareness of the threat and consequences of nuclear terrorism because increasing terrorism results in growing chance to have different nuclear security events like a radiological terror attack or other possibilities for different type of radiological crime scenes. Besides, all nuclear or other radioactive materials found out of regulatory control needs investigation behind in order to find linkages between the material and people, places and events. That is nuclear forensics is a crucial tool of a state's Nuclear Security Infrastructure in order to support the investigation [1].

The safe and secure collection of these materials at a crime scene, together with ensuring chain of custody is the area of radiological crime scene management (RCSM) [2] which should be supported by nuclear forensics through the in-field categorization of the material to plan the safe and secure transport, to inform the receiving laboratory and to ensure chain of custody also during the nuclear forensic examination. RCSM is a relatively new and emerging area in all over the world, only a few states have developed operating procedure for RCSM.

In Hungary, a Nuclear Security Working Group has been established in 2016 with agreement and participation of all the relevant competent authorities as the Hungarian Regulatory Body, Hungarian Police, Counter Terrorism Centre, Disaster Management, Bomb Squad, Traditional Forensics Institute, Defense Forces, Health Services and others. At that time Hungary committed to establish its national Nuclear Security Response Framework and the RCSM procedure. In the frame of a national project funded by the Hungarian Ministry of Interior, the RCSM procedure was developed in cooperation of the Hungarian Police Criminal Forensics Department and the Centre for Energy Research, Nuclear Forensics Laboratory. 400 investigators were trained for the RCSM procedure in Hungary, in 2019 and this training was integrated into the crime scene investigation educational program of the Hungarian Police Academy. Besides, the procedure was published [3] in English in 2020 and successfully demonstrated at the International Conference on Nuclear Security of the IAEA in February 2020 (ICONS2020) and at the 65th General Conference of the IAEA in September 2021 in Vienna.

The Hungarian RCSM procedure has some unique characteristics, law enforcement and scientists cooperating work at the scene (strong scientific support by nuclear forensic experts and nuclear physicists), a special dressing procedure and contamination control. During the development of the operating procedure, Hungary has also established close connection between RCSM and nuclear forensics through HPGe in-field categorization of the material collected at the scene.

This work will present the Hungarian Operating Procedure for Radiological Crime Scene Management, some real cases as case studies, its successful demonstration at international events, as well as its proper application during the 7th Collaborative Material Exercise, organized by the Nuclear Forensics International Technical Working Group (ITWG), „Crime-Scene-in-the-box”.

Poster Session 2 / 44

## **Nuclear Forensic Characterization of Uranium Ore Concentrates by Fourier-transformation Infrared Spectroscopy**

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Fourier-transformation Infrared Spectroscopy (FTIR) is an analytical method used to determine the infrared spectrum of the absorption, emission, photoconductive or Raman-scattering of solid, liquid or gaseous substances. Therefore FTIR allows the determination of the chemical composition or the molecular structure of substances, therefore it can be considered as a suitable technique for origin assessment of different type of nuclear materials.

The FTIR technique has been known in the field of nuclear forensics for many years, however it is not widely used in advanced nuclear forensic examination as compared with more advantages and more precise data obtained by X-Ray Diffraction measurements. On the other hand, it offers a good alternative to expensive largescale equipment as a cheap, fast and simple technique for less developed countries with limited capacity of analytical techniques used for nuclear forensics.

In our measurements, several uranium ore concentrate samples were analyzed with various but known origin. The aim of this study was to identify the uranium molecular components of the samples as well as other possible technological impurities, that could collectively provide information on the manufacturing method, i.e. the origin of the material.

After comparing the infrared spectra of the different samples partly with the same, partly with different origin, it was obtained that even for samples with the same chemical composition, it was possible to identify differences between the uranium ore concentrate samples. That is why the technique provides a reliable solution for the qualitative identification of uranium ore concentrates and a simple but effective tool for origin assessment.

#### Oral Session #5 – Analytical Methods for Analysing Radiological and Nuclear Evidence / 45

### A novel methodology for Nuclear Forensic Examination: Positron Annihilation Spectroscopy

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The Positron Annihilation Spectroscopy (PAS) is a well known and widely used analytical technique in the material science. PAS is typically used to measure structure defects in the materials, e.g. to determine material defects in various metal alloys or the molecular structure of medicines. This is also why it is used in nuclear industry to study the material structure of irradiated metal alloys and many other types of materials, where it is necessary. The use of PAS for the measurements of nuclear materials is partially limited. It is because the measurements of nuclear materials by PAS is primarily dependent on the radioactivity of the test material, as its magnitude increases the background of the PAS spectrum. This impairs the detection limit of the instrument.

In nuclear forensic examination, PAS has not been used as an analytical equipment before. However, since the data provided by PAS are based on molecular structure, it may be suitable for origin assessment for nuclear materials.

In our research, we are looking for answers to decide, whether PAS is suitable for measuring nuclear materials for origin assessment, or not. Can it be useful in nuclear forensic measurements as a novel alternative instrument, or not? Therefore, various uranium ore concentrate and uranium oxide samples of known origin were examined by PAS. Based on the obtained results, the study of electron momentum distributions by Doppler-broadening spectroscopy has not revealed significant differences between the samples. Nevertheless, uranium oxide and the uranium ore concentrate samples could be distinguished easily and, also, positron lifetime data made differences even inside a group of samples.

Based on our results we can conclude that the PAS is a suitable instrument for performing nuclear forensic measurements.

**Oral Session #2 – Capability Development and Sustainability / 46**

## **Role of International Recommendations in Development and Maintenance of Stable Nuclear Forensics Capabilities**

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### Description

This paper describes the main results of work on the elaboration of certain rules for the development of international guidance documents in the field of nuclear forensics and radiological crime scene management. The recommendations for the fields of application, the consultancy team formation as well as the content of these documents are defined.

International nuclear forensics documents can play a significant role in helping countries to develop and maintain its stable national nuclear forensics and radiological crime scene management capabilities. They gather experience gained by different countries in solving problems related to setting analytical tasks and analyzing nuclear or other radioactive samples or samples contaminated with trace amounts of radionuclides.

International recommendations for the development and maintenance of stable nuclear forensic and radiological crime scene management capabilities may have two main directions:

- Proposals for the organization and improvement of the national system for identification of Nuclear or other Radioactive Material found Outside the Regulatory Control (MORC);
- Recommendations for collection and analyzes of samples in the context of a criminal investigation where nuclear forensic analysis are required.

Proposals for the improvement of the national system should be developed by a team of specialists in different fields and from different countries. The team of authors should include representatives of law enforcement agencies and judicial authorities: experts in the field of legislation, practitioners conducting criminal investigations. Moreover, both kinds of analysts should also be represented: experts in the field of nuclear forensic examinations as well as experts in traditional forensic techniques. If the document under development includes practical recommendations on interactions with judicial authorities or court of law, then experts from countries with different judicial systems and with different rules and traditions of investigative agencies should be present during the consultancy meetings. If the recommendations contain proposals for structural changes in existing national services and organizations, they should be discussed with representatives of such services and organizations from different countries. Such proposals should have clear purposes and contain only carefully verified definitions, basic concepts and approaches. These definitions, concepts and approaches must comply with the definitions, basic concepts and approaches developed and adopted in other areas of forensic science, and should not contradict the culture of the forensic community. It is highly appreciated that state decision makers take part in the development of these recommendations. Their experience makes it possible to evaluate the applicability of the proposed recommendations, estimate their viability and, if necessary, adjust the recommendations.

Concretization of general concepts, such as international cooperation, interdepartmental interaction and others, is necessary. Such concretization allows the persons, studying these recommendations, to understand what aspects of these concepts are being discussed and to recognize the resources which are necessary for performing these components of the recommendations.

One of the successful examples of the document being developed in the fields of nuclear forensics in the international format is a document helping countries perform self-assessment of their own capabilities in this field (GICNT Self-Assessment Tool).

Recommendations for performing forensic examination of physical evidence can be useful only if they are developed by analysts who have experience in performing such examinations. Herewith, it is not necessary in these recommendations to provide detailed information about all the possibilities of the methods and on the general rules for processing the measurement results. Such information can be gleaned from textbooks and other special literature. Nevertheless, an overview of commonly applied analytical techniques which proved their efficiency within laboratory inter-comparison exercises dedicated to nuclear forensics and radiological crime scene (e.g. Collaborative Material Exercise

series or Round Robin Exercises) are needed. Such overview can be helpful for Member States which start developing their analytical capabilities using the risk informed approach.

In such recommendations it is important to focus on the informativeness and features of the analysis, which are due to the physical specificity of the samples, which can be held as evidence in the investigation, as well as due to juridical status of these samples. Examples of such recommendations are documents developed by nuclear forensics international technical working group (ITWG).

#### Oral Session #5 – Analytical Methods for Analysing Radiological and Nuclear Evidence / 47

### Origin assessment of sealed neutron sources with X-ray radiography, a new method for nuclear forensic investigations

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In the recent years sealed radioactive sources are getting more interest from nuclear forensics point of view, because these materials, unlike the more rigorously guarded nuclear materials, are relatively easier to access due to their use in the industry or in the medical care system. As well-known, nuclear forensic science uses destructive and non-destructive methods for the analysis of an unknown material. In the case of sealed sources, for the characterization, non-destructive analysis is the simplest way, i.e., there is no need to open the capsule. X-ray radiography is a widely used technique in the medical field and also in industry, and it gives a non-destructive alternative for the nuclear forensic analysis of different kind of radioactive materials.

Prior to X-ray radiography, the sources were characterized physically, visible-light photos were made, and the main physical characteristics and properties were described. An X-ray radiography method was developed and used to characterize 11 (7 pieces of <sup>252</sup>Cf and 4 pieces of <sup>244</sup>Cm) sealed neutron sources. The 2D projections were acquired at the X-ray facility of the bimodal imaging station of the Budapest Neutron Centre, RAD. We operated the portable X-ray tube (ERESCO 42 MF3) at 200 kV voltage with a 0.6 mm Sn filter to produce a more energetic beam, which was necessary to penetrate the sealing more effectively. The spatial resolution was around 0.2 mm.

For the comparison we investigated sealed sources of Russian and US origin (Figure 1.).

Figure 1 - Sealed Cf-source from USA

Through our investigations we found clear similarities but significant differences among sources from the same and the different manufacturers, respectively. It means difference in the encapsulation of the sources, the size, homogeneity, and the shape of the source material. Therefore, these methods can clearly help to identify the sources from unknown origin, and hence, their manufacturer and owner, or at least it is possible to identify the country or territory where the source has been made. It will allow authorities to review the licensees' physical protection system. To identify the sources, additional parameters should also be investigated such as X-ray fluorescence properties and gamma-ray emissions.

This research presents a novel non-destructive methodology to analyse sealed radioactive sources and contributes to the expansion of the nuclear forensic library in Hungary.

keywords: sealed sources, X-ray radiography, nuclear forensics

#### Oral Session #6 – Novel Techniques Applied to Nuclear Forensic Examinations / 48

### Application of Artificial Intelligence in Nuclear Forensics: Findings of a Dedicated Workshop

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### Introduction

Whenever nuclear or other radioactive material is found out of regulatory control, the investigating authority needs to identify the radiological hazard associated with the material, the potential origin of the material and the route it has taken after regulatory control was lost. In order to answer these questions, (samples of) the seized material may be subject to nuclear forensic analysis. Characteristic parameters, colloquially referred to as “nuclear forensic signatures”, are measured; such as the isotopic composition, elemental composition, molecular structure, morphology, decay products or trace elements.

Data interpretation is typically achieved by comparative evaluation (e.g. classification) using data obtained from samples of known processing history and origin (e.g. from national nuclear forensic library). Statistical techniques and subject matter expertise are traditionally used to infer processing history and potential origin of the material.

The application of Artificial Intelligence (AI) tools, however, may offer new avenues in data evaluation in nuclear forensics.

### Present Work

The paper describes the outcome of a dedicated workshop, which was co-organized by the European Commission Joint Research Centre (JRC) and the Norwegian University of Life Sciences (NMBU). The workshop brought together experts from two different communities, which haven't been exposed, to each other very much: nuclear forensic scientists and experts in the field of Artificial Intelligence. The event was sponsored through the JRC's Enlargement and Integration program and gathered relevant experts from EU Member States, from Norway, Ukraine, Turkey and Japan, affiliated with universities or research organizations. The workshop aimed at transferring knowledge from fields such as medical image analysis or food analysis in particular with a view to authentication, compliance verification and classification.

The key challenges with data in nuclear forensics arise from small data-sets, inhomogeneous (aggregated) data, incomplete data-sets, sensitivity/confidentiality of data, transparency of conclusions and confidence in conclusions. The workshop addressed these challenges by presenting and examining areas such as explainable AI, feature selection, predictive AI or multi-block data. Examples were provided where AI methodologies such as machine learning or deep learning are applied for analyzing data originating from simple measurements (isotope ratios, concentrations), from recording spectra (infra-red, Raman, gamma ray), from images (photography, optical microscopy, electron microscopy) or from complex analytical techniques (hyperspectral images, tomography).

Through the round table discussion a clearer picture of a path forward could be established, which was captured in five recommendations:

- Promote the use of distributed learning models to enable handling of sensitive data
- Establish an interface between subject matter experts and feature/parameter selection (by AI) to increase transparency of conclusions
- Develop multi-block data analysis for comprehensive description of nuclear material using information from various measurement techniques
- Examine the application of transfer learning
- Investigate the usefulness of synthetic data for training, re-training and validating models

The findings of the workshop will inspire the work program of the nuclear forensic community and serve as basis for multi-lateral cooperation. As the discussions have shown, the area of nuclear forensics can significantly benefit from applying AI methodologies.

Poster Session 2 / 49

## Nuclear Smuggling Detection and Deterrence Radiochronometry:

## Past, Present, and Future - Review of Radiochronometry Collaborations and Challenges

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### Technical Summary

For nearly two decades, the U.S. Department of Energy/National Nuclear Security Administration (DOE/NNSA) has sponsored nuclear forensics collaboration between the United States and partner countries in the field of radiochronometry – the science of age dating nuclear material. This collaborative research is currently managed by the DOE/NNSA Office of Nuclear Smuggling Detection and Deterrence. Partner countries have included: Armenia, Australia, Canada, China, European Union, France, Israel, Japan, Republic of Korea, Romania, and the United Kingdom, and new projects are under development with Kazakhstan and Ukraine. The goal of these partnerships has been to advance radiochronometry capabilities throughout the global forensic community in order to support the investigation of nuclear material found out of regulatory control.

Significant contributions to the forensic community have been achieved through international collaboration and will be summarized in this presentation. These include: publication of radiochemistry and mass spectrometry methods for radiochronometry; the generation and publication of measured <sup>230</sup>Th/<sup>234</sup>U and <sup>231</sup>Pa/<sup>235</sup>U model ages for uranium certified reference materials; publication of consensus model ages for plutonium certified reference materials; radiometric (alpha and gamma counting) measurements of model ages; multi-instrument radiochronometry studies; and the development of new reference materials to support radiochronometric analyses.

The field of radiochronometry has evolved over time and we will summarize how international partnerships have evolved concurrently. New chronometers have been developed to support age dating (e.g. <sup>231</sup>Pa/<sup>235</sup>U, <sup>226</sup>Ra/<sup>230</sup>Th, <sup>227</sup>Ac/<sup>231</sup>Pa) and discordance between new chronometers is now being used to understand how nuclides used for age dating behave during complex fuel cycle stages, such as uranium metal production and UF<sub>6</sub> storage. Through partnership and discussions between international subject matter experts, we have identified challenges specific to radiochronometry and the uranium and plutonium production cycles. These challenges will be discussed to encourage future partnership that will support the application of radiochronometry during nuclear forensic investigations. To summarize, this presentation is a review of international collaboration in the field of radiochronometry that discusses where we came from, what we have accomplished, and addresses where we need to go.

Poster Session 1 / 50

## Investigation of sealed Cm-244 neutron sources from nuclear forensics aspect using non-destructive methods

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Eight Cm-244 sealed radioactive sources (example: Figure 1.) with unknown origin were investigated by non-destructive methods from nuclear forensics aspect including physical characterization, handheld X-ray fluorescence (XRF), high resolution gamma-ray spectrometry (HRGS) and X-ray radiography. The goal of the research was to carry out origin assessment, to find key parameters and to test the relevant evaluation methods in case of the forensic investigation of Cm-244 sealed sources.

During the physical characterization procedure, pictures were taken with conventional camera (forensic photography) and optical microscope to analyze the surface and the morphology of the sealed sources. The dose rates were measured from 0.1 m and 1.0 m distances. Physical characterization is a routine procedure in nuclear forensics which is relatively quick and can provide essential information for the investigation, e.g. serial number, isotope and activity by observing the capsule (Figure 1.); and method of application from the structure (see beryllium windowing in Figure 2.a).

Figure 1: 0723LM Cm-244 sealed source under optical microscope

Figure 2: 0723LM Cm-244 sealed source

The main chemical composition of the capsules was determined by using a handheld XRF device. The analysis showed that the material of the encapsulations (Ni-Cu alloys) in the case of six out of eight sealed sources were very similar to each other, which may refer to a common production technology and/or the method of use. The other two sealed sources were Cu-Zn alloys.

In the measured gamma-ray energy spectra, the decay products and some prominent fission products of the present curium isotopes (Cm-243, Cm-244, Cm-245) were identified. Furthermore, age dating method was successfully tested – which was originally developed for Cf-252 [Apostol et. al., 2019]. This age calculation is based on the evaluation of the Cs-137 and I-132 fission product photopeaks (661.7 and 667.7 keV, respectively). The results were well acceptable which means the deviations of the values were within 1 year in the case of 25-35 years old sources. In addition, the absolute activities of Cm-244 sources were estimated (it was assumed that the radioactive sources could have been considered as point sources) and they were compared with the “computed” activities derived from the initial activities on the certificates (the deviations from the “computed” activities were on average 20%) and the activity ratios of some detected curium and neptunium isotopes (Cm-243/Cm-244, Np-239/Cm-243, and Np-239/Cm-244) were calculated. The research also aimed to search for signs of any unusual contamination which could be linked to the manufacturing process. In the case of two samples the 333.4 keV and 388.2 keV gamma energy peaks of Cf-249 were found.

Some X-ray radiographic pictures of the sealed curium sources were also taken, based on which the presence of tungsten shielding in sealed radiation sources could be confirmed.

This research presents results about the combination of non-destructive techniques used to analyse sealed neutron sources, which contributes to the expansion of the nuclear forensic library in Hungary.

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### Oral Session #7 – Signature Research on Isotopic Signatures and Age-Dating / 51

## LG-SIMS oxygen isotope and impurity distribution characterization of U-O-bearing particles as signatures of process history

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**Abstract:** Oxygen isotopes and impurities may provide nuclear forensic signatures related to processing of uranium materials. These signatures can be evaluated by secondary ion mass spectrometry (SIMS). Here we describe such SIMS characterization of U-O-bearing materials and discuss the need for reference materials to understand signatures from real materials.

**SIMS oxygen isotope characterization:** The various chemical reactions for converting uranium ore concentrates (UOCs) to desired species (e.g. UO<sub>2</sub>, U<sub>3</sub>O<sub>8</sub>, etc.) can fractionate oxygen isotopes in potentially distinguishable ways. Oxygen from the local environment may also impart their isotope signatures during processing. Bulk analysis of U-O-bearing materials can provide <sup>18</sup>O/<sup>16</sup>O ratios with a high-level of precision for evaluating process histories. SIMS can complement bulk oxygen isotope data by providing additional signatures related to isotope homogeneity at micron-level scales (e.g. particles from swipes, small fragments of fuel pellets). The isotope homogeneity or heterogeneity of a material may be related to (1) equilibrium kinetics specific to processing reactions and/or (2) the presence or lack of multiple endmember oxygen isotope sources. Figure 1 shows example SIMS particle data from New Brunswick Laboratory certified reference material (CRM) 129-A (U<sub>3</sub>O<sub>8</sub>) and two UOCs, one consisting of U<sub>3</sub>O<sub>8</sub> and one consisting of uranyl peroxide (we note the instrument was calibrated to produce a mean  $\delta^{18}\text{O}$  value of 0‰ for CRM 129-A). Here, the two U<sub>3</sub>O<sub>8</sub> samples have resolvable mean  $\delta^{18}\text{O}$  values, and the uranyl peroxide sample has a lower particle-to-particle variability when compared to the U<sub>3</sub>O<sub>8</sub> sample datasets. These characteristics likely reflect the specific process histories of each material.

**Mapping impurity distributions by SIMS:** Distributions of impurities within U-O-bearing materials may be related to process history, and can be revealed through SIMS isotope mapping. Figure 2a shows an example of O, F, Cl, and U isotope signals from UO<sub>3</sub> particles (sample 1). In this case, the F and Cl impurities are co-located with the uranium and oxygen signals from the particles. Figure 2b shows a three isotope plot (<sup>19</sup>F/<sup>16</sup>O vs. <sup>35</sup>Cl/<sup>16</sup>O) of (1) sample 1 UO<sub>3</sub> particle data (black data; left side of the plot); and (2) particle data from a second sample consisting of U<sub>3</sub>O<sub>8</sub> material (maroon and green data; right side of the plot). Whereas the UO<sub>3</sub> data are tightly clustered, indicating co-location of impurities with the U-O-bearing particles, the U<sub>3</sub>O<sub>8</sub> material shows three clusters of data, including a high F residue and a high Cl residue. These impurity distribution differences are likely due low temperature processing that didn't separate F and Cl from U (e.g. the UO<sub>3</sub> sample) compared to high-temperature sintering that separated U, F, and Cl (e.g. the U<sub>3</sub>O<sub>8</sub> sample).

**The need for reference materials:** In order to interpret oxygen isotope and impurity distributions of real materials, it is vital to have reference materials with known process histories that collectively represent a meaningful parameter space of material processing. In part, this can be achieved through laboratory synthesis of materials through various chemical reactions and conditions. Subsequent characterization of these materials can then trace oxygen isotope ratios and impurity distributions back to how the material was synthesized, to better understand signatures of real materials.

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**Oral Session #6 – Novel Techniques Applied to Nuclear Forensic Examinations / 52**

## **APPLICATION OF ALPHA-AUTORADIOGRAPHY FOR DETECTION AND IDENTIFICATION OF ALPHA-EMITTING CONTAMINATION IN NUCLEAR FORENSICS EXAMINATION**

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### Introduction

The objects of investigation in nuclear forensics may be represented as soil samples, vegetation, tissue fragments, filter materials, biological samples, everyday objects contaminated with particles of nuclear and other radioactive materials (NRM). The analysis of such objects, during the investigation, first of all, requires the use of non-destructive methods of analysis. Sources of  $\alpha$ -radiation located at these objects can have low radioactivity, and it is practically impossible to register them using radiometric equipment. Usually, the carriers of these particles are materials and objects that have a developed relief and low electrical conductivity or have an extended geometry. Because of these features, as well as because of the strong gas release, their microanalysis using devices based on the use of focused electron and ion beams is difficult.

In this regard, for the detection of point sources of  $\alpha$ -radiation, an alpha-autoradiographic study may be effective. This method allows not only to localize the sources of alpha radiation but also to draw a preliminary conclusion about their isotopic composition, without affecting the integrity of the research object. This study estimates the informativity of alpha-autoradiography method for analysis of biological samples, tissue fragments, filter materials and soil samples contaminated with NRM particles.

### Experimental part

A study had started with a model experiment to obtain the dependence of the geometric parameters of the tracks on the energy of the alpha radiation source. This work describes the process of preparing samples and instruments required before exposure, as well as the process of transferring localized particles to a substrate suitable for instrumental analysis by probe methods.

After 70 days of a track detector exposure over a biological sample, tracks from six point sources of alpha radiation were found (Fig. 1). Geometric parameters of these tracks correspond to alpha-emitting energy of uranium. The presence of uranium was confirmed by Scanning Electron Microscopy with Energy Dispersive X-Ray Analysis (SEM-EDX).

In the soil samples found track clusters correspond to plutonium particles. In this case, the exposure time required to localize plutonium particles is significantly less than that for uranium particles. It is possible to localize a particle of a hundred nanometers within 96 hours and 2 hours will be enough for micron-sized particles (Fig. 2). The image of a plutonium particle (1  $\mu\text{m}$ ) is shown in Fig. 3.

### Conclusions

As a result of the study of biological samples, tissue fragments, filter materials and soil samples contaminated with NRM particles, a preliminary conclusion about the presence of uranium and plutonium in these particles was made. Moreover, alpha-emitting particles were localized on objects that were not suitable for analysis by probe methods. The high sensitivity of the method allows localizing uranium microparticles of hundreds of nanometers.

## Poster Session 4 / 53

# ANALYSIS OF BIOLOGICAL SAMPLES CONTAMINATED BY NRM FOR NUCLEAR FORENSICS EXAMINATION

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### Introduction

Objects of nuclear forensics investigation usually are sources of ionizing radiation (IR), environmental samples from the crime scene and other objects contaminated with nuclear and other radioactive materials (NRM). Nevertheless, people and other living organisms may become witnesses or victims of an incident associated with NRM. In this case, biological samples contaminated with NRM become another type of object for investigation.

Studies related to the analysis of such samples, due to their uniqueness, are rare and not widespread. This paper describes the study of biological samples of various types and origins, contaminated with

NMR and their imitators, using a wide range of analytical methods: Image Plate, alpha autoradiography, scanning electron microscopy (SEM).

#### Experiment

For the analysis of biological samples, first of all, non-destructive and low-destructive methods of analysis are used, since the objects of study contain small amounts of material, often just several microparticles. Probe methods of analysis usually give a large amount of information about the material (morphology, elemental composition, isotopic composition). In most cases, this information is sufficient to answer the questions of the investigation about the nature and origin of the material. In this work, the objects of study were samples of the internal organs of mice, as well as the lungs of humans and mice. Lungs samples are thin waxed sections transferred on a glass slide or carbon disk. The research has shown that the Image Plate is a fast and non-destructive method for detecting distributed sources (Fig. 1). At the same time, the most effective method for detecting point sources of alpha radiation is alpha autoradiography. Using this method, with a long exposure time, six sources of alpha radiation in the sample were found (Fig. 2). Further, these point sources were investigated by probe methods. A preliminary conclusion about uranium content in the particles was made based on the results obtained by alpha-autoradiography.

Unlike alpha autoradiography, probe methods require preliminary preparation of the sample for analysis. One way to prepare a waxed section of a biological sample is by sputtering a thin gold layer (20-30 nm) on it. After this preparation, particles detected by alpha autoradiography were found and analyzed by probe method. These particles included uranium and oxygen. The particles have a size of about hundreds of nanometers, and they have undefined shapes.

#### Conclusion

The study of biological samples contaminated with NRM requires an integrated approach using various methods of analysis and sample preparation techniques. The results obtained during the study of biological objects are highly informative for nuclear forensics. Therefore, its study is important not only for answering investigative questions related to the origin of NRM but also for answering questions related to the effect of the discovered material on humans and other living organisms exposed to it.

### Oral Session #4 – International Cooperation and Exercises / 54

## EXPERIENCE IN THE APPLICATION OF FORENSIC EXAMINATION TECHNIQUES IN THE IAEA REGIONAL EXERCISES ON NUCLEAR FORENSICS

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#### Introduction

The need for joint investigations of seized radioactive samples by both NRM specialists and forensic experts, who study these samples using traditional forensic techniques is no longer in doubt. However, the most significant steps in identifying the problems arising from the interaction of specialists from different scientific schools and different departments, in finding ways to solve these problems, can be made in the process and during recognizing the results of the exercises. This understanding has been reflected in exercises organized by the International Technical Working Group on Nuclear Forensics [1], as well as in the recent IAEA regional nuclear forensics exercises for the CIS and Eastern Europe countries. It should also be noted that the need for coordinated actions by these specialists, as well as investigators, arises before the start of samples analyses in laboratories. It arises at the crime scene. Therefore the main goal of the exercise in 2021 was to develop the interaction of investigators, forensic experts and NRM specialists at different stages of a crime investigation.

#### The specifics of the exercises

The exercises organized by ITWG and the IAEA regional exercises have very different implementations. The first includes familiarization with the scenario of the incident and the subsequent analysis of samples sent by the organizers in different national laboratories. Duration of analysis is two

months. Regional exercises are held for a week and include a simulation of an incident with NRM dispersion, which differs from the real one in that instead of real radiological dispersible devices (RDDs), their mock-ups are used, in which NRM are replaced by their non-radioactive simulators. And the participants in the exercises perform the roles of both: members of the investigative and operational teams, taking items and environmental samples at the scene of incidents, and participants of expert groups, taking part in the analysis of some of the items and samples and answering questions of the “investigative authorities”. The limited exercise time necessitates some simplification of the items and samples analysis procedure, however, the used research methods must fully ensure the logic of the incident investigation.

#### **Methods and results of investigation**

Lead powder as well as powder of tungsten with altered isotopic composition, imitating NMR powders, and a solution of non-radioactive cesium, imitating a solution of cesium-137, were dispersed over certain areas in the conducted exercises. Traces of the same powders contaminated some items in the room that played the role of a workshop in which RDDs were manufactured. The following analytical methods have been used in laboratory studies: inductively coupled plasma mass spectrometry (ICP-MS), scanning electron microscopy (SEM) in combination with energy dispersive X-ray microanalysis (EDX), and secondary ion mass spectrometry (SIMS). These methods were used in the analysis of items and samples taken at the site of dispersion of the NRM simulators and in the workshop. In addition to physical and chemical analysis, the exercise included traditional forensic fingerprints research. These fingerprints were left on one of the items found in the area of cesium dispersion, as well as on one of the items in the workshop.

The variety of objects at the incident scene and diversity of the research methods made it necessary the interaction of different specialists and determination of the sequence of research techniques – the study of some evidence on the surface of an object can damage or destroy other evidence. The exercises showed:

- Analysis of environmental samples by ICP-MS revealed the area covered by sputtered cesium;
- Studies of the morphology and elemental composition of powder particles by the SEM-EDX techniques as well as measuring of isotopic composition of tungsten in particles by SIMS methods showed the identity of the powders collected at the site of their dispersion and in the workshop;
- Investigations of the fingerprints left on the drone, from which the cesium was sprayed, and on the objects in the workshop, showed that they were left by the same person.

#### **Conclusions**

The paper shows that the analysis of objects, collected during the exercise, using ICP-MS, SEM-EDX and SIMS, as well as the study of fingerprints left on the objects, allow to achieve the goal of the exercise – practicing joint actions of the investigation participants.

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## **Oral Session #4 – International Cooperation and Exercises / 55**

### **Spectral Flavor of the Month: Spectroscopic challenges for technical experts**

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The science of spectrometry is well established but the method of doing the actual analysis can be varied based on instrumentation available and software used. This skill set is key across a range of applications in nuclear security – from an initial detection through material characterization. The Department of Energy’s National Nuclear Security Administration’s Office of Nuclear Smuggling Detection and Deterrence has developed a monthly round robin gamma spectrometry drill to hone and sustain the gamma spectrometry skills of their partner countries based only on their analytical skills. This is done by providing participants with a scenario, relevant spectra, and ancillary information and asking participants to provide responses to investigative questions and questions that

focus on response efforts rather than giving analytical answers using multivariate techniques. The responses require justification for the answers the participants derive, which often lean on complex physics principles and may provide for instructive discussions internal to the teams. The drills are developed to be agnostic of any software requirements so the teams can use the tools available to them. Topics covered have ranged from Naturally Occurring Radioactive Material to medical to industrial sources, and are drawn from real-world experiences or observations. Drills have been submitted from participating teams as well as US organizers, encouraging discussion of shared experiences and increasing participant interaction. By continuing to practice this skill set, participants' (and organizers') confidence in their spectrometry and analysis methods can be increased and new methods can be learned and applied. Partners also retain the previous drills for internal use and potentially as a tool for new hires. This type of round robin spectrometry on this scale is a new way of exercising spectrometry capabilities simultaneously on a multilateral level. Without the need for on-site or in-person activities, it has been a successful means of engaging partners in a virtual setting while travel restrictions are in place. This paper discusses the methods used as part of the Spectral Flavor engagements, addresses areas of success and improvement, and methodologies for path forward in remote multilateral capability exercises.

#### Oral Session #5 – Analytical Methods for Analysing Radiological and Nuclear Evidence / 56

### 30 years Nuclear Forensic Analysis at the JRC Karlsruhe – Support provided to EU Member States and Other Partner Countries

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At the beginning of the 1990's, incidents of illicit trafficking of nuclear materials generated a serious concern: An unexpected threat appeared and effective countermeasures based on nuclear forensic capabilities had to be quickly deployed. The first seizures of smuggled radioactive material were reported in 1991 in Switzerland and in Italy. In the subsequent years numerous similar incidents were reported in Germany, Czech Republic, Hungary and other central European Countries. Apart from the need of determining the nature of the illicitly trafficked materials and their intended use, the authorities requested information about the materials' origin.

With its range of advanced chemical and isotopic analytical methods that had been established mainly for nuclear safeguards purposes, as well as using its long-standing expertise on nuclear material analysis, JRC Karlsruhe was in an excellent position to help addressing the nuclear security challenge of European citizens. Therefore, EURATOM approached the JRC Karlsruhe and the first nuclear forensic analysis was performed there in 1992. During the following three years, twenty-one investigations of materials seized in Germany were performed. The majority of those cases concerned uranium fuel pellets that were intended for use in early-generation graphite-moderated or pressurized-water nuclear power reactors. However, the most serious incidents involved kilograms of highly enriched uranium, several hundred grams of mixed uranium-plutonium oxide, and weapon-grade plutonium. In the early years, most of the investigated incidents were related to intentional unauthorized movement of nuclear or other radioactive material. More recently, the materials found out of regulatory control and subject to nuclear forensic analysis were linked to unauthorized acts such as illegal possession or unauthorized disposal of radioactive waste.

As the phenomenon of "illicit trafficking" persisted, the JRC Karlsruhe established a specific nuclear forensic research and development program in 1997. The research concentrated first on the analysis of trace elements as indicators of environmental contamination, e.g. measurements of the oxygen-18/oxygen-16 isotopic ratio in uranium oxides, to gather information on the geolocation of the production site, and surface roughness characterization of fuel pellets in connection with grinding processes. In order to narrow down the number of possible production places of nuclear materials, the "age" (i.e. the date of last chemical separation) of U and Pu is a very important factor. The age determination method for Pu was developed using multiple parent/daughter ratios by thermal ionization mass spectrometry and additionally the method was also demonstrated to be applicable for particles using Secondary Ionization Mass Spectrometry. In the beginning of 2000s, the work on age determination methods by mass and alpha spectroscopy was extended to uranium. Later, research was conducted on the propagation of metallic impurities in various uranium materials at

the front end of the nuclear fuel cycle. Similarly, rare earth elements (REE), anionic impurities, and stable isotope ratios of Pb, Sr, S and Nd have been investigated as potential signatures for the origin determination of uranium and they have been tested using a comprehensive set of uranium ore concentrate (UOC) samples from mines around the world.

In the run-up to the FIFA World Cup 2006, which was hosted in Germany, JRC jointly with German Law enforcement started developing capabilities at the interface of nuclear forensics and traditional forensics (e.g. fingerprint, DNA). In the preparation phase of the event, the issue of collecting and examining radioactively contaminated evidence, e.g. after a dirty bomb explosion, was raised. Together with the German Federal Criminal Police (BKA), a dedicated glove box was designed and set up at JRC Karlsruhe, where latent fingerprints can be developed for radioactively contaminated items following the established protocols of the police. Ongoing studies include the particles formed during a dirty-bomb detonation. Fundamental studies on formation mechanisms using laser heating devices are complemented by real explosion tests on non-radioactive surrogate materials.

The presentation will review the trends observed in the type and nature of incidents, the analytical methodologies used in the casework and highlight some of the nuclear forensic signatures that were identified as useful for the nuclear forensic investigations.

#### Oral Session #4 – International Cooperation and Exercises / 57

### Nuclear forensics teamwork consolidated around CMX exercises

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Actually, my abstract is not ready. If it's possible to add it in the next week (<17 December), that would be great. If not, forgive me for this trouble.

The contents of my presentation is about the France participation in the CMX exercises, and more especially about CMX-7. Which analysis devices are used to nuclear forensics, what organization is set up especially for CMX, what results, which teams are involved for RN material and for classical evidence, how they interact together, what is to improve / lessons learned, etc.

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### Teaching Nuclear Forensics Material Analysis – Getting Creative with Virtual Tools

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In order to help partner countries meet their nuclear security objectives, Lawrence Livermore National Laboratory (LLNL), together with Los Alamos National Laboratory (LANL), through partnership with the U.S. Department of Energy's National Nuclear Security Administration (DOE/NNSA) Office of Nuclear Smuggling Detection and Deterrence (NSDD), have assisted over twenty countries to develop, sustain, and advance indigenous nuclear forensic capabilities. This is achieved through scenario-based policy discussions, hands-on laboratory-based training sessions, and material sample exchanges.

The ongoing COVID-19 pandemic has adversely impacted the ability of nuclear forensic assistance

providers to implement hands-on capacity building and in-person scientific exchanges. To meet the challenge, LLNL and LANL developed and implemented unique virtual interaction initiatives for continued interaction with international partners. In this context, LLNL and LANL virtualized the workshop on Analytical Plan Development in Support of Investigations, which aims to provide guidance in the development and execution of an analysis plan for nuclear or other radioactive material found outside of regulatory control.

In support of NSDD, LLNL and LANL subject matter experts (SMEs) modified the workshop materials, first presented in 2015, for enhanced interactive engagement during the virtual live sessions. In advance, participants read and watch materials hosted on a password-protected website, nuclear-forensics.org, which cover analytical techniques such as gamma- and alpha-spectrometry and isotope ratio measurements. These materials range from fact sheets to narrated presentations and animations, allowing participants with diverse backgrounds to learn basic concepts at their own pace. Short quizzes using Mentimeter during the virtual training help instill important concepts.

Following the guidance of the IAEA Nuclear Security Series 2-G: Nuclear Forensics in Support of Investigations, participants were guided through a three-stage fictitious scenario exercise between lectures, where they were asked to put themselves in the roles of laboratory staff performing a nuclear forensics examination. Participants were given the opportunity to work with and interpret real nuclear forensics data derived from the Nuclear Forensics International Technical Working Group (ITWG) CMX-4 collaborative material exercise. Participants compared and contrasted the provided datasets to interpret information and assist a fictitious investigative authority in answering key questions about the materials out of regulatory control. Participants also learned how they might answer questions posed by investigating authorities in furtherance of prosecution, a key contextualizing component connecting forensic laboratory scientists with the broader investigatory process. Furthermore, participants were exposed to interpretation methods that help determine material origin, which aside from assisting the investigation directly, can help illuminate nuclear material security gaps and smuggling pathways.

By the end of the workshop, participants obtained a better understanding of how they can assist the investigatory authority by providing data in furtherance of prosecution of alleged smugglers, as well as how to determine the potential origins of nuclear or other radioactive material out of regulatory control. To this end, virtualizing NSDD's capacity building activities such as the Analytical Plan Development workshop during unprecedented worldwide travel restrictions enhances the ability of the partner country to investigate nuclear material smuggling incidents, ultimately supporting enhanced deterrence of future smuggling events.

## Oral Session #6 – Novel Techniques Applied to Nuclear Forensic Examinations / 59

### Development of a Method for Image Analysis for Nuclear Forensic Examinations

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As part of an agreement between the Japan Atomic Energy Agency (JAEA) and the U.S. Department of Energy's National Nuclear Security Administration (DOE/NNSA) Office of Nuclear Smuggling Detection and Deterrence (NSDD), the JAEA-ISCN (Integrated Support Center for Nuclear Non-proliferation and Nuclear Security) and DOE/NNSA cooperatively aim to advance technology development in nuclear forensics. The most recent project in nuclear forensics involves a benchmarking study of image analysis software used by the participating laboratories to characterize particle morphology of powder samples through scanning electron microscopy (SEM). For this study, two different software packages were applied by up to five different users, to determine the level of similarity and consistency of the results. The ability of computational software packages to perform a robust statistical analysis of SEM images is an important tool in a nuclear forensic examination

in support of investigations. This statistical analysis includes sample characteristics such as the distribution of particle size, aspect ratio, diameter, and circumference. Since these morphological parameters can be characteristic of material process history or origin, they elucidate potential signatures of the nuclear material under investigation. However, further method development is required to ensure consistency between image analysis protocols and computational software packages, as well as the interpretation of the results.

This work will describe the preliminary conclusions of this on-going project, including the optimization of the image analysis and sample preparation procedures, the development of JAEA's computational tool for the quantification of particle images, and the automated particle analyses and comparisons between the JAEA-developed image analysis tool and the Morphological Analysis for Material Attribution (MAMA) software, an export-controlled program developed at Los Alamos National Laboratory (LANL) and used by the U.S. National Laboratories. Included in this effort is the development of protocols for quantifying overall uncertainty of morphological measurements and general reporting. This was completed by sharing a set of ten SEM images collected at LANL with analysts at Lawrence Livermore National Laboratory (LLNL) and JAEA. All analysts (JAEA, LLNL, and LANL) were to complete the quantitative particle analyses independently and then results from all ten images were compiled by LANL so that the four particle characteristics listed above could be compared.

The preliminary results of this project reveal that the new JAEA software and the established U.S. DOE MAMA software return generally similar results for the evaluated particle metrics. The benchmarking study, which was also designed to compare the influence of sample preparation by different analysts, suggests SRM 1984 is a reliable and robust test material, and confirms that the two software packages are converging on generating consistent results. These results encourage future work in developing enhanced sample preparation protocols based upon the results achieved with what may be considered an ideal sample, SRM 1984. Repeating the study with less ideal samples that more closely mimic samples that may be encountered in the international forensics community would provide significant benefit to the community.

This study supports the notion that the ability to objectively describe and quantify particle samples using SEM images and image analysis software is crucial for national and international nuclear forensic evaluations. Advancing the technology development and statistical evaluation tools in this area will provide the international nuclear forensics community with increasing capabilities for determining sample origin and process history through its morphological characteristics.

## Oral Session #2 – Capability Development and Sustainability / 60

### Building and Maintaining an Enduring Nuclear Forensics Capability – UK Case study

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Nuclear forensics is defined by the International Atomic Energy Agency (IAEA) as the “examination of nuclear and other radioactive materials using analytical techniques to determine the origin and history of this material in the context of law enforcement investigations or the assessment of nuclear security vulnerabilities”. It is a complex, multi-disciplinary, multi-faceted activity that draws upon a diverse range of technical capabilities from crime scene through to the use of technically advanced analytical methods on nuclear or radiological materials. Many Member States have established nuclear forensic capabilities, and ‘Nuclear Forensic Libraries’. The UK presented at this conference in 2019 on the construct of the hub-and-spoke, virtual library model which establishes a network of knowledgeable subject matter experts from across the defence and civil nuclear sectors, with access to information and data to aid the provenance of Material Out of Regulatory Control (MORC).

Further details of this model are provided to highlight the cost-effective establishment and deployment of a robust nuclear forensic library capability, able to deliver the required, highly visible deter-

rent whilst simultaneously able to provenance nuclear and radiological materials without the need to establish dedicated teams or capabilities. Through harnessing the UK's defence and civil nuclear national laboratories, an effective and cost-effective nuclear forensics capability has been established. As national laboratories, their respective core missions provide the basis for an enduring technical community that is applying and developing its expertise in the delivery of nuclear solutions to its customers. These programmes, for example include support to addressing the challenges associated with managing the UK's nuclear legacy, supporting current operating facilities, and enabling the contribution of nuclear to achieving net zero. These, and other capabilities are directly applicable to the identification and provenance of MORC if ever needed in a nuclear forensics' context.

Core national programmes aiming to develop advanced future fuel cycles as part of the UK energy strategy, and other complementary programmes to maintain skills, ensure that the UK nuclear industry is vibrant, and attracts high calibre scientists. This provides a route to sustaining and maintaining expertise critical to an enduring nuclear forensics capability, coupled with knowledge management to ensure access to records and transfer of knowledge and experience from present SME's. Facilities utilised for the safe and secure delivery of the UK nuclear programme provide much of the infrastructure and capability to attract such individuals and deliver this approach to sustaining an enduring nuclear forensic capability.

### Oral Session #3 – Radiological Crime Scene Management / 61

## **Radiological Crime Scene Management Training provided at the European Nuclear Security Training Centre (EUSECTRA): hybrid remote-hands-on training to efficiently complement in-person trainings**

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The European Nuclear Security Training Centre (EUSECTRA) was inaugurated under this global name about than 10 years ago and is operated by the European Commission, Joint Research Centre (JRC). It, includes a large variety of capacity building and professional development activities, which span from the hands-on training for nuclear security and safeguards actors (e.g. front line officers, law enforcement and nuclear inspectors) to educational efforts in both nuclear security and safeguards. EUSECTRA aims to improve Member States' capabilities to address the threats associated with illicit incidents involving nuclear or other radioactive materials by providing hands-on training using real nuclear materials to front-line officers, their management, trainers, and other experts in the field, and in particular in the field of radiological crime scene management (RCSM). Based on the unique combination of scientific expertise, specific technical infrastructure, and the availability of a wide range of nuclear materials, EUSECTRA complements national training efforts by providing realistic scenarios with real, special nuclear material.

EUSECTRA represents a substantive enduring and sustained core activity at JRC and positions nuclear security training at the centre of its extensive nuclear counter-terrorism and nuclear non-proliferation portfolio. EUSECTRA has been providing RCSM training to a number of countries worldwide, such as recently a series of training courses behalf of DG HOME addressing law enforcement officers from national organizations of EU Member States.

The training is organised to provide participants basic and expert knowledge in radiological crime scene management. Radiological crime scene management is the process used to ensure safe, secure, effective and efficient operations at a crime scene where nuclear or other radioactive materials are known, or suspected, to be present. The training aims at bridging law enforcement procedures, radiation protection measures and nuclear measurement expertise in processing a crime scene which is (or is expected to be) associated with radioactive material. Self-protection, evidence collection, evidence management, contamination control, initial identification of the radionuclides and radiological assessment are the primary topics of this one-week-course. Therefore, awareness is built



through lectures on the different types of radiation and their detection but also on their impact on evidences. The potential threat posed by misuse (in a malevolent or terrorist act) of nuclear and radiological material out of regulatory control, put the collection and preservation of evidence in the spotlight. Realistic, scenario based and practical hands-on exercises have helped participants to understand the risks and challenges associated with working in a radiological crime scene environment and how integrate it in the standard operating procedures of police and radiation protection. The trainees lead these exercises under supervision of the trainers. Interventions at the scene are discussed prior to starting any action. Once the crime scene has been processed, the management of the radiological crime scene and the problems encountered are critically discussed with a view to improve. Focus is given to ensuring that all actions at a radiological crime scene are carried out in a way that maintain the integrity of the criminal investigation and that all relevant criminal investigative procedures are applied through effective radiological crime scene management and that participants should be able to integrate some basic protocols at national level to manage such a crime scene.

The EUSECTRA serves primarily as platform for enhancement of efficient networking and capacity building between experts from EU Member States. During provision of the course, special attention is given that participants interact amongst them in a collaborative and knowledge sharing manner. The trainings are used for exchanging good practices while recognizing that protocols and procedures might be different from country to country. Moreover, the resources available in case of radiological crime scene are not the same in each country and some collaboration might be helpful to improve or create protocols for radiological crime scene management. EUSECTRA training situations with multi-national training groups serve also to strengthen border-crossing networking among specialists and support community building.

Law enforcement experts - related to CBRN counter terrorism- from several EU MS national authorities, Europol and other international organization (such as IAEA or FBI) have joined this specialized training as trainers. Their participation has consolidated and enhanced the learning experience by sharing knowledge and skills earned during real cases. Moreover, it contributes to create a culture which encourages the generation of practical ideas and better ways of working by capturing and dispersing best practice well beyond its original purpose.

It might be worth mentioning that a few years ago, representatives of Hungary were trained in RSCSM at EUSECTRA. Benefitting from this experience, they developed their own national curriculum, cascaded the acquired knowledge, and trained over 400 police officers. This exemplary practice is shared as a success story subsequent EUSECTRA trainings.

During the course, the trainees got access to large portfolio of training materials, radiation measurement instruments and other equipment available at the training facility. The practical exercises benefit from the wide range of radioactive sources and specific nuclear materials (low enriched uranium and highly enriched uranium, reactor and weapon grade plutonium, caesium source) available at EUSECTRA.

While the core activities of the trainings are field exercise with emphasis hands-on elements, the recent pandemic has restrained physical trainings and the value of on-line and remote trainings has come to light in such tough situation. The EUSECTRA Remote Interactive eTraining was developed at soon after the start of the Covid19 crisis. It should be noted, though, are intended to complement, not to substitute physical trainings (which will remain EUSECTRA core activities). In the medium and long term, the EUSECTRA Remote Interactive eTraining sessions will be used as introductory or refresher courses.

EUSECTRA puts strong emphasis to offer training course that are as interactive as possible using all modern technical tools available and to focus on field/practical exercises involving the use of a broad range of instruments and nuclear or other radioactive materials.

Detailed description of the trainings will be presented in the scope of this paper.

**Oral Session #1 – Legal Framework and the CPPNM/A / 62**

## **Challenges in establishing and maintaining a national nuclear**

## **forensics support framework and importance of international co-operation**

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The field of nuclear forensics is recognized as an indispensable tool in the management of nuclear security events, in particular those resulting from the trafficking of nuclear materials and nuclear materials beyond regulatory control. During this decade, the agency, in cooperation with certain specialized organizations and other partners, has developed various nuclear forensics tools, including procedures and guidelines, equipment. Thus, this work and the promotion of nuclear forensics through the sharing of knowledge and experience constitute an important part of the analysis, traceability and prevention of nuclear and radiological emergencies.

Thus, nuclear forensics involves many actors including frontline responders, nuclear scientists, doctors, regulators and law enforcement and in this regard, coordination between these entities varies from country to country. Therefore, the creation and operation of the nuclear forensics framework is country-specific and its sustainability depends on the coordination and support of decision-making systems.

In this regard, a wider and more extensive involvement of the IAEA, in raising awareness and initiating the creation of nuclear forensics support centers through appropriate tools, such as TC-IAEA projects, at the levels national, regional or interregional and by facilitating international cooperation in the field. A holistic approach with the guidance, assistance and continued support of the IAEA are key elements in stimulating and expanding support for nuclear forensic analysis in most Member States.

### **Oral Session #8 – Laboratory Capabilities / 63**

## **Development of Nuclear Forensics Capabilities at CNL**

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Canadian Nuclear Laboratories (CNL) has a long history of research and development in nuclear fuels as well as expertise in handling and characterization of radioactive materials. CNL has been leveraging its knowledge base and specialized facilities to support the enhancement of Canadian nuclear forensics capabilities. Most of this work is conducted through projects within the Federal Nuclear Science and Technology (FNST) Work Plan, a program established and managed by Atomic Energy of Canada Limited to facilitate work at CNL to contribute to the Government of Canada's health, science, innovation and climate change objectives. In addition, CNL also performs work in partnership with Government of Canada departments and agencies under the auspices of the Canadian Safety and Security Program, administered by Defence Research and Development Canada's Centre for Security Science, which funds science and technology initiatives to enhance Canada's preparedness for prevention of and response to potential threats.

Nuclear forensics (NF) uses a wide range of techniques to characterize materials in the context of an interdiction and subsequent possible legal or national security investigation. Considerations are given to safety, preservation of evidence and ultimately may include a wide range of non-destructive and destructive analyses in the context of nuclear security event involving nuclear and/or radioactive material. Materials of interest may include nuclear materials as well as radioactive sources used for medical and industrial purposes. CNL's program of work on nuclear forensics focuses on several areas, including development of new methodologies, determination of signatures of nuclear materials and radioactive sources, and providing support to the national NF capability.

Signatures of Radioactive and Nuclear Materials

Signatures of radioactive and nuclear materials can provide investigators with useful information about materials such as its origin, fabrication process, use, or history. Thus, one area of work for CNL is to investigate novel signatures of radioactive sources, irradiated and unirradiated fuels, and irradiated reactor alloys.

Age dating of radioactive sources is an important component of nuclear forensics as it aids in the determination of the provenance and origin of the materials. Cesium-137 is one of the most common radionuclides used in sealed radioactive sources. For  $^{137}\text{Cs}$  radiochronometry, the separation of  $^{137}\text{Cs}$  and  $^{137}\text{Ba}$  prior to analysis of  $^{137}\text{Ba}$  is required as the stable daughter products must be measured by mass spectrometry. The manual separation techniques are advantageous for ease of set-up in hot cell facilities, however they are typically time intensive and separation efficiencies can be operator dependent. CNL evaluated a radiochronometric method for  $^{137}\text{Cs}$  sources using Ion Chromatography (IC) for the separation of barium and cesium which would allow for automation of the separations and potentially a reduction in time and operator error. The performance of the IC separation was investigated through the use of inactive standard materials and analysis performed by inductively coupled plasma – mass spectrometry (ICP-MS). The study found that while separations of cesium and barium can be achieved by IC even in complex radioactive solutions, the limitation of the method's operating concentration range is not compatible with the concentrations of  $^{137}\text{Ba}$  in the low activity (or diluted) sources that can be handled outside a hot cell. The dilutions required to enable sample handling outside of the cell often brings the Ba concentrations into the very low ppb range, in which, at these levels interferences from natural occurring Ba in the environment can become problematic. Any method developed for radiochronometry needs to be robust for various complex matrices, large concentration ranges and suitable for various installation requirements (e.g., fume hoods and hot cells).

CNL has extensive information gained from post-irradiation examination (PIE) of nuclear fuels. Signatures and trends in PIE data have been analyzed using human computed techniques by investigating correlations with physical meanings. However, it is expected that statistical methods, such as principal components analysis, can be utilized to determine additional correlations which enable attribution. The work involves scoping the best approach to applying statistical methods to fuel signatures using available measured data, not only in the PHWR industry but also in the LWR industry. The feasibility of using simulated data is being explored as well as data pre-treatment automation, to minimize necessary labour. Algorithms are being developed and applied to determine the level of confidence in attributions.

Part of the NF work at CNL is the investigation of isotopic signatures of nuclear fuel materials of interest to Canada including power reactor, research reactor and other experimental fuels. Fuel types that have been investigated previously include uranium-based and mixed-oxide fuels, and the current focus is research reactor fuel with thorium-based fuels being considered for future work. An investigation into possible signatures of unirradiated fuels has also been initiated.

CNL has experience measuring nuclide ratios to estimate the fluence of reactor alloys for supporting ageing management and life extension. These techniques are being adapted for application to nuclear forensics. The focus is on materials that may be sampled during the facility operating life (e.g., structural materials, control or absorber rods, scrape samples, etc.). Materials of potential application include aluminum, stainless steel, zirconium, and Inconel alloys. Various samples of irradiated reactor alloys from reactors NRU and NRX with a known operating history and original chemical compositions are available for analysis and method development. Simulations of their operating history will be done using reactor physics codes, and samples of these materials will be analyzed using mass spectrometry to validate the isotopic ratios of selected elements of interest obtained from the simulations. Once validated, the goal is to use these isotopic ratios to “back-calculate” the irradiation history of the material which can then be compared to the operating history of the reactor and thereby validate the methodology.

#### Novel Non-Destructive Techniques

There is a need to develop capabilities to identify the origin of undeclared nuclear materials through improved detection methods, specifically novel non-destructive techniques (NDT) and characterization methods for powders, in the context of nuclear forensics. CNL has been supporting the Canadian effort in nuclear forensics including participation in national and international exercises that have used a variety of non-destructive and destructive techniques as well as specialized knowledge such as nuclear fuel expertise. The non-destructive techniques used at CNL include a variety of instruments in surface science (e.g., SEM/EDX, TEM, XPS, Auger, and SIMS), material science (e.g.,

neutron imaging, activation, and diffraction) and radiochemical analysis (gamma spectroscopy, mass spectrometry). A number of these techniques were applied to an unknown legacy material to enable full accountancy and safe, secure disposition. Subsequent verification of the NDT results through destructive analysis and mass spectrometry supported the value of the NDT analysis. CNL is currently looking to systematically evaluate the current state of various NDTs and explore the potential of existing and emerging techniques in the context of supporting nuclear forensics. Opportunities for collaborations with Canadian institutions is also being explored to expand the techniques available to CNL for nuclear forensics applications.

Some materials of interest to NF are found in powder form, such as uranium ore concentrates, uranium dioxide powder, radioactive sources (e.g., CsCl and SrCl salts), and post-detonation debris particles. As new fuels are developed for advanced reactors, such as small modular reactors, novel characterization techniques may be required for powders (e.g., fuel salts). There are a number of techniques that can be applied, including particle size measurement and morphology characterization techniques. Collaboration with international organizations, which can be in the form of benchmarking of the different techniques, is being planned.

#### Conclusion

CNL is continuing development work on new and innovative methodologies as well as leveraging its knowledge base and specialized facilities to support the enhancement of Canadian nuclear forensics capabilities. Work is being conducted on a wide variety of materials of interest including nuclear materials and radioactive sources used for medical and industrial purposes that may be found pre- or post-dispersion. CNL's program of work on nuclear forensics focuses on several areas, including development of new methodologies as applied to radiochronometry, determination of signatures of nuclear materials and radioactive sources, and application of non-destructive techniques.

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### Oral Session #7 – Signature Research on Isotopic Signatures and Age-Dating / 64

## Signature research on isotopic composition and age dating

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Nuclear forensics is essential in response to incidents involving nuclear and other radioactive materials out of regulatory control. Scientists are expected to apply extensive analytical tools resulting with clear and credible answers, needed by decision makers. The answers are related to a large set of practical questions, like: what is the origin of the materials found out of control, what is the route by which they arrived to the scene, can the material be attributed to a specific facility, what is the "age" of the material, and more.

Most illicit trafficking incidents of nuclear materials are related to natural or low enriched uranium [1]. Several signatures of uranium ore concentrate, such as chemical composition, rare earth element pattern, strontium and neodymium isotope ratios, anionic impurities and others, provide information on the material's history [2, 3, 4, 5, 6]. These analytical techniques are well developed and were widely adopted by the nuclear forensics community. However, more analytical tools are needed to enhance and strengthen the ability to achieve origin and route attribution.

Analysis of stable isotopes is a common, well established analytical technique, applied in many scientific fields, such as geochemistry, geology, earth science, and others. Elements such as hydrogen, carbon, nitrogen, oxygen, sulfur, bromine, chlorine and lead exhibit natural isotope variability [7, 8, 9, 10], related to geographic location and/or to the production processes. Therefore, the determination

of their isotopic composition might serve as an additional significant signature, contributing to the important process of attribution.

Though being attractive to be used for attribution while conducting a nuclear forensics investigation, the implantation of stable isotopes analysis in relevant incidents, is rather small. The reasons for that will be described.

The use of various chronometers for the determination of the material's age, demands the application of chemical separation procedures, due to age shift which might occur by the presence of unwanted "daughters" in the solution. That is an important signature attracting the scientific community for ongoing improvements.

We will present recent published researches reporting on stable isotopes analysis related to nuclear forensic and model age chronometers as well.

#### Poster Session 4 / 65

## JOINT EXAMINATION OF HAIR FRAGMENTS, SPORES AND URANIUM PARTICLES USING REM-PMA

**Author:** Alina Nitrean<sup>1</sup>

<sup>1</sup> *Forensic and Legal Expertise Center, General Police Inspectorate of the Ministry of Internal Affairs, Chisinau, Republic of Moldova*

### Introduction

The increase in the number of incidents with NRM [1] and the resonance of some crimes, as well as some ambiguous results of the investigation [2, 3, 4], are the cause of increased attention for the prospect of investigating such crimes and incidents. In the 1990s and 2000s, in the investigation of incidents and crimes with NRM, all attention was focused on determining the characteristics of NRM. But for the last few years, there was a growing need to jointly investigate confiscated radioactive evidence by nuclear forensics specialists and experts in traditional forensics. This trend was also reflected in the exercises organized by the international nuclear forensic community, primarily in the exercises of the CMX series. If the previous exercises, CMX 4 and CMX 5, were focused on determining the nuclear-physical characteristics of samples [5, 6], the last CMX 6, already included the definition of traces that are the subject of traditional forensic disciplines: (fingerprint examination) and traceology [7].

A conflict of interest may arise when examining samples with both nuclear forensic evidence and traditional forensic evidence. Also, preparing the specimen for testing or examining evidence by certain methods may damage or destroy other evidence that other experts will later examine. In this context, the task is to develop the operating regime of the equipment and to establish the sequence of analysis of evidence in the forensic examination, which will allow obtaining the information useful for the investigation, as much as possible. Such tasks are successfully performed under the IAEA Residential Assignment Programs, for example, on the application of SEM-EDX methods for the purposes of nuclear forensic examination at the Laboratory for Microparticles Analysis, Moscow, Russia, 2021.

### Formulation of the problem

One of the widely used methods in forensic examinations is a combination of scanning electron microscopy and X-ray microanalysis (SEM-RMA). Using SEM-RMA, in traditional forensic examinations, the characteristics of shot products, explosion products and explosives, precious metals, various organic fibers, morphological characteristics of human and animal hair, spores and others are determined [8]. At the same time, SEM-RMA is successfully applied in the nuclear industry to study of a number of NRM characteristics. Electron microscopy makes it possible to study the morphological characteristics of NRM samples: geometric parameters of products, grain sizes, fracturing and surface porosity. X-ray microanalysis makes it possible to establish the elemental composition of a material even if only its microparticles are found at the scene of the incident [5, 6].

SEM-RMA is considered a non-destructive method. However, it is non-destructive in the case of analysis of samples traditionally studied by this method. At the same time, the effect of the electron beam on all types of traces that can be found on the samples taken from the incident site, and, first of all, having an organic nature, has not been studied. In turn, the study of traditional forensic traces, for example, fingerprints by the method of cyanoacrylate fumigation, may lead to the impossibility of subsequent determination of NRM characteristics that are essential for the investigation. Thus, examining one piece of evidence on the surface of a test sample may damage or destroy other evidence.

The purpose of this work was to prepare samples containing objects characteristic of research in a traditional forensic laboratory as well as in a laboratory investigating NRM and to determine the effect of subsequent research procedures on the safety of various types of evidence. As a result, the optimal research sequence for this type of sample should be determined and the optimal modes of operation of the electron microscope should be established.

The experimental part

To solve the problem, were prepared the samples that contained hair fragments and pollen particles (spores of various trees: pine, birch and cedar), typical for traditional forensics, as well as uranium particles - the subject of research by NRM specialists. Microparticles of uranium (for contamination) were taken from samples previously examined in the CMX-5 exercise [6] at the "Microparticle Analysis Laboratory", Moscow, Russia. All objects were deposited together on carbon substrates.

In fig. 1 shows a picture of a hair fragment (a) with a cedar spore (b) on its surface. On the surface of the spore, in turn, a particle of uranium was found. The study of this and other samples, without providing a drain of electricity from it, showed that spores and hairs are charged with an electron beam and become "invisible" to SEM. Therefore, to remove the charge, a thin, approximately 30Å, layer of gold was deposited on the surface of the samples. However, when studying even gold-deposited samples at an accelerating voltage of 20-30 kV and beam currents typical for the analysis of uranium microparticles, the surface of the hair and spores is destroyed. Therefore, the study of such samples should begin with an analysis of the morphological characteristics of hair and pollen at an accelerating voltage of 5 kV and beam currents of 10-11-10-10A. It is expedient to investigate uranium particles at an accelerating voltage of 20 kV and beam currents of about 1 nA. In this case, hair and spores are destroyed, but the necessary information about them has already been obtained.

Conclusion

It this work, it is shown that for the effective study of samples containing both hair, pollen particles, and uranium microparticles, it is necessary to spray a thin layer of conductive material on the sample. On the sprayed sample, it is necessary, first of all, to investigate the morphological characteristics of the hair and spores. These characteristics are determined at an accelerating voltage of 5 kV and beam currents of the order of tens of Picoamperes. Then it is possible to analyze uranium microparticles, as well as other NRM fragments at accelerating voltages up to 30 kV and beam currents up to units of Nanoamperes.

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Poster Session 4 / 66

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