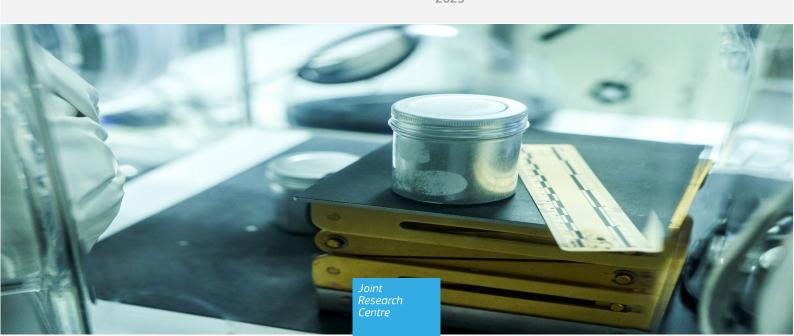


Nuclear Forensics Casework at the Joint Research Centre

Enhancing security of the European citizens

Tsiogka, V., Wallenius, M., Mayer, K.

2023



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Contents

Abstract	1
Introduction	2
1 Theoretical Background	4
1.1 Nuclear Materials	4
1.2 The Nuclear Forensic Analysis	5
1.2.1 Sample Characterization	5
1.2.2 Nuclear Forensic Interpretation	7
2 Analysed Cases and Trends	8
3 Case Examples	10
3.1 Uranium Fuel Pellets in a Garden	10
3.2 Plutonium in a Suitcase	11
3.3 Weapons-Grade Uranium in Scrap Metal	13
3.4 Illicit Trafficking of Highly Enriched Uranium around Europe	15
3.5 Gambling Using Radioactive Playing Cards	16
4 Impacts	
5 Conclusions	20
References	21
List of abbreviations and definitions	24
List of figures	25
List of tables	26

Abstract

Illicit trafficking of nuclear and other radioactive material poses a serious threat to the global safety and security. The European Commission's Joint Research Centre (JRC) provides analytical support and develops improved methods to strengthen nuclear security at the level of competent national authorities and international organizations. With its expertise in nuclear forensics, the JRC enables EU Member States and other interested parties to adequately respond to nuclear security events and make informed decisions on the threats associated with nuclear and other radioactive materials out of regulatory control.

This report aims at familiarizing decision makers and the general public with the nuclear forensics casework performed at the JRC in support to EU Member States and other partner countries. Using non-scientific language, the report presents a comprehensive overview of the scientific field of nuclear forensics, explaining its basic principles and its relevance. Next, it takes a closer look into five selected cases that the JRC investigated during the 30 years of its involvement. The report's purpose is to highlight the importance of having this capacity within the European Union, which enhances consequently the security of the European citizens.

Introduction

Illicit trafficking of nuclear and other radioactive material poses a serious threat to the safety and security of the EU Member States. While nuclear security is a national responsibility, the Joint Research Centre - Karlsruhe in Germany uses its unique nuclear infrastructure and competencies to provide analytical support and to develop improved methods for strengthening nuclear security at the level of competent national authorities and international organizations. With its expertise in nuclear forensics, the JRC enables the interested parties to make informed decisions on the threats associated with nuclear and other radioactive materials out of regulatory control.

Nuclear forensic science (short: Nuclear Forensics) assists (criminal) investigations by identifying the origin of interdicted nuclear material out of regulatory control. The International Atomic Energy Agency (IAEA) defines Nuclear Forensics as "the examination of nuclear or other radioactive material, or of evidence that is contaminated with radionuclides, in the context of legal proceedings under international or national law related to nuclear security" [1].

In practice, nuclear forensics primarily aims to answer the following questions:

- What is the origin of the interdicted material?
- Who was the last legal owner?
- Is there a link with previous cases?

The answers to these questions enable the investigative authorities to decide on their course of action: determine if there has been a breach of law and what should be the appropriate next step, if any. The JRC's role is to provide scientific evidence which helps the investigative authorities to obtain these answers. This is achieved by carrying out analytical examinations of the nuclear or other radioactive material in question. In a sense, the JRC functions as an 'expert witness' who offers their scientific opinion in the course of a possible criminal investigation.

The challenge posed by nuclear material out of regulatory control is twofold: it poses a nuclear proliferation risk and it consists a radiological hazard. The non-proliferation of nuclear materials is a primary security concern at the national and international level. It is regulated by several international treaties, which have as an ultimate goal the nuclear disarmament, meaning the reduction or elimination of nuclear weapons¹. Notable examples are the Treaty on the Non-Proliferation of Nuclear Weapons (NPT) and the Comprehensive Nuclear-Test-Ban Treaty (CTBT). Furthermore, the creation of improvised nuclear weapons might not be as destructive as that of traditional nuclear weapons, but it could lead to devastating consequences for the safety of the citizens [2]. Finally, the radiological hazard poses a serious health risk for the citizens, as well as a risk to the environment.

The JRC has been at the forefront of nuclear forensics in Europe since more than 30 years, analysing its first case in 1992. The early 1990s was the time when the collapse of the Former Soviet Union created a special window of opportunity for the development of the illicit trafficking of nuclear materials. On the one hand, there was the poorly controlled and safeguarded facilities holding nuclear material in the former Soviet States. On the other hand, there was the financial instability that made the trafficking of nuclear and other radioactive materials look like an appealing route to make effortless profit. Simply put, the availability of the material and the need for financial security, gave birth to this form of criminal activity.

Since the beginning of the JRC's nuclear forensics activities until the present time, it has dealt with over 60 cases. The first seized sample was brought to the JRC by the European Atomic Energy Community (Euratom) Safeguards Office in March 1992. Euratom's logic behind this request was that the JRC already had the infrastructure and the expertise in other areas of the nuclear fuel cycle, such as nuclear safeguards analysis or nuclear fuel development. This infrastructure and expertise could now be used to analyse unknown nuclear materials popping up in various places in Europe and trace them back to their origin.

Ever since, the JRC has kept a leading position in the field of nuclear forensics, producing pioneering knowledge and keeping pace with the current developments. More specifically, it has an important role in the scientific

¹ For an indicative list, see https://www.iaea.org/resources/treaties/iaea-related-treaties

community by its noteworthy publications, as well as its collaboration with international stakeholders. Through its active presence, the JRC contributes to the strengthening of international nuclear security.

The purpose of this report is to familiarize the interested reader in a non-scientific language with the work done by the JRC in the field of nuclear forensics, provide some real-life case examples and, finally, demonstrate its relevance for the EU citizens. The report starts with presenting a theoretical background, briefly explaining the concept of the nuclear forensics analysis. Next, it examines the current trends in nuclear forensics and in nuclear security in general, following with a closer look on five selected cases that have been investigated at the JRC. It concludes with a brief reference to the impacts of the JRC's work in nuclear forensics.

1 Theoretical Background

1.1 Nuclear Materials

Uranium is a radioactive element that occurs in nature as a heavy metal [3]. The uranium isotopes found in nature are uranium-234, uranium-235 and uranium-238. The production of nuclear fuel and nuclear weapons requires isotopically enriched uranium, which is obtained in uranium enrichment plants. Enriched uranium has a higher percentage of the isotope uranium-235 than what is naturally occurring. Table 1 provides an overview of the classification according to the IAEA:

Table 1: Classification of uranium based on relative abundance in uranium-235 isotope

Uranium type	Abundance in uranium-235
Natural Uranium (NU)	0.7%
Depleted Uranium (DU)	<0.7%
Low Enriched Uranium (LEU)	>0.7% and <20%
High Enriched Uranium (HEU)	≥20%

Source: IAEA Nuclear Safety and Security Glossary, Non-Serial Publications, INTERNATIONAL ATOMIC ENERGY AGENCY, Vienna, 2022.

Plutonium is produced by the neutron irradiation of uranium in nuclear reactors [4]. It consists of the following isotopes: plutonium-238, plutonium-239, plutonium-240, plutonium-241 and plutonium-242. The percentage of these isotopes in a given quantity of plutonium can vary significantly, depending on the reactor type that produced them and on the duration of the irradiation in the reactor. Plutonium is highly radioactive and toxic.

Nuclear materials are fissile, this means that when exposed to neutrons, these atoms can split into two fission products. This fission reaction releases large amounts of energy and 2-3 neutrons. The latter can induce further fission reaction, i.e. lead to a chain reaction. The steady and controlled fission reaction is used in nuclear reactors (i.e. for peaceful purposes) while the uncontrolled chain reaction results in a (extremely) rapid release of energy (i.e. an atomic bomb) [5]. The most common fissile materials are **uranium-235** and **plutonium-239** [6]. As pointed out above, these isotopes are essential constituents for fuel in nuclear reactors for producing electricity or for creating nuclear weapons [7].

Hence, nuclear materials are associated with a radiological hazard (due to their radioactivity) and with a proliferation risk (due to their fissile character and the dual applications). Particularly for the latter reason nuclear materials are put under scrutiny and strict regulatory control. This is based on binding international treaties: the Euratom (European Atomic Energy Community) Treaty for EU Member States [8] and the Non-Proliferation Treaty (NPT) which is applicable world-wide [9]. Compliance with these treaties is rigorously verified by the International Atomic Energy Agency (for the NPT) and by the European Commission (for the Euratom Treaty). Moreover, nuclear material is subject to strict physical protection during use, storage and transport according to the Convention on the Physical Protection of Nuclear Material (CPPNM) and its Amendment [10].

In a few instances, however, when preventive and protective measures have failed, nuclear material is encountered out of regulatory control. In such cases, the competent national authorities need to identify the origin of the intercepted material. It is exactly the role of nuclear forensics to re-establish the history of illicit nuclear material and provide hints on when, where and for what purpose the material was produced. This is achieved by exploiting information which is inherent to the material, measuring characteristic material properties.

1.2 The Nuclear Forensic Analysis

The nuclear forensic analysis comprises of the following steps: sample collection and categorization, sample characterization and interpretation of the analytical results [6]. Its ultimate goal is to provide the competent national authorities with a technical expert-opinion based on scientific evidence that will support their investigation. The JRC's expertise comes into play in the second and third stages, after the competent national authorities intercepted the material, carried out the evidence and sample collection, and performed an initial examination of the material. The JRC's role is to either complement the national capabilities, or to provide these capabilities where there is a lack. Although nuclear security is a national responsibility and not obligated by any treaty, it is advised states to have so-called core capabilities (basic instrumentation and knowledge) to perform the initial examination of intercepted radioactive materials to categorise them, i.e. what it is (e.g. uranium, plutonium or a radioactive source) and how much approximately (e.g. a few grams, kilograms or a contamination). However, often states may lack the infrastructure and – most importantly – the specific expertise for more advanced nuclear forensic analysis to answer questions about the materials' origin.

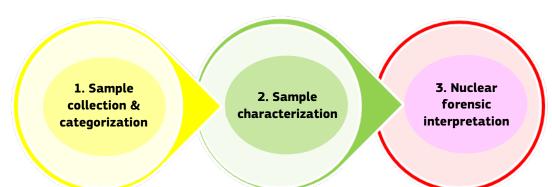


Figure 1: Flowchart of nuclear forensic analysis

Source: Adapted from Fedchenko, Vitaly. The New Nuclear Forensics: Analysis of Nuclear Materials for Security Purposes, Oxford University Press, Oxford, 2015

1.2.1 Sample Characterization

The sample characterization takes place in a nuclear laboratory. Its aim is to determine those material parameters (such as physical, chemical, elemental and isotopic properties) which allow drawing conclusions on the processing history and the potential origin of the material. These characteristic parameters are often referred to as "nuclear forensic signatures". To achieve this goal, scientists at the JRC use a wide range of analytical techniques. Ultimately, they want to extract as much information as possible out of the material, to be able to find its origin. The nuclear forensic scientists work just like the pathologist who examines the body of the victim to extract information about its cause and time of death, age, etc., in order to assist the criminal investigation. The more one knows about the material, the easier to solve the crime.

Nuclear forensic scientists at the JRC come from different backgrounds, forming a multidisciplinary team of chemists, physicists, material scientists and more. They have a wide range of sophisticated, analytical tools in their arsenal, as well as rich experience in analysing material from the nuclear fuel cycle, making the nuclear forensics laboratory at the JRC one of the leaders in this discipline². Depending on the sample, different techniques are applied, but there is usually a specific logic to be followed³.

The first step is to conduct a visual inspection (including photography) and non-destructive analysis. The use of a non-destructive technique means that the sample is kept intact, in its original form, without destroying the evidence. At this stage, the focus is on getting a general overview of the material and its characteristics. The most common technique to be used at this point is gamma spectrometry [11]. With the appropriate equipment,

5

² For a virtual tour of the laboratory see: <u>Nuclear Safeguards and Forensics Laboratory | European Commission (europa.eu)</u>

³ For an analysis scenario see: <u>Atomic detectives - YouTube</u>

the scientists can do a quick, initial 'screening' of the material and decide on the further course of action. Since every radioactive isotope emits gamma radiation at specific energies, this step is very important to i) verify the correctness of the initial categorization (typically performed in the field using portable equipment) and ii) provide more accurate information on the nature of the material (e.g. determine the level of enrichment of uranium).

Based on the findings of the non-destructive analysis subsequent steps are defined. These may include optical microscopy, measurement of physical dimensions and microanalysis tools. The imaging techniques allow to essentially zoom in the samples and examine the samples under high magnification. This way, the microstructure of the sample can be assessed. In addition, these techniques can help with determining if a sample is homogeneous or heterogeneous, meaning whether it has different components.

In the latter case, the JRC can analyse individual particles independently using very powerful tools that can yield information on the constituents of a material mixture. Some of the analytical tools that are commonly used by the JRC are briefly presented in Table 2.

Lastly, a very important thing to keep in mind is that while carrying out the nuclear forensic analysis, scientists need to maintain the chain of custody [1]. This means that they need to track and document the handling of the material in every step. They need to work under strict controls and follow established protocols. Since the results of the analysis will be likely used in legal proceedings, it is crucial to assure that they can be trusted.

Table 2: Analytical techniques commonly used by the JRC for the nuclear forensic analysis

Technique	Туре	Use
HRGS (High Resolution Gamma Spectrometry)	Non-destructive	Isotopic composition Quantification of present radioisotopes
TIMS (Thermal Ionization Mass Spectrometry)	Destructive (chemical sample preparation required)	Isotopic composition (of U or Pu)
SIMS (Secondary Ion Mass Spectrometry)	Particle analysis	Isotopic mapping Isotopic composition of individual particles
ICP-MS (Inductively Coupled Plasma Mass Spectrometry)	Destructive (dissolution and dilution required) Chemical analysis	Trace element concentrations Isotopic composition
LA-MC-ICP-MS (Laser Ablation Multi- Collector Inductively Coupled Plasma Mass Spectrometry)	Microanalysis Spatial analysis	Isotopic composition Trace element analysis
SEM (Scanning Electron Microscopy) coupled with EDX (Energy Dispersive X- ray analysis)	lmaging	Microstructure Elemental mapping

Source: Adapted from IAEA, Nuclear Forensics in Support of Investigations, Vol. 2-G (Rev. 1) of Implementing Guides, INTERNATIONAL ATOMIC ENERGY AGENCY, Vienna, 2015 & Fedchenko, Vitaly., The New Nuclear Forensics: Analysis of Nuclear Materials for Security Purposes, The New Nuclear Forensics: Analysis of Nuclear Materials for Security Purposes, Oxford University Press, Oxford, 2015

1.2.2 Nuclear Forensic Interpretation

The goal of the analyses discussed above is to determine the nuclear forensic signatures in the sample. These requires, however, an interpretation (i.e. they need to be explained) before any nuclear forensic conclusions (e.g. about the origin of the material) can be drawn. Nuclear forensic signatures are basically a set of characteristics that can give hints about the history of the material, i.e. how, when and where the material was produced. Some of the signatures that nuclear forensic scientists are interested in, are the following: elemental composition, isotopic composition, impurities, microstructure, age and morphology [12], [13]. By measuring, for example, the ratio of uranium-234 to thorium-230, the scientists can calculate when the last chemical separation of uranium took place (i.e. the age of uranium).

When the signatures have been determined, the nuclear forensic scientists compare them to known information in their archives or databases or in the open literature; and this is complemented by subject matter expertise. For instance, the JRC has its own database on commercial power reactor fuels and related nuclear fuel pellets. One can think of this process to be similar of running a fingerprint sample from a crime scene against a police database, trying to see if there is a match with a known suspect. In the same vein, a nuclear forensic sample can be matched to a material of known origin. For example, one could identify the production site of a uranium fuel or the geological area where the uranium ore was mined from.

The most challenging part occurs in cases when the sample cannot be linked to any known "suspect". In such cases, the scientists can conduct a thorough open-source research and work under the principle of exclusion. These cases illustrate also the importance of international collaboration: the more information and knowledge is exchanged among competent authorities and scientists involved, the easier it becomes to trace the origin of the interdicted material.

2 Analysed Cases and Trends

The phenomenon of illicit trafficking of nuclear and other radioactive material dates back to the early 1990s, as discussed earlier. The end of the Cold War and the collapse of the Soviet Union led to a poor control of nuclear materials in some regions. This resulted in unauthorized access to nuclear material and, by consequence, to cases of theft or diversion. The motivation of the perpetrators was usually attributed to anticipated financial gains. While a black market of nuclear material remains subject to speculation, there have been multiple reported incidents of attempted sale of such materials throughout [14]–[16].

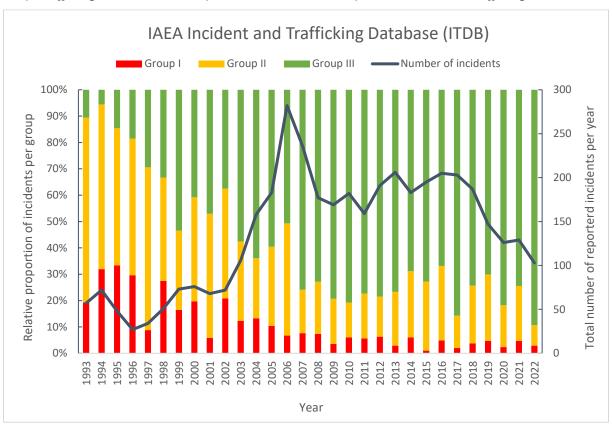


Figure 2: Number of incidents of nuclear and other radioactive material out of regulatory control reported to the ITDB. Group I: Trafficking or malicious use, Group II: Undetermined intent, Group III: Not connected with trafficking or malicious use

Source: Adapted from itdb-factsheet.pdf (iaea.org)

The Incident and Trafficking Database (ITDB) of the IAEA (established in 1995) is a tool to share information between participating states, but also to study and identify the trends in the field of nuclear security [17]. Essentially, it is a database that contains information on worldwide incidents of nuclear and other radioactive material out of regulatory control. Participating states provide the relevant data on a voluntary basis. The database comprises incidents such as: illegal possession of material, illicit trafficking and smuggling of material, unauthorised disposal of material, thefts and discovery of lost radioactive sources. Every year, a factsheet is published which gives information on the number and type of the reported incidents. By the end of 2022, a total number of 4075 incidents had been reported to the ITDB. About 14% of the incidents are related to nuclear materials, while the majority of incidents refers to radioactive sources containing radioisotopes such as Cs-137, Am-241 or Co-60.

According to Figure 2, which contains the information from the latest⁴ ITDB factsheet, one can make the following observation: While the earlier years were characterized by less incidents but more serious threats, in

⁴ At the time of writing.

the later years the incidents increased in number but are considerably less threatening. This is substantiated by the decrease in incidents of Group I. Many of the cases during the 1990s concerned illicit trafficking of nuclear material, some of them involved weapons-grade uranium, mostly in gram quantities with few exceptions of kilogram ones. As the years progressed, the number of incidents of illicit trafficking of nuclear material, decreased. In contrast, the number of incidents involving radioactive sources have increased. More specifically, most of the recent incidents concern stolen or lost radioactive material coming from the industrial or medical field without the intent of malicious use.

The overall increase in the reported incidents observed over time can be explained by two facts: the number of states reporting to the ITDB has steadily increased (currently 144), and the states have spent significant efforts in implementing radiation detectors at key points (border crossings, seaports, airports, etc.) enabling detection. On the other hand, the shift in the type of illicit trafficking incidents can be explained by other factors: reinforcement of physical protection of nuclear facilities and materials, which has made the diversion of nuclear materials considerably more difficult; criminalisation of the unlawful use or possession of nuclear or other radioactive materials; realisation of the unprofitability of the "nuclear material trade".

From the onset of the phenomenon of illicit trafficking of nuclear material, the JRC Karlsruhe has been involved in the response to these incidents and provided nuclear forensic support to EU Member States and beyond. In the initial period the JRC received samples from alarming cases, involving low enriched uranium fuel pellets, highly enriched uranium, several hundred grams of plutonium-uranium mixed oxide powder, and weaponsgrade plutonium. In the more recent years, the JRC has been mostly analysing samples from cases of scrap metal contaminated with uranium. The trend in types of illicit incidents as discussed above is reflected also in the casework performed at the JRC: while initial nuclear forensic support was related to intentional nuclear smuggling and attempted sale of material, more recent cases are linked to unlawful disposal or illegal possession. By today, the JRC Karlsruhe analysed samples from more than 60 incidents.

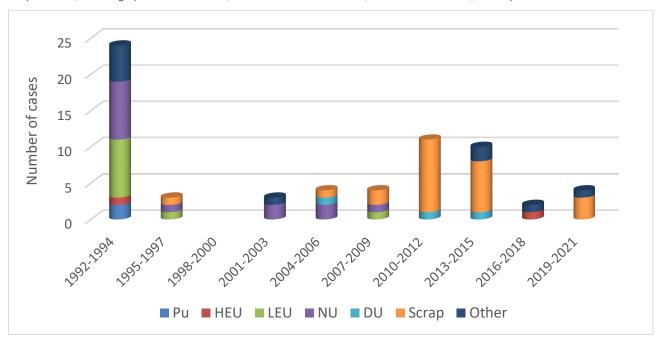


Figure 3: Analysed cases at the JRC Pu=plutonium, HEU=highly enriched uranium, LEU=low enriched uranium, NU=natural uranium, DU=depleted uranium

Source: JRC Karlsruhe

Of course, as with every criminal activity, one should keep in mind that we are likely to see only the tip of the iceberg. The 'dark figure' of illicit trafficking incidents remains unknown. Illicit trafficking may remain undetected or detected incidents might not be reported (for national security reasons). The threat of nuclear terrorism, armed conflicts in countries possessing nuclear materials, political instability in countries where uranium is mined and the covert or overt attempts of states to develop nuclear weapons are, however, reasons for concern. Therefore, it calls for sufficient preparedness at any time and the ability to respond to any possible scenario.

3 Case Examples

In the following section we want to illustrate the nuclear forensics work of the JRC and the support provided by presenting selected examples. The choice of cases aims to illustrate the variety of materials and of incident types that the JRC has dealt with.

3.1 Uranium Fuel Pellets in a Garden

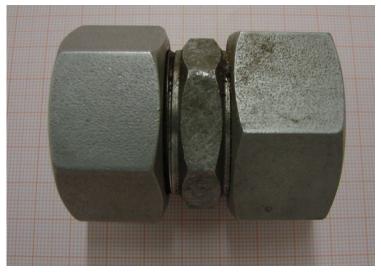
A bizarre but nonetheless intriguing case occurred in February 2007 in Germany, when the authorities seized 14 uranium pellets, which are typically used as fuel in nuclear reactors, from a private property. The pellets were found in Lauenförde, hidden in the garden of a man who claimed to have them in his possession since 1991. After some unsuccessful attempts to submit them to the authorities throughout the years, in 2007 he took a step further: he wrote a letter to the German chancellor Angela Merkel, informing her of the existence of the pellets. This resumed the search action and the authorities finally unearthed the nuclear material which was in a metallic container, wrapped in a plastic bag. Afterwards, they sent the seized material to the JRC Karlsruhe for analysis.

The first step was to find out the uranium-235 enrichment of the pellets. This was done by gamma spectrometry and it showed that all 14 pellets had identical enrichment. More specifically, the pellets had uranium-235 enrichment of 3.44%. This meant that they belonged to the low-enriched uranium category and were very likely intended for a so-called light-water reactor.

The next step in the nuclear forensic analysis was visual inspection of the material. Uranium fuel pellets are created from natural or low enriched uranium oxide powder [18]. After going through a specific processing, pellets take their final form and they appear as small ceramic cylinders (approximately 1 cm diameter and 1 cm height). The confiscated pellets were morphologically (i.e. from the dimensions) identical to each other but had one unusual characteristic: there were numbers engraved on the front faces. In addition, all of them were slightly damaged showing spallings at the edges (Fig.4).

Figure 4: One of the 14 uranium pellets and the metallic container holding the pellets





Source: JRC Karlsruhe

Since the pellets were identical in their morphology and their U-235 enrichment level, only one was chosen for destructive, more detailed analyses. The JRC investigators used mass spectrometry (TIMS) to determine precisely the uranium isotopic composition of the material. They found minute amounts of uranium-236, which indicates that the uranium used for the production of the pellets was recycled, i.e. the uranium had been in a nuclear reactor before it was reprocessed, thus it had not been enriched directly from natural uranium.

Another type of mass spectrometer (ICP-MS) was used to determine the impurity content in the pellets, as well as the age of the uranium. The impurities in the material provide clues on the production process of the pellets. In this case, the impurity level was found to be extremely low, indicating a thorough chemical purification which is untypical for most of the uranium fuel fabrication plants. The age of the uranium has a straightforward meaning, as it points at the date of last chemical purification of the uranium. In the present case, the results indicated November 1990 as the production date of the uranium.

After conducting the analytical measurements and compiling all these signatures, namely the physical characteristics, the uranium enrichment, the uranium isotopic composition, the impurities' content and the age of the pellets, it was time to compare them with known information. For this purpose, the JRC used its database, which contains information on the characteristics of commercial power reactor fuels. With the help of the database, it was possible to track the material's origin to one manufacturer in Europe: the Siemens fuel plant in Hanau, Germany.⁵ The database also revealed that the pellets were intended for a specific kind of reactor type, the so-called pressurised light-water reactor. Notably, the interdicted pellets were not in their final form, which suggested that they had been diverted while they were still in the production stage.

It remains a mystery how the pellets exactly ended up hidden at the man's garden. Some speculations, however, have been expressed. "Der Spiegel", for example, mentions that the man obtained the pellets through another unidentified individual in the course of some illicit drug trade [20]. Once it was clear that the intercepted material was of low radiological and proliferation risk, the media focused on the following question: why was regulatory control lost over the uranium fuel pellets in the first place? Shouldn't this material have been better guarded? [21]. A debate sparked regarding the physical protection measures of nuclear facilities [22].

Furthermore, there was a brief fascination with the man who had hidden the pellets. As mentioned, he had actually contacted the authorities ten years prior to the discovery of the pellets, but at that time he did not manage to point to their exact location [23]. According to some sources [24], he suffered from mental health problems; this might have played a role in why he was not believed at first. What arguably made the difference this time, was his convincing sketch of the container where the pellets were kept. This initiated the successful investigation.

The media also took a special interest in the work of the JRC's "nuclear detectives", as it was clear that they played a key role in solving this case. The nuclear forensics experts of the JRC shared their insights with BBC's *Science Focus* [25], as well as with *Chemical & Engineering News* [26]; they explained how they tracked down the manufacturer of the uranium pellets and they talked about their overall contribution to the field of nuclear forensics.

3.2 Plutonium in a Suitcase

One of the most prominent and controversial cases of smuggling of nuclear material occurred during a sting operation in 1994, in Munich, Germany. A Colombian passenger of a regular Lufthansa flight arriving from Moscow was apprehended at Munich Airport, carrying a suitcase that contained 560 g of plutonium-uranium mixed powder and 210 g of lithium metal (Fig.5). His two Spanish accomplices were also arrested. Although the quantity of the material was insufficient for the creation of a nuclear weapon, it carried the risk of nuclear terrorism and posed serious radiological hazard.

The scientific analysis began by looking into the plutonium to uranium ratio in the mixture. This information would help with determining the type of the reactor that the material was intended for. Despite the initial hypothesis that the sample would be intended for uranium-plutonium mixed oxide fuel (MOX)⁶, their analysis showed otherwise. The composition of the mixture was different than what was expected. The analysis showed that the powder mixture contained plutonium which was close to weapons-grade, having 87% abundance in plutonium-239. Weapons-grade material, however, is assumed to contain at least 93% plutonium-239. The uranium in the mixture was low-enriched having uranium-235 enrichment of 1.8%.

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⁵ The operation of the plant ceased completely in 1995 [19].

⁶ A mixture of plutonium oxide and uranium oxide that is used as fuel in some reactors instead of pure uranium oxide.

Figure 5: X-ray photo of the suitcase containing the can of plutonium-uranium powder and the piece of lithium metal

Source: JRC Karlsruhe

As the sample called for further investigation, the decision was made to take an alternative route and look instead for the type of reactor that the plutonium came from. As pointed out earlier, plutonium does not occur in nature but is produced in nuclear reactors. Depending on the reactor type the plutonium is produced in, the plutonium isotopic composition may vary [27]. Based on model calculations for a range of reactor types, it was possible to exclude those types of reactors as possible origins of the plutonium where the measured isotopic composition did not match the calculated values. Eventually, these considerations lead to the conclusion that the plutonium had been produced in a RBMK type reactor. This is a Russian type reactor which has only been deployed within the former Soviet Union. However, the plutonium could not be related to the specific composition of the uranium in the sample.

Another remarkable observation was obtained from electron microscopy, which showed that the plutonium consisted of two different types of particle morphologies. The final conclusion was that the plutonium oxide was produced by two different processes, however using the same starting material (same isotopic composition) and that the uranium was not directly connected to it. This lead to the conclusion that the powder mixture was most likely just residuals from some fuel production experiments.

Finding out the age of the material is another important parameter that helps to trace back its origin. By using gamma spectrometry, the age of the plutonium was determined, i.e. the point of time when the plutonium was last time chemically purified. An additional mass spectrometry tool (SIMS) was used to verify the age and to analyse the different particle types. The final inference was that the plutonium in the confiscated material had been produced in 1979.

As for the lithium metal, the analysis showed that it was enriched in the isotope lithium-6, i.e. it was not of natural origin. Although not radioactive itself, lithium-6 can be used as a raw material in the construction of a thermonuclear bomb. In sum, the combination of a powder mixture of uranium and plutonium oxide and lithium-6 makes scientifically no sense. However, to a lay person it might suggest that they constitute components (or base materials) for a thermonuclear weapon. It appears plausible that the combination of lithium-6 and plutonium was chosen intentionally by the perpetrators. More importantly, the radiological hazard associated with this material was critical: some argued that it could be enough to poison the water supply of the whole Germany [28].

This was a very interesting case that drew large public attention and created political debate [29]. *Science* journal covered the decisive role the JRC played in the "hunt for the plutonium", explaining the challenges met during the nuclear forensic investigation [30]. The "Plutonium Affair", as it was called by the German media, sparked a lot of controversy, as the Federal Intelligence Service (BND) was accused of staging the smuggling

of the nuclear material. It was argued that in the course of the sting operation, the police offered such an enormous monetary amount that anyone would have been enticed into selling nuclear material. The investigative journalists of Der Spiegel, the German magazine that uncovered the story (presented as political scandal), read the case as a part of a pre-election campaign. They claimed it was a political game: an effort to show that there is an illegal market of nuclear material, with Russia being the supplier, and that Germany's response to it was strict. Due to the fact that this case was heavily politicised, the country of origin was left unconfirmed.

3.3 Weapons-Grade Uranium in Scrap Metal

An analytically complex case took place in September 2010 in Dordrecht, the Netherlands. A radiation alarm was triggered at a scrap metal recycling facility, when multiple items from a scrap metal cargo were found to be contaminated with uranium (Fig.6). The cargo had been shipped from St. Petersburg, Russia. The initial analysis, carried out at the National Institute for Public Health and the Environment of the Netherlands (RIVM), showed that the objects were contaminated with different uranium-235 enrichment levels: from 11% up to 35%. Further investigation was sought at the laboratories of the JRC Karlsruhe, which received samples of the various items.



Figure 6: The objects found at a scrap metal yard contaminated with uranium

Source: RIVM. Netherlands

The analysis started with the necessary visual inspection of the samples, which were in a powder form. This was an essential step to get a first impression of the material and how it looked like, and to get an idea on how to proceed best. In this case, the material showed to be clearly inhomogeneous.

The next important step was gamma spectrometry analysis to assess the uranium-235 enrichment. The results were similar, but not identical to the ones obtained earlier in the Netherlands. More specifically, there were two samples with low enriched uranium and seven samples with high enriched uranium. Moreover, there was a significant discrepancy between the results from the Dutch laboratory and the JRC regarding the uranium-235 enrichment of one sample: 30% vs. 18%, respectively. This discrepancy was a strong indication that the sample was very likely inhomogeneous also from the uranium-235 content.

After that, sub-samples were taken, dissolved and chemically treated in order to determine precisely the isotopic composition of uranium by mass spectrometry. Six out of nine samples yielded different results depending on the method used (mass spectrometry vs. gamma spectrometry). This inconsistency was again an indication of inhomogeneity in the samples, because only a small fraction of samples is used for mass spectrometry analysis as opposed to gamma spectrometry by which a full sample is analysed non-destructively.

The next step was to analyse the uranium content and the impurities in the material. As the interdicted material was scrap metal, it was expected that the samples would contain not only uranium, but also a lot of other elements. The analyses showed that the uranium content was similar in each sample, with only a small variation. The same applied to the impurity pattern of the material, which was also similar in all samples.

As the material was still suspected to be inhomogeneous, the next step was to examine the samples at the particle level. This was started by assessing its morphology, using electron microscopy. Electron microscopy (coupled with the EDX) is a powerful tool that can offer great insights especially when examining inhomogeneous materials [12]. It provides images at high magnification and allows at the same time to determine the elemental composition of each particle. In this case, the JRC investigators found out that the uranium particles occurred in two morphologies: small grain sized particles and rather large fragments, resembling the ones found typically in nuclear fuel. Each sample consisted of both types of uranium particles.

The last microanalytical step was mass spectrometry (SIMS) used for particle analysis. With this, it was possible to study individual particles and check if the material was indeed a mixture of various uranium-235 enrichments. In the end, two populations were identified: low enriched uranium with 2-7% uranium-235 enrichment, and high enriched uranium with 96% uranium-235 enrichment. This pattern was found in all nine samples, which meant that the contaminated scrap metal items came definitely from the same facility. They were very likely cut off from one larger object.

As a final analysis, the age of the uranium or, in other words, the date of the last chemical separation of the uranium was determined. The conclusion was that it took place sometime between the late 70s and the early 80s. Due to the fact that the uranium was inhomogeneous, it meant that the determined age was essentially an average age of the different uranium components.

The most challenging part of the nuclear forensic examination consists in the interpretation of the analytical results. Would it be possible to make sense of the measurement data obtained from inhomogeneous samples with high impurity content, scraped off the surface of metal pieces discovered among a cargo of scrap metal in a recycling facility in the harbour of Rotterdam? Combining information from the JRC's databases and from open-source research, the goal was to identify the facility from which the material could be originating.

The appearance of the contaminated scrap metal objects suggested that they originally could have been parts of a glovebox, which is typically used in nuclear facilities for handling radioactive materials. The population of particles of low enriched uranium (of 2-7%) pointed to the use in power reactor fuels. On the other hand, there were particles with remarkable high uranium-235 enrichment (of 96%). Such uranium is used only in very particular applications, for instance in reactors intended for space use [31]. The contaminated scrap metal must have come from a facility that handled both types of uranium materials. After consulting the open source information, using the subject matter expertise, and taking into account the origin of the cargo (i.e. St Petersburg), a possible origin of the material could point to the Luch Scientific Production Association located at Podolsk, Russia. Luch was founded in 1946 and is, to date, involved with nuclear research and development, as well as power production activities.

Radioactively contaminated scrap metal has become a significant challenge worldwide during the first decade of the 21st century. The phenomenon was of such magnitude that the IAEA demanded increased screening [32]. Most of the reported incidents in Europe occurred in the Netherlands, where the busy Rotterdam seaport and the largest scrap metal yards are located. A sophisticated radiation detection architecture in place at these scrap metal facilities enables the operators to detect radioactively contaminated items or sealed sources that are either intentionally or negligently disposed with the scrap metal. According to the ITDB, objects containing radioactive isotopes such as strontium-90, radium-226, cesium-137 or cobalt-60 have been encountered in scrap metal. If molten with the scrap metal, the resulting steel would be radioactively contaminated and by consequence also the consumer products (e.g. cutlery, buckles, hammers, screwdrivers). The radiological hazard will obviously have economic impacts for the steel (or more generally for the metal) recycling facility. Contaminated scrap metal still remains a challenge to date, however there has been a decrease during the last few years, at least, in the number of uranium contaminated scrap metal cases. This can be attributed to the diligent work between the Netherlands and the JRC to investigate collaboratively such cases and bring it to the worldwide attention.

3.4 Illicit Trafficking of Highly Enriched Uranium around Europe

An important question in nuclear forensic investigation is whether the incident in question is linked to other, previously reported cases. One such case occurred in June 2011 in Chisinau, Moldova. The police arrested six individuals suspected of selling highly enriched uranium. They confiscated a glass vial that contained 4.4 grams of uranium powder. The glass vial was inside a lead container. The enrichment of the material (73% of uranium-235) and the way it was packaged, showed remarkable similarities to seized material of two other cases, one in Bulgaria (1999) and one in France (2001) [33]. Although Moldova is not a European Union Member State, an existing partnership allowed for the JRC to offer its expertise and analyse a sample of the confiscated material. Scientists from Moldova witnessed the examinations performed at the laboratories of the JRC Karlsruhe and contributed drafting the report.

Since the incident in Moldova appeared to be the third in a series of connected cases, it was crucial to examine whether the link was indeed there; the suspicion was not enough. To establish a linkage, information on the following three parameters was necessary: isotopic composition, age of the material and particle morphology. The scientific analysis in this case was particularly challenging, due to the fact that the obtained sample was very small. At the time, the case was still under investigation by the Moldovan authorities and as a result, they could only provide a swipe sample: an invisible amount of powder in a sheet of paper (Fig.7).

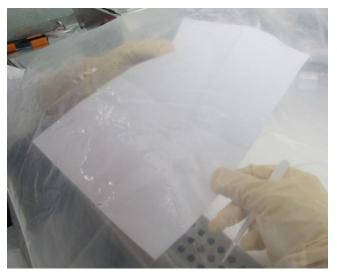


Figure 7: The sheet of paper with invisible amount of uranium obtained for nuclear forensic analysis

Source: JRC Karlsruhe

Working with such a small sample meant in practice that it was impossible to follow the usual procedure. Instead, special preparations needed to be made in order to extract from the swipe sample as much of the material as possible. To be more exact, the focus was on the particle level. By consequence, one could not perform bulk analyses like the routine gamma spectrometry or mass spectrometry. Microanalytical techniques had to be applied from the start.

The first step was to determine the isotopic composition of the uranium. Particle analysis by SIMS technique revealed that the sample was high enriched uranium. More specifically, the uranium-235 enrichment was 73% and the material was homogeneous. In the second step, electron microscopy was used to assess the morphology of the particles, i.e. the shape and size.

The last step was to examine the age of the material. Under the given circumstances, the established procedure had to be adjusted and needed to be downscales in order to handle the minute amount of sample. As such, a combination of methods was used, and the analyses led to the conclusion that the material was produced approximately in December 1992.

Taking all the findings into account, it was possible to confirm that there is a very close resemblance with the materials seized in 1999 in Bulgaria and in 2001 in France. The three seized samples most likely had the same origin of production. Unfortunately, the exact origin could not be identified, due to lack of reference data.

Not being able to confirm the origin is not uncommon; it is indicative of the complexity of nuclear forensics as a discipline. At the same time, a nuclear forensic analysis is just one element in the investigation of the crime at interest. This underlines the importance of combining nuclear forensics with traditional forensics to successfully resolve a criminal investigation.

These three connected cases trigger the question of whether there is more material at large, coming from the same producer and perhaps at risk of falling in the wrong hands. Or could it be that some material was discovered but not reported? In all three incidents, the perpetrators followed a similar pattern: they used the uranium, which was later confiscated, as an indicative sample for the buyer: they showed them a small amount of their allegedly larger stock [33]. If the buyer was to offer the right price, more of the material would become available. Despite the fact that the origin of the material could not be identified beyond doubt, it was argued that in all three cases, the material came from a nuclear facility in Russia [33], [34].

Geographically speaking, Moldova is a country of the Black Sea region which has shown to be particularly affected by illicit trafficking of nuclear and other radioactive material [35]. Moreover, after the disintegration of the Soviet Union, the region of Transnistria is not under control of the Moldovan government and is suspected to be a safe haven for organized crime and smugglers of firearms and other goods [36]. According to a Moldovan police official, it seemed very likely that a single criminal group was behind these three cases. He also suspected a larger stockpile to be hidden in Transnistria [33].

This case illustrates well the importance of the international cooperation in nuclear security. Whereas the IAEA cannot perform nuclear forensic analysis themselves, they can initiate and facilitate a dialog between their Member States and the nuclear forensic laboratories providing analytical support. Another highly useful forum in this respect is the ITWG, where scientists and law enforcement meet annually at a "working-level" to discuss new developments in nuclear forensic research and show some case work. The JRC's high-level reputation in nuclear forensics is recognised in both fora, and it was one of the reasons to enable the nuclear forensics investigation in this particular case at the JRC.

3.5 Gambling Using Radioactive Playing Cards

A peculiar case that landed at the laboratories of the JRC was one that involved radioactive playing cards. To be exact, in November 2017 in Berlin, the German authorities confiscated radioactive chips that had been cut out from playing cards; the chips were contaminated with iodine-125. The material was found in a facility processing household waste during a routine check and the police was able to trace it back to a specific restaurant [37].

lodine-125 is an isotope that is commonly used for cancer treatment. It emits gamma radiation and its half-life is relatively short, 60 days [38]. The radiation hazard it poses is relatively low: health risks occur only with direct contact.

The JRC was asked to conduct nuclear forensic analysis with the following objectives: confirm the presence of iodine-125 in the material, look for the existence of other radioactive isotopes and examine the inner part of the chips which were thought to contain some kind of a metal disk where the radioactivity was deposited.

As usual, the analysis started with a visual inspection of the chips. Their dimensions were measured and they were photographed (Fig.8). The dose rate of the gamma radiation was measured by a portable radiation detector, and interestingly, the radioactivity seemed to be more concentrated on the red side of the radioactive chips rather than on the white side. Next, gamma spectrometry was used to identify the radionuclides in the samples. Apart from iodine-125, no other radioactive material was detected.

According to the initial analysis conducted by the German authorities in Berlin, the iodine-125 was placed on a very thin metal disk made of lead, which was enclosed in the inner part of the playing cards. With this in mind, the decision was made to take apart one of the chips, extract the disk and further analyse it. "Destroying" the chip and at the same time trying to keep the disk intact proved to be a challenge: the material was glued together and it was very fragile. Eventually, it turned out that between the red and white paper there was a very thin metallic layer that covered the whole chip, and not only the centre.

65 - 60 - on 55 55 -5 50 -45 45 45 40 35 S 35 -30 -30 30 25 To Indianalia 25 25 15 3 ուղադարարարարար 08 07 08 08 08 08 08 08 08 08 08 01 8 MM WW

Figure 8: One of the playing cards (a chip) photographed on both sides

Source: JRC Karlsruhe

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Afterwards, the different parts of the chip were examined with an electron microscope combined with an x-ray technique, which gave insights into the morphology and elemental composition of the metallic material. The analysis showed that the thin layer of the dark matter inside the chips consisted of lead sulphide (PbS). The presence of a metallic lead disk could not be confirmed; there was no such indication.

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The most probable scenario for the use of these chips was that they were used by players (gamblers) who wore a special detector in their wrist, although it was not clear which game they were used for [39]. Iodine-125, like any of radioactive material, has no smell and cannot be seen, making it ideal for cheating. This was not the first time that radioactive playing cards made their appearance in the gambling world: more cases have been reported, before and after this specific incident. For instance, similar cases occurred in 2018 and 2019 in Romania, which led to a criminal investigation. That investigation revealed that the cards were used for the so-called Xóc Đĩa, a Vietnamese gambling [40]. The sophistication of this cheating scheme suggests that there might be links to a criminal organization, however this has not been confirmed yet.

4 Impacts

The work of the JRC in nuclear forensics aims at developing capabilities and providing the necessary support to EU Member States (and other partnering states) to fight illicit trafficking and other unauthorised use of nuclear and other radioactive materials. In a sense, the nuclear forensics work at the JRC ultimately contributes to the European Security Union Strategy. While incidents involving nuclear material do not happen very often, their potential harm is critical (low probability, high impact). Concerns around nuclear security become even more relevant when taking into consideration the current geopolitical status that has set states upgrading their nuclear arsenals [41].

The JRC addresses the nuclear security challenges by building strong expertise in the field of nuclear forensics. The casework in support of EU Member States' authorities is supplemented by R&D activities, by strong international collaborations, by capacity building projects with Member States and outside the EU, and through relevant training activities at the JRC's European Nuclear Security Training Centre (EUSECTRA).

Policy

First and foremost, the nuclear forensics at the JRC serves the European Commission's policies, by providing evidence-based knowledge and expertise, also via various other DGs, such as DG HOME, DG INTPA, FPI and DG ENER. Nuclear security (including nuclear forensics) is a national responsibility, hence Member States need to implement and sustain appropriate measures. These measures, however, can be complemented by the JRC capabilities and consequently reinforce national capacities. Specifically, the JRC's ability to provide nuclear forensic support to Member States' authorities investigating cases of nuclear material that has been found out of regulatory control, provides the law enforcement with the necessary insights to prosecute perpetrators. At the same time, looking at the bigger picture, the state-of-the-art nuclear forensic capabilities and expertise, which the JRC offers, serve the general security policies of the EU. In addition to the operational support to Member States, the JRC also shares its expertise and helps through dedicated projects enhancing Member States' nuclear security preparedness. By undertaking capacity building activities, e.g., in the EU accession countries, Caucasus and Black Sea region, the JRC enhances the nuclear security also in the neighbourhood regions to EU. Moreover, in the context of series of Nuclear Security Summits, the JRC hosted and co-organized Counter Nuclear Smuggling (CNS) workshops in partnership with the USA. These CNS workshops highlighted the importance of having a national nuclear security architecture, which includes nuclear detection and nuclear forensic capabilities. The JRC also works with the International Atomic Energy Agency on promoting a nuclear security culture and they initiated e.g. an improvement of the IAEA's Incident and Trafficking Database. And these are only a few selected examples, how the JRC has contributed to the nuclear security worldwide. The JRC has been providing policy support at various levels in promoting nuclear security. It is, however, important to ensure transparency of the policy needs and policy implementation in order to further enhance the benefits of the JRC's service to the Member States.

Scientific Community

Having been a pioneer at the nuclear forensics field since its beginning, the JRC keeps contributing to the knowledge production and expertise within the scientific community. Nuclear forensic scientists at the JRC publish peer-reviewed papers in well-respected scientific journals continuously. Such R&D work has contributed to numerous ground-breaking developments in the field of nuclear forensic signatures. This work is highly recognised worldwide and many of these signatures are put in use in laboratories that are only now starting to build their nuclear forensic capacity. In addition, the JRC maintains strong bonds with international collaborators, such as the IAEA and the ITWG; they work together to share knowledge and stay up to date with the latest developments. For instance, the JRC contributes to IAEA's activities in developing nuclear forensics capability by participating in consultancies on drafting guidance documents, by participating in the coordinated research projects (CRP) and by developing, hosting and supporting implementation of training courses and by hosting trainees. In addition to this, the JRC has been co-chairing ITWG since its foundation in 1995 (together with the USA) and contributing to the work of ITWG's various task groups. What is more, the JRC provides technical trainings, expert meetings and workshops to states inside and outside the EU, helping them develop their own nuclear forensic competencies. At the same time, the JRC keeps on learning from other emerging fields, such as Artificial Intelligence (AI), and applying that to nuclear forensics.

Society

One should keep in mind that the work of the JRC in nuclear forensics to support the EU policy making, and its strong presence in the scientific community, essentially serve one goal: keeping Europeans safe. This comprises

not only the aspects of security, but also radiation safety, public health, economy and the environment. The JRC's expertise in nuclear forensics has demonstrated, through the case work described in this report, that it can provide a significant contribution to keeping European safe and secure. With the JRC's support, Member States are in a better position to respond to illicit incidents, to malicious or criminal acts involving nuclear material. This also contributes to deter nuclear terrorism. The fact that the number of nuclear smuggling incidents involving strategic material such as highly enriched uranium or plutonium has gradually decreased could be indicative of the effectiveness of nuclear security measures and of the deterrent effect of nuclear forensics. The overall number of incidents (see Figure 2), however, has remained around 150 per year. Yet, the public awareness of this threat is poor and media do report only spectacular cases, which have become rather exceptional.

While nuclear forensics is a highly specialized scientific field with only few expert laboratories globally, it is important to maintain these capabilities within the EU and make them available to Member States when needed. Working towards advanced nuclear forensic capabilities is a measure of preparedness and of vigilance. The latter is key in protecting the European citizens.

5 Conclusions

Illicit trafficking and smuggling of nuclear or other radioactive material is a critical security concern internationally. When nuclear or other radioactive material ends up in the wrong hands, the safety of the citizens is at stake, be it through careless or negligent handling or be it through malicious acts. The latter encompasses intended exposure to or dispersion of radioactive material, "dirty bombs" or improvised nuclear weapons. The consequences of such acts would be devastating for the public, the environment and the economy. International treaties and conventions have been put in place to encourage states to criminalize the unlawful possession and use of nuclear and other radioactive materials, and rigorously investigate and prosecute such incidents.

The JRC has been at the forefront of the fight against illicit trafficking of nuclear or other radioactive material already a couple of decades. Since the occurrence of the first incidents in the early 1990s, the JRC has offered its expertise in nuclear forensics to support law enforcement agencies and policymaking bodies of EU Member States, third countries as well as international organizations. Nuclear forensics aims ultimately at identifying the possible origin of the interdicted material, its intended use, its place and date of production and the last legal owner. This is a condition to improve physical protection and safeguarding of the nuclear materials at the place of theft or diversion and thus prevent future incidents.

With its multidisciplinary scientific team, rich experience and state-of-the-art technical equipment, the JRC has offered its expert insights on over 60 real-life nuclear forensic investigations. Apart from its casework, the JRC has a vibrant presence in the scientific community, contributing with its research and development activities, its publications, its training and its collaboration with international partners. By leveraging its expertise and resources, the JRC continues to play a vital role in fortifying international efforts to prevent nuclear proliferation, detect illicit activities, and maintain global peace and stability.

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List of abbreviations and definitions

CPPNM Convention on the Physical Protection of Nuclear Material

CTBT Comprehensive Nuclear-Test-Ban Treaty

DG ENER Directorate-General for Energy

DG FPI Directorate-General for Service for Foreign Policy Instruments

DG HOME Directorate-General for Migration and Home Affairs
DG INTPA Directorate-General for International Partnerships

DU Depleted Uranium
EU European Union

EUSECTRA European Nuclear Security Training Centre

Euratom European Atomic Energy Community

HEU Highly Enriched Uranium

IAEA International Atomic Energy Agency

ITDB IAEA's Incident and Trafficking Database

ITWG Nuclear Forensics International Technical Working Group

JRC Joint Research Centre

LA-ICP-MS Laser Ablation Inductively Coupled Plasma Mass Spectrometry

LEU Low Enriched Uranium

NPT Treaty on the Non-Proliferation of Nuclear Weapons

NU Natural Uranium

R&D Research and Development
SEM Scanning Electron Microscope

SIMS Secondary Ion Mass Spectrometry

TIMS Thermal Ionization Mass Spectrometry

List of figures

Figure 1: Flowchart of nuclear forensic analysis	5
Figure 2: Number of incidents of nuclear and other radioactive material out of regulatory control reported t the ITDB. Group I: Trafficking or malicious use, Group II: Undetermined intent, Group III: Not connected wi trafficking or malicious use	ith
Figure 3: Analysed cases at the JRC	9
Figure 4: One of the 14 uranium pellets and the metallic container holding the pellets	10
Figure 5: X-ray photo of the suitcase containing the can of plutonium-uranium powder and the piece of lithium metal	12
Figure 6: The objects found at a scrap metal yard contaminated with uranium	13
Figure 7: The sheet of paper with invisible amount of uranium obtained for nuclear forensic analysis	15
Figure 8: One of the playing cards (a chip) photographed on both sides	17

List of tables

Table 1: Class	ification of ι	ıranium based	on relat	ive abundan	ce in uraniuı	m-235 isotope	2	4
Table 2: Analy	tical techniq	ues commonly	used by	the JRC for	the nuclear	forensic analy	/sis	6

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