Nuclear forensics—a methodology providing clues on the origin of illicitly trafficked nuclear materials

Klaus Mayer,* Maria Wallenius and Ian Ray

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1. Introduction

Reports on seizures of nuclear material (i.e., uranium or plutonium) and of radioactive sources continue to attract the attention of the public and are a reason for concern due to the hazard associated with such material. Once the material has been detected and secured, the questions on the intended use, the radiological hazard and the origin of the material need to be answered. Classical forensic techniques address the criminalistic part of the case, i.e., the identification of the suspect criminal. The phenomenon of nuclear smuggling and illicit trafficking of nuclear material has led to the development of a new branch of science: nuclear forensics. The key issue is the identification of the origin of the material, in order to improve the physical protection measures and prevent future thefts or diversions. The key challenge is the specificity and complexity of the nuclear area and the particular requirements for handling such material. Nuclear forensic science makes use of analytical techniques that were actually developed for applications related to the nuclear fuel cycle, hence appropriate and safe handling of the samples during the investigations is assured. For interpretation of the results, nuclear forensic science relies to a large extent on the expertise and experience of the investigating scientists. Knowledge in areas such as radiochemistry, nuclear physics, reactor physics, materials science and in the nuclear fuel cycle are required. The conclusions, however, need to be supported by reference data wherever possible.

2. Methodological approach

Nuclear forensic investigations will start after material has been seized and categorized as ‘nuclear material’. They are carried out in order to answer specific questions on the nature

Klaus Mayer received his MSc in Chemistry from the Technical University of Karlsruhe in 1985, followed by a PhD in Radio- and Analytical Chemistry in 1987. After a post-doctoral fellowship at ITU, he joined the Institute for Reference Materials and Measurements in 1990 and worked on high accuracy isotope mass spectrometry and isotopic reference material for uranium and plutonium and on nuclear safeguards. In 1996 he returned to ITU, responsible for the set-up and the installation of safeguards on-site laboratories. Today, he is in charge of the Safeguards Programme of ITU and of the development of measurement methodologies for nuclear materials, including nuclear forensic analysis. He is author of over 150 publications, and acts as co-chairman of the International Technical Working Group on Nuclear Smuggling (ITWG).

Maria Wallenius received her MSc in Radiochemistry at the University of Helsinki, Finland in 1994. She worked at the University of Helsinki as a research scientist in the safeguards project (Determination of uranium content, isotopic composition and impurities in fresh nuclear fuel by various techniques) and then moved to the Institute for Transuranium Elements (ITU) in 1996 to study for a PhD in Radiochemistry (“Origin determination of reactor produced plutonium by mass spectrometric techniques: Application to nuclear forensic science and safeguards”) which she obtained in 2001. She continued at the ITU as a research scientist developing new methods using mass spectrometry (especially using TIMS and ICP-MS) in the field of safeguards and nuclear forensics.

Ian Ray received his MA in Natural Sciences at the University of Cambridge in 1966 and his DPhil at the University of Oxford in 1971 in the field of high resolution electron microscopy. After a short period lecturing in the University he joined the Institute for Transuranium Elements in Karlsruhe, Germany, in 1976 and took over responsibility for the electron microscope laboratory. Since 1999 he has been responsible for the coordination of Nuclear Forensic Science activities in the Institute.
of the material and its origin, such as the intended use, the mode of production, the plant and production batch, the last legal owner and the smuggling route. The investigations may comprise conventional forensic tests applied to radioactive material, the morphology of the material, the structure of the material components, the composition of traces in the material and its packing, the isotopic composition of the nuclear material itself and of minor constituents.

Nuclear forensic investigations basically draw upon the information inherent to the material. Nuclear material is generally of anthropogenic origin, i.e., the result of a production process. The nature of this production process is reflected in the elemental and isotopic composition of the material as well as in its microscopic and macroscopic appearance. All of these parameters can be measured using the appropriate analytical technique. Some parameters can be combined to a “nuclear fingerprint”, i.e., they are characteristic for the mode of production of the material. Hence, they may provide a clue as to the origin of the material. Nuclear material is either produced by uranium mining, which is normally followed by isotope enrichment of uranium, or by neutron capture (e.g., in a reactor), which transforms uranium into the transuranium elements neptunium, plutonium, etc. The isotopic composition of the latter depends on the reactor conditions and thus allows the drawing of conclusions on the reactor type and the fuel initially used.1

Consequently, a suite of analytical techniques, specifically adapted to the needs of radioactive material, is required.

3. Analytical techniques

The analytical techniques used in nuclear forensic investigations may be subdivided into two categories: commonly applied chemical and physical analytical methods and radio-analytical methods. The latter make use of the radiation emitted from the material. This radiation is characteristic for the emitting nuclide. The two categories of analytical techniques offer results of complementary nature, thus providing a maximum of information on the material under investigation. Table 1 summarizes the analytical techniques used most commonly in nuclear forensic investigations. Some techniques are applicable to radioactive materials only (e.g., alpha or gamma spectrometry) but most of the methods are commonly applied in analytical chemistry or in materials science (e.g., secondary ion mass spectrometry [SIMS], scanning electron microscopy [SEM]). In the latter case the techniques need to be adapted to the specific requirements associated with handling radioactive materials (glove-boxes, shielding, etc.).

Controlling the radiological hazard is of paramount importance at all stages of the investigation. Furthermore, attention should be paid to preserving classical forensic evidence. In the nuclear analytical laboratory, the material is first subjected to visual inspection. This may already reveal useful information on the material itself (e.g., physical form, geometry, primary packing) and provide the starting point for further analysis. It may be complemented by imaging techniques, namely optical microscopy for examination of the sample at high magnification.

If the analysed material contains fuel pellets, their dimensions (height, diameter and the size of a possible central hole) and mass are measured. These so-called macroscopic parameters, together with the 235U enrichment, are characteristic and they can already reveal the reactor type, where the pellets are used.

Seizures occur under varying circumstances: at border crossings, during searches on specific targets or just by accident. They reveal nuclear material in different amounts and different shapes. Figs. 1–4 illustrate this variety.

3.1. Radiometric methods

Radiometric techniques measure the radiation that radioactive nuclides emit when they decay to a daughter nuclide. Most of the heavy nuclides (e.g., U and Pu) decay by emitting an alpha particle. However, gamma radiation is also often emitted after the alpha decay to bring the daughter nuclide from an excited state into the transuranium elements neptunium, plutonium, etc.

### Table 1 Techniques used for analysing seized nuclear material

<table>
<thead>
<tr>
<th>Techniques/methods</th>
<th>First analysis</th>
<th>Information</th>
<th>Detailed analysis</th>
<th>Information</th>
</tr>
</thead>
<tbody>
<tr>
<td>Radiological</td>
<td>Estimated total activity</td>
<td>Radiological hazard</td>
<td>SEM (EDX)</td>
<td>Microstructure and elemental composition</td>
</tr>
<tr>
<td></td>
<td>Dose rate (α, γ, n)</td>
<td>Precautions</td>
<td>XRD</td>
<td>Crystal structure</td>
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<td></td>
<td>Surface contamination</td>
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<td>TEM</td>
<td>Microstructure</td>
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<td>Physical</td>
<td>Visual inspection</td>
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<td>characterization</td>
<td>Photography</td>
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<td>Sise measurement</td>
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<td>Optical microscopy</td>
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<td>Radiography</td>
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<td>Weighing</td>
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<td>Traditional forensic analysis</td>
<td>Fingersprints, fibers</td>
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<td>Isotope analysis</td>
<td>γ-Spectroscopy</td>
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<td>Elemental/Chemical analysis</td>
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state to the ground state. Each nuclide emits gamma rays of energies characteristic of this particular nuclide.

3.1.1. Gamma spectrometry. Gamma spectrometry is the first technique that is used when seized nuclear material is investigated. This is essentially due to the non-destructive character of the technique, gamma rays, i.e., photons of some ten up to several hundred keV, are only slightly attenuated by the packing material (unless shielding like lead is used). Thus, already the initial measurements in the field (e.g., in border control points) are carried out with simple, portable gamma spectrometers. They aim at a categorisation of the material, i.e., distinguish between naturally occurring radioactive material, radioactive source, radiotherapy nuclide or nuclear material.

In laboratories, more sophisticated gamma spectrometers, so-called high resolution gamma spectrometers (HRGS), are used. Their energy resolution is much better than in the portable instruments, thus gamma rays with energies very close to each other can be resolved in the spectrum. Specific codes like the MGA,2 FRAM3 or MGAU4 codes are used to deconvolute the low-energy spectra observed for plutonium and uranium, respectively, and allow calculation of the isotopic composition of the material.

It should, however, be noted that some nuclides like $^{242}$Pu or $^{236}$U cannot be detected by gamma spectrometry; in these cases mass spectrometry offers a useful analytical alternative. An example of a gamma spectrum of a natural uranium sample is shown in Fig. 5.

3.1.2. Alpha spectrometry. Alpha particles, i.e., $^{4}$He$^{+}$ having energies of 3–8 MeV, are stopped for example by a paper sheet, because of their strong interaction with matter. Consequently, an alpha measurement through packing material or shielding is impossible. Alpha spectrometry is a destructive technique, which requires rather laborious sample preparation. This may include dissolution, chemical separation and target preparation. Pu/Am separation is especially important because the alpha particles emitted by $^{238}$Pu and $^{241}$Am have similar energies and thus overlap in the spectrum. Similarly, the alpha energies of $^{239}$Pu and $^{240}$Pu are very close and cannot be
Inductively coupled plasma mass spectrometry (ICP-MS) is a versatile technique that came to the market in the 1980s and was used mainly for impurity measurements. Samples are usually introduced in the form of an aerosol, which is generated by nebulizing the sample solution. The ions are produced in the plasma at temperatures of 5000–8000 K. The salient features of ICP-MS are the multi-element capability, the high sample throughput, the good sensitivity and the large dynamic range. In modern instruments magnetic sectors are replacing the quadrupoles for mass separation. Consequently, multi collector detection (enabling simultaneous detection of different isotopes) can be combined with the ICP-source, and isotope ratios can be measured at precisions similar to those of the TIMS technique. ICP-MS measurements have been applied for impurity measurements as well as for isotope ratio measurements in seized samples and in samples of known origin.5,6

3.2.2. Inductively coupled plasma mass spectrometry. Inductively coupled plasma mass spectrometry (ICP-MS) is a useful technique for the determination of the isotopic composition of samples. Its elemental coverage encompasses lithium through uranium, with the ability to determine impurity levels from the sub-ppb range to the percent level. In glow discharge mass spectrometry (GD-MS) the solid sample serves as the cathode for the sputtering and ionisation processes. Basically, it can be used for the same purpose as ICP-MS, i.e., impurity and isotope ratio measurements.7 GD-MS can be a very effective tool for panoramic impurity analysis in solid samples. However, it suffers from matrix-effects and does not reach the degree of precision and accuracy achieved in TIMS or ICP-MS. Due to the small spot size that is sputtered, sample heterogeneity may yield misleading results. The fact that the analysis is directly performed from solid samples and that very small samples can be investigated are most advantageous features of this technique.

3.2.3. Glow discharge mass spectrometry. Glow discharge mass spectrometry is the most comprehensive and sensitive technique available for the analysis of inorganic solids. It is capable of analyzing conducting, semi-conducting and insulating samples. Its elemental coverage encompasses lithium through uranium, with the ability to determine impurity levels from the sub-ppb range to the percent level. In glow discharge mass spectrometry (GD-MS) the solid sample serves as the cathode for the sputtering and ionisation processes. Basically, it can be used for the same purpose as ICP-MS, i.e., impurity and isotope ratio measurements.7 GD-MS can be a very effective tool for panoramic impurity analysis in solid samples. However, it suffers from matrix-effects and does not reach the degree of precision and accuracy achieved in TIMS or ICP-MS. Due to the small spot size that is sputtered, sample heterogeneity may yield misleading results. The fact that the analysis is directly performed from solid samples and that very small samples can be investigated are most advantageous features of this technique.

3.3. Microstructural techniques

3.3.1. Profilometry. Profilometry is used to measure, for example, the surface roughness of the materials and crater depth after sputtering. In the nuclear field this technique can be used to determine the surface roughness of fuel pellets. Fuel pellets are finished by grinding to bring the cylindrical shape to the specified diameter with low tolerance. There are two different grinding procedures used, so-called wet and dry grinding. By wet grinding a generally smoother surface is achieved than by dry grinding. Thus, if pellets for a certain
type of reactor are produced in several fabrication plants, they can be distinguished from each other by surface roughness, if the plants use different grinding methods.10

3.3.2. Electron microscopy. Electron microscopes use a focused beam of electrons of high energy to examine objects on a very fine scale. Interactions with the sample affect the electron beam. These effects are detected and transformed into an image, a spectrum or a diffraction pattern. This may yield information on topography, morphology, elemental composition and crystallographic structure, and lead to the development of the concept of a microstructural fingerprint.11

3.3.2.1. Scanning electron microscopy. Scanning electron microscopy (SEM) provides pictures of the surface of objects at high magnification and with a large depth of field. SEM detects the electrons that are back-scattered or emitted (i.e., secondary electrons) from the specimen’s surface. The characteristic X-rays emitted from the specimen due to fluorescence provide information on the chemical elements contained in the material. Preparation of the samples is relatively easy since most SEMs only require the sample to be conductive. In nuclear forensics, the application of SEM is particularly interesting for powder samples or inhomogeneous samples. Different components can be investigated separately for their particle morphology and elemental composition, thus possibly providing hints on the production process.

A sample seized at Munich Airport in 1994 consisted of a powder mixture of three components: uranium, plutonium rod shaped particles and plutonium platelets. These last were investigated in detail by SEM and TEM as shown in Figs. 6–11. While the SEM pictures do not allow us to unambiguously distinguish between the two materials, TEM analysis provides complementary and useful information.

3.3.2.2. Transmission electron microscopy. Transmission electron microscopy (TEM) makes use of the electrons that have passed through the specimen. Therefore only thin layers can be examined. The sample preparation is rather laborious. However, the resolution reaches the sub-nanometre range.

The results obtained by SEM and TEM clearly show that the two materials originate from different processes. The significantly different grain size distribution indicates that the techniques for precipitation and the conditions for calcining the material were not identical.

4. New developments

When the phenomenon of nuclear smuggling appeared in the early 1990s and the first seized samples needed to be investigated, the methods available in other fields (e.g., safeguards analysis) were applied for nuclear forensic analysis;

Table 2  Typical sample sizes and typically achieved measurement uncertainties

<table>
<thead>
<tr>
<th>Technique</th>
<th>Parameter investigated</th>
<th>Typical sample mass or particle diameter</th>
<th>Typical relative measurement uncertainty</th>
</tr>
</thead>
<tbody>
<tr>
<td>TIMS</td>
<td>Isotope ratios</td>
<td>10 ng–1 μg</td>
<td>0.05%–1%</td>
</tr>
<tr>
<td>ICP-MS</td>
<td>Isotope ratios</td>
<td>0.1 ng–1 μg</td>
<td>0.1%–2%</td>
</tr>
<tr>
<td></td>
<td>Concentration</td>
<td>0.1 pg–1 μg</td>
<td>1%–30%</td>
</tr>
<tr>
<td>GDMS</td>
<td>Isotope ratios</td>
<td>1–100 μg</td>
<td>1%–10%</td>
</tr>
<tr>
<td></td>
<td>Concentration</td>
<td>1–100 μg</td>
<td>15%–25%</td>
</tr>
<tr>
<td>SIMS</td>
<td>Isotope ratios</td>
<td>&gt;0.5 μm</td>
<td>1%–10%</td>
</tr>
<tr>
<td></td>
<td>Concentration</td>
<td>&gt;0.5 μm</td>
<td>10–20%</td>
</tr>
</tbody>
</table>

Fig. 6  SEM picture of a PuO2 reference sample of a known fabrication plant.

Fig. 7  SEM picture of PuO2 platelets from a sample seized in 1994 at Munich Airport, Germany.

Fig. 8  The platelet size distribution was established by SEM for the two samples. It does, however, not show a significant difference between the two samples.
no specialised methods were available. Later, the methods were adapted and focused to the specific needs of nuclear forensics. However, it was then noticed that new methods dedicated only to the investigation of nuclear forensic materials needed to be developed in order to obtain specific information. In this chapter the main developments from recent years are discussed.

4.1. Age determination

Determination of the age of materials is common in geology and archaeology. The age of organic materials (e.g., bones) is determined using the $^{14}$C “clock”, whereas inorganic materials (e.g., minerals) have several more possibilities for age determination (e.g., measurement of isotope abundance ratios in Sr/Rb, Sm/Nd, U/Pb). Basically, the disintegration of a radioactive (parent-) isotope and the build up of a corresponding amount of daughter nuclide serve as a built-in chronometer. The same principle is applicable to nuclear materials, albeit under different boundary conditions, because the time periods to be determined (age of the material, i.e., time elapsed since the last purification of the material) are short compared with the half-life of the nuclides. This means that in this short time only a small amount of daughter nuclide will grow-in. Thus, the resulting parent/daughter ratios are always very high, which often makes the direct measurement impossible. Additionally, a separation and some very sensitive measurement techniques are required. Useful parent/daughter pairs are $^{234}$U/$^{230}$Th and $^{235}$U/$^{231}$Pa, and in the case of plutonium, $^{239}$Pu/$^{234}$U, $^{239}$Pu/$^{235}$U, $^{240}$Pu/$^{236}$U and $^{241}$Pu/$^{241}$Am. As the daughter products are also radioactive, the granddaughters (e.g., $^{234}$U/$^{226}$Ra) as well can be used for the age determination.

First, the age determination was developed for bulk plutonium samples. This is classically achieved by a $\gamma$-spectrometric measurement of the $^{241}$Pu/$^{241}$Am ratio. In a more recent work, the samples were spiked with $^{244}$Pu, $^{243}$Am and $^{233}$U, and elements were separated and measured by TIMS. If consistent ages are obtained for the four useful parent/daughter relationships, systematic errors can be excluded. Incomplete separation of the initial material will not properly set the clock to zero. The impact on the age determination of residual uranium in a plutonium sample was studied for different types of plutonium.

Age determination of Pu particles poses a particular challenge. In the case of single particles, no U/Pu/Am separation can be performed; the isotope ratios need to be measured directly by SIMS. Parent/daughter ratios suffering from isobaric interferences (e.g., $^{241}$Pu/$^{241}$Am) cannot be used for age determination. As outlined above, the consistency of results allows the drawing of conclusions on possibly present residual uranium. For an accurate age determination, measurement effects (due to, for example, different ionisation potentials of U and Pu) need to be quantified and corrected for.

Owing to the long half-lives of the uranium isotopes and consequently the small amounts of daughter products growing in, age determination of uranium is more challenging than for plutonium. The preferred parent/daughter relation is the $^{234}$U/$^{230}$Th, although $^{234}$U is a minor abundant isotope in uranium. U age determination has been demonstrated for a wide range of $^{235}$U enrichments, from natural uranium up to highly enriched material. Three different methods for quantifying the daughter nuclides have been tested, i.e., alpha spectrometry using $^{228}$Th as a spike, TIMS using $^{235}$Th as a spike and ICP-MS without spiking, in other words direct ratio measurement. U age determination has also been demonstrated using the other parent/daughter relationship $^{235}$U/$^{231}$Pa by alpha spectrometry; however, this suffers from a lack of suitable spike isotopes of Pa. The longest living Pa isotope after $^{231}$Pa is $^{233}$Pa, with a half-life of 27 days.
4.2. Reactor type determination (Pu production)

Plutonium is generated in nuclear reactors after neutron capture of uranium and subsequent decay of the intermediate product. Heavier isotopes of plutonium are produced by further neutron captures. The probability for this reaction (the so-called cross section) depends on the energy of the neutrons and varies also from one Pu isotope to the other. Different reactor types show different neutron energy distributions, therefore the plutonium isotopic composition is a key parameter for the identification of the reactor type where the Pu was produced. The isotopic composition of Pu is influenced by several parameters. These include the neutron spectrum of the reactor (hard or soft, i.e., fast or thermal neutrons), initial U fuel composition (\(^{235}U\) enrichment) and burn-up (duration and intensity of irradiation in the reactor). The isotopic composition of Pu in most common reactor types (e.g., light-water reactor, heavy-water reactor, fast breeder reactor) can be calculated using computer codes, e.g. ORIGEN\(^{20}\) and SCALE\(^{21}\); thus measured data from seized samples can be compared to calculated values.

A correlation was found that separates the main reactor types clearly from each other (Fig. 12).\(^{22,23}\) The \(^{238}Pu\) abundance (x-axis) is affected by the initial enrichment of the \(^{235}U\) in the fuel, i.e., the higher the enrichment, the higher the \(^{238}Pu\) abundance. The \(\gamma\)-axis (\(^{242}Pu/^{240}Pu\) ratio) is affected by the neutron spectrum, i.e., the softer the spectrum, the higher the ratio.

4.3. Geolocation

The natural variations in the isotopic composition of certain elements may provide clues on the geographic origin of the material, and is in fact one of the methodologies used in geolocation.\(^{24}\) Oxygen in nature consists of three stable isotopes, \(^{16}O\) (99.762%), \(^{17}O\) (0.038%) and \(^{18}O\) (0.200%). However, the isotopic composition can vary slightly due to the different chemical and physical reactions (e.g., isotope exchange, evaporation, condensation) and lead to isotopic fractionation. Relative variations up to 5% in the \(^{18}O/^{16}O\) ratio have been observed in the \(\text{UO}_2\) product. An intensive study was performed using three mass spectrometric techniques, TIMS, SIMS and GDMS. In particular the TIMS and SIMS results reveal significant differences between \(\text{UO}_2\) pellets produced in different locations.\(^{25-27}\)

Also other parameters are being studied for geolocation purposes. In natural uranium the chemical impurities may provide information on the ore body where the material was mined. Furthermore, it could be shown that the isotopic composition of stable elements (e.g., Pb\(^{28,29}\)) also shows significant variations that may be attributed to the origin of the material. TIMS and ICP-MS are used for accurate determination of the isotopic composition of the relevant elements.

5. Reference data

The information obtained from the analysis of seized nuclear material may be of endogenic or of exogenic nature. The first is self-explanatory, e.g. the age of the material, also the intended use (e.g., power production, weapons) can be deduced directly from the measured data. The second category requires reference data for comparison in order to identify, for example, the place of production. Information on nuclear fuel materials has been compiled in a relational database from the
open literature and from bilateral agreements with fuel manufacturers. The database contains information on pellet geometry, uranium enrichment, specified values for chemical impurities, manufacturer and reactor type and location. The database is used to guide the analysis in a step-by-step approach. The identification of the origin of the material is achieved on the basis of the exclusion principle.30–32 The database has recently been complemented by an electronic literature archive on non-conventional fuels.33

6. International co-operation

Nuclear forensic science is closely related to the phenomenon of illicit trafficking, thus to nuclear security and nuclear safeguards. A border crossing threat is associated with it, which calls for an internationally co-ordinated response.34 The International Technical Working Group on combating nuclear smuggling (ITWG) was established some ten years ago, in order to advance the science of nuclear forensics for attributing nuclear material.35 This is achieved by exchange of information, by developing procedures and recommendations and by exercises.36,37

A number of bi- or multilateral assistance programmes have been set up in order to improve the detection capabilities and to arrange for nuclear forensic assistance.38 Also, the International Atomic Energy Agency promotes the development of nuclear forensics and facilitates the provision of assistance to requesting states which do not have their own nuclear forensic capabilities.39

7. Conclusions

In the last fifteen years we have see the emergence of a new and potentially hazardous form of smuggling: that of nuclear and radioactive materials. This triggered the development of a new discipline in science, enabling support of law enforcement authorities in combating illicit trafficking and dealing with criminal environmental issues: nuclear forensics. Existing analytical techniques, as used in material science, in nuclear material safeguards and in environmental analysis, were adapted to the specific needs of nuclear forensic investigations. Characteristic parameters (e.g., isotopic composition, chemical impurities, macro- and microstructure) can be combined to a “nuclear fingerprint”, pointing at the origin of the material. Further research is being carried out, aiming at identifying other useful material characteristics in order to reduce the ambiguities often remaining in the interpretation of the data and in the source attribution. New methodologies need to be developed, validated and implemented in order to determine parameters with good precision and accuracy. The availability of up-to-date reference on nuclear material is essential in order to identify the origin and the intended use of the material, or to exclude certain origins.

Significant progress has been achieved in a relatively short time in this new and fascinating discipline. Due to the nature of the material involved and the related handling problems, the specific adaptation of measurement instrumentation, the complexity of the data interpretation and the particular expertise required, only few laboratories are working in this area. However, the hazards involved with nuclear smuggling and the potential relation with nuclear terrorism are the driving forces for deploying and further improving this methodology.

Klaus Mayer,* Maria Wallenius and Ian Ray

European Commission, Joint Research Centre, Institute for Transuranium Elements, P.O.Box 2340, 76125 Karlsruhe, Germany.
E-mail: mayeri@itu.fzk.de

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