

# Evaluating environmental sampling data for Safeguards – principles and advances

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**Abstract:** Since the inception of using environmental sampling for Safeguards purposes at the IAEA to detect undeclared nuclear activities, the applied analytical techniques, evaluation methods and software have evolved significantly. Also, the number of environmental samples that are collected each year during inspection activities has been steadily growing. The increase in data quality, quantity and variety presents challenges to the analysts who receive and evaluate these data on an operational basis. This paper gives an overview of the basic principles of environmental sampling and the advances that have been made to increase effectiveness and efficiency of evaluation. This includes the life-cycle of an environmental sample, the state-of-the-art methods that are used for analysis as well as the various data types and methods that are used for evaluation. Examples with model data will be presented to demonstrate how multiple types of data can be utilized together to confirm the absence of undeclared activities. Finally, an overview of ongoing and planned improvements is provided.

## 1. Introduction

Verification of compliance of States with their safeguards agreements under the Treaty on the Non-Proliferation of Nuclear Weapons (NPT) as conducted by the International Atomic Energy Agency (IAEA) involves a wide range of technical measures that continue to evolve. [1][2] One of these measures, environmental sampling (ES), was added to the toolbox of IAEA safeguards in 1995.

Verifying the correctness of State declarations focuses on declared activities to detect the possible diversion and/or misuse of nuclear material. On the other hand, verifying their completeness focuses on ensuring the absence of undeclared nuclear materials and/or activities. The importance of detecting undeclared activities became clear in the 1990s with the discovery of the clandestine weapon program in Iraq and was addressed, inter alia, by introducing the Additional Protocol (AP), which expanded the IAEA's access to information and locations, and by fostering the use of new verification activities. [2][3]

ES is a central tool to verify the absence of undeclared activities and materials as it is capable of detecting minute amounts of nuclear and other materials that are released into the environment. These signatures are picked up by Agency inspectors via environmental sampling (e.g., cotton swipes), identified and measured in laboratories with various techniques, see Figure 1, and evaluated at the IAEA Headquarters. The ES process allows the IAEA to contribute to drawing conclusions about the absence of undeclared nuclear materials and activities at the sampled location(s) and in the State as a whole.

Today, approximately 600 environmental samples are collected per year, the results of which contribute strongly to the IAEA's safeguards conclusions. This paper gives an overview of principles and advances of ES with a focus on the synergy of combining different analytical methods, types of data, and modelling tools.



**Figure 1.** (a) Standard ES cotton swipe kit, destructive analysis (DA) sample bottles, pre-inspection check (PIC) sample.  
 (b) Examples of taking environmental samples.  
 (c) Examples of mass spectrometry analytical techniques for ES.

## 2. Safeguards inspections and environmental sampling

The capability of ES to detect undeclared nuclear material and activities can be applied at every step of the nuclear fuel cycle (NFC). In fact, environmental samples are collected by Agency inspectors from a wide range of NFC facilities and sites, i.e. from uranium mines to nuclear waste repositories, and also from other nuclear research and non-nuclear locations depending on the applicable Safeguards agreement(s). However, it is particularly important to monitor for undeclared nuclear materials and activities at certain, sensitive steps of the NFC such as: at enrichment facilities (to monitor for undeclared enrichment levels, feeds, modes of operation), reactors (to monitor fuel/target compositions type and burnup levels), and hot cell facilities (to detect undeclared reprocessing activities, e.g. plutonium separation).

Other areas where ES can be helpful include, for example: characterization of uranium in the byproduct of phosphate fertilizer plants; characterization of research with applicability to nuclear fuel cycle development (e.g. cross-section studies or post-irradiation examination of nuclear materials); and the handling of non-nuclear materials associated to the nuclear fuel cycle (e.g. zirconium for fuel cladding, or maraging steel for centrifuges). Such investigations can support a State-level safeguards approach, where the State is viewed as a whole to address plausible pathways by which nuclear material suitable for use in a nuclear weapon could be acquired. [4]

ES samples are usually collected during visits at declared facilities or at a broader range of locations in a State with an AP in force. Prior to verification activities, the inspectors wipe their hands and clothes with a pre-inspection check (PIC) sample. These PIC samples are routinely monitored to assess the inspectors' uranium signatures to check for potential signatures that may have affected the field sample signatures.

The collection of an environmental sample involves collecting multiple, replicate swipes that are taken in the same manner from the same spot(s) to allow for analysis by multiple techniques at multiple laboratories and archiving. Typical ES kits are shown in Figure 1a; example swiping activities in Figure 1b. Most commonly, ES samples are collected with cotton swipes; the cotton material contains traces of natural uranium (NU), typically in the range of 3 to 5 ng per swipe. The amount and isotopic composition of these naturally occurring uranium traces, as well as the cleanliness of the laboratories, are frequently checked by distribution of both blind and open blank swipes for QC purposes to the laboratories. Another type of swipe, made of cellulose, is designed for insertion into a hot cell and carries less than 1 ng of NU.

A special sampling kit, the Koshelev filter, has been designed for insertion into the gas flow of enrichment facilities to minimize environmental influences on the sample load. This type of sample collection – used in specific cases – remains installed in the gas flow between inspections, i.e. it is inserted during one inspection and collected during the next one.

Destructive analysis (DA) samples (e.g. nuclear material) can also be collected by inspectors with the goal to characterize the material to trace its origin and its suitability for certain nuclear processes. These samples can consist of powder, solid, liquid and gaseous materials and include plutonium, depleted, natural and enriched uranium in different chemical forms.

The total number of environmental samples collected during routine inspections and CAs has been increasing over recent years, see Figure 2. Samples taken during CAs usually require a more investigative analysis approach.

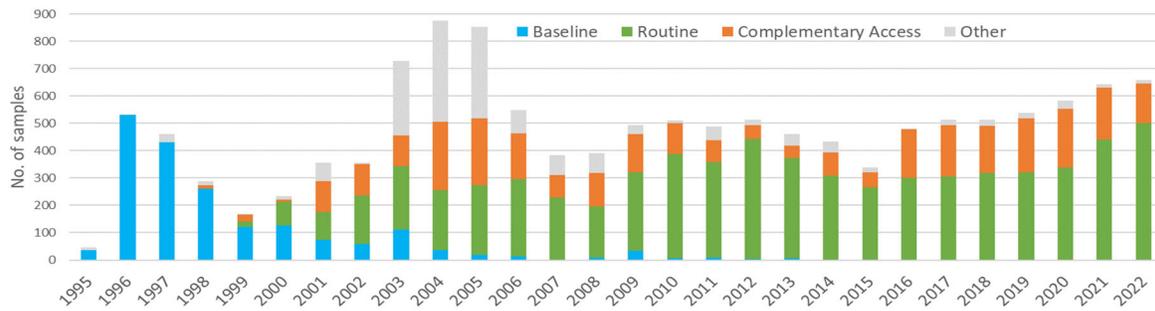


Figure 2. Number of samples taken per year and inspection type

### 3. Methods and techniques for sample analysis

With support of the Member States, the IAEA has established a Network of Analytical Laboratories (NWAL), currently including the IAEA's Environmental Sample Laboratory (ESL) and Nuclear Material Laboratory (NML) in Seibersdorf, Austria, plus 24 external laboratories from eleven Member States and the European Commission. In principle, the samples can be analyzed within the NWAL with any type of chemical or physical techniques for a wide variety of materials. Generally, the analytical techniques can be categorized into bulk and particle methods, see Table 1.

During analysis by the NWAL, the samples are only identified by a sample number; all sampling location information remains unknown to the NWAL. The screening of field samples is done at the IAEA's Seibersdorf Analytical Laboratories (SAL) and usually consists of high-resolution gamma-ray spectroscopy (HRGS), to detect a standardized list of gamma emitters and measure their activity, and x-ray fluorescence (XRF), to detect amounts of uranium in the µg-range. After screening, the samples are distributed within the NWAL (including SAL) for further analysis.

The PIC usually undergoes screening with a combination of delayed neutron counting (DNC, for  $^{235}\text{U}$ ) and neutron activation analysis (NAA, for  $^{238}\text{U}$ ) to measure uranium content and estimate its enrichment level. The PIC can be subjected to further, more sensitive analysis, e.g. particle mass spectrometry (see below) to characterize the uranium isotopes of particles on the PIC.

The main analytical goal for most environmental samples is to detect and characterize uranium and plutonium in the sample. To achieve this, mass spectrometry (MS) is commonly used, see Figure 1c and Table 1. Numerous MS techniques with different advantages and disadvantages are applied throughout the NWAL. [5][6] Also, MS techniques can be categorized into bulk and particle methods.

Results from bulk analysis represent the total amount and the average composition of material on the swipe, i.e. the mass of uranium and plutonium as well as their average isotopic abundances. [6] The process usually involves isotope dilution followed by thermal ionization mass spectrometry (ID-TIMS) or inductively coupled plasma mass spectrometry (ID-ICP-MS). The strengths of bulk analysis are the capability to detect very small amounts of materials, to determine the U/Pu mass ratio (to assess Pu separation), as well as age-dating (e.g. through the  $^{241}\text{Am}/^{241}\text{Pu}$  ratio or through comparing the measured  $^{241}\text{Pu}$  assay to an assumed reactor model). Some bulk analysis laboratories are also capable of handling hot samples (of high radioactivity).

Particle analysis involves mapping out individual particles in the  $\mu\text{m}$ -range and submitting selected particles to MS to measure the uranium (and possibly plutonium) isotopic composition(s). This is particularly useful for identifying different nuclear materials on a swipe, e.g. uranium of different enrichments or different burn-ups. The commonly used MS techniques for particle analysis are the Large-Geometry Secondary Ion MS (LG-SIMS) and the Fission-Track Thermal Ionization MS (FT-TIMS). [5] The advantage of particle analysis over bulk analysis is to detect different materials in one sample. However, particle analysis techniques require expertise in finding and identifying the different particles of interest within a multitude of particles. LG-SIMS is a relatively fast technique with high sample analysis capacity and has a highly effective screening capability. FT-TIMS, on the other hand, involves longer waiting times due to neutron irradiation of the sample load, but can better characterize very low abundances of  $^{236}\text{U}$  and can accurately measure both uranium and plutonium isotopes in single particles.

**Table 1.** Overview of common analytical techniques used in the NWAL for environmental samples

Scope	Technique	Output
Whole sample: “bulk techniques”	High-resolution gamma spectrometry (HRGS)	Gamma emitters
	X-Ray fluorescence spectrometry (XRF)	U quantity
	Liquid scintillation counting	Alpha & beta emitters
	Delayed neutron counting (DNC)	$^{235}\text{U}$
	Neutron activation analysis (NAA)	$^{238}\text{U}$
	Laser-induced luminescence analysis	U
	Isotope dilution mass spectrometry (IDMS)	U, Pu, Am quantity and isotopes
	Thermal ionization mass spectrometry (TIMS)	U, Pu quantity and isotopes
	Inductively coupled plasma mass spectr. (ICP-MS)	U, Pu quantity and isotopes, other elements
	Accelerator mass spectrometry (AMS)	$^{129}\text{I}$ , $^{236}\text{U}$ , Pu isotopes
Selected individual particles on the sample: “particle techniques”	Fission track +TIMS	U, Pu isotopes
	Secondary ion mass spectrometry (SIMS)	U isotopes
	Large geometry SIMS (LG-SIMS)	U isotopes
	Scanning electron microscope (SEM)	U, Pu, other elements
	Electron microprobe (EMP)	U, Pu, other elements

A recent development allows the estimation of the age of single uranium particles based on measuring the daughter-parent ratio of  $^{230}\text{Th}/^{234}\text{U}$ . [7] Based on the assumptions that no  $^{230}\text{Th}$  was present following the industrial processing of the uranium and that only the  $^{234}\text{U}$  decay contributes to the ingrowth of  $^{230}\text{Th}$ , the time since industrial processing of the uranium can be estimated. The quality and uncertainty of the results are generally better for higher enrichments, older and larger particles with more  $^{234}\text{U}$  and  $^{230}\text{Th}$  being available, but can be impeded by the presence of other materials in the matrix (e.g. environmental NU).

Particles can also undergo analysis by a Scanning Electron Microscope (SEM), which can measure elemental composition, i.e. the presence of chemical elements beside uranium and plutonium, and create pictures of the morphology. [8] This includes e.g., the presence of oxides, fluorides or chlorides, which can be helpful in identifying the process in which the particle was formed or for which process the material was intended. The IAEA is also maintaining a library of SEM images to allow the comparison between different particle morphologies. [9]

DA samples are subjected to material characterization, which includes the determination of the uranium enrichment as well as the concentration of uranium and other elements. Impurity analysis of relevant trace elements allows to trace uranium materials through their specific signature and to assess the suitability of the material for certain nuclear processes [10], e.g. by comparing its characteristics to relevant ASTM standards to determine if the material is suitable for fuel fabrication.

Moreover, collected samples and materials can be subjected to other analysis methods, e.g. alpha, beta or chemical analysis as well as additional HRGS, in order to answer specific questions. [11]

#### 4. Drawing conclusions from different types of data

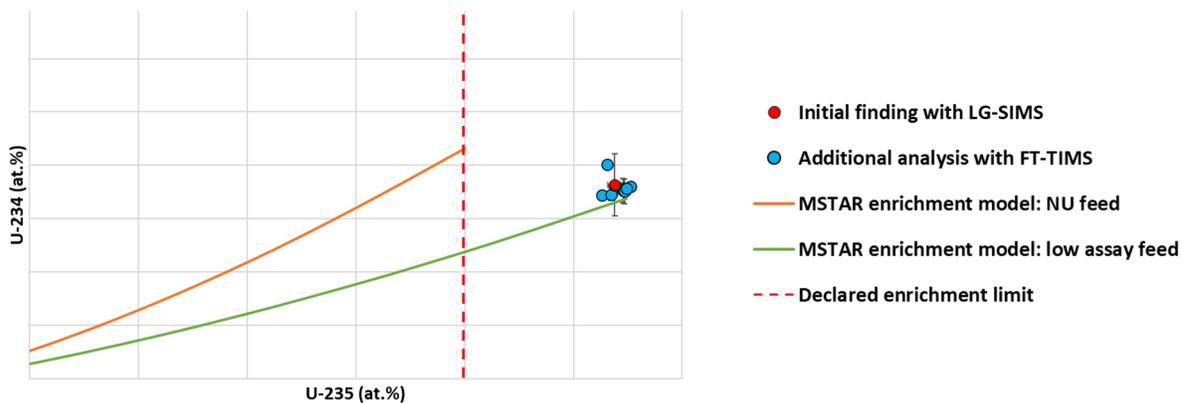
Environmental samples are usually not subjected to every type of analysis available, but to a selected combination of techniques to answer safeguards questions related to the sampled location. Normally, a sample is subjected to at least two different types of analysis, i.e. replicate swipes are sent to different laboratories and/or analyzed by different techniques. Several hypothetical examples are discussed below, illustrating the combination and synergy of different analysis techniques, data types, and modelling tools.

**Example 1 – uranium enrichment facility:** Enrichment facilities are subject to multiple safeguards verification techniques. ES is applied to detect: (a) uranium enrichment outside the declared range, (b) the use of undeclared feed materials, and (c) undeclared modes of operation. To achieve this, the isotopic composition of the various uranium materials collected in the sample has to be measured, in particular the  $^{235}\text{U}$  abundance for (a), and additionally the  $^{234}\text{U}$  and  $^{236}\text{U}$  abundances for (b) and (c). Since within the environment of an enrichment facility all signatures from feeds, tails and products can be found and bulk analysis would only measure the average uranium isotopic composition on the swipe, particle analysis is preferred over bulk analysis for such facilities. Often, a sample from an enrichment facility is submitted to LG-SIMS and FT-TIMS particle analysis to exploit their respective advantages.

In this example, an ES swipe sample is collected from a centrifuge enrichment facility that produces LEU with enrichments suitable for power reactors. The sample is initially subjected to LG-SIMS and FT-TIMS particle analysis at two different laboratories. LG-SIMS particle analysis finds one uranium particle that slightly exceeds the declared enrichment limit, while FT-TIMS detects particles only within the declared enrichment range. As such, the detection is considered as unconfirmed and

another sample from the same collection is submitted for additional FT-TIMS analysis. Similar particles are found and confirm the existence of the signature, see initial finding and additional analysis results in Figure 3. Furthermore, the DNC screening and particle analysis of the PIC sample does not yield any uranium signature.

Modelling the centrifuge enrichment process for  $^{234}\text{U}$ ,  $^{235}\text{U}$  (and potentially  $^{236}\text{U}$ ) isotopic abundances using the MSTAR code [12] shows that the feed material used to generate this signature must have been depleted in  $^{234}\text{U}$  and  $^{235}\text{U}$ , see enrichment model for low assay feed in Figure 3. Given the findings outside of the declared enrichment range are confirmed via multiple techniques and laboratories and analysis of the PIC sample does not indicate inspector cross-contamination, the operator would be asked to explain these findings.



**Figure 3.** Model particle data from LG-SIMS and FT-TIMS at an enrichment facility compared to MSTAR model enrichment scenarios with NU feed (orange line up to declared enrichment limit) and low assay feed (green line, consistent with particle findings).

**Example 2 – research reactor and hot cells:** Hot cells are subjected to ES in order to monitor declared activities for that hot cell (e.g., medical isotope separation) and to detect undeclared reprocessing activities (i.e., the separation of plutonium from irradiated uranium). As such, it is important to measure the activities of gamma-emitting fission and activation products with HRGS, determine the isotopic composition of uranium/plutonium particles with particle analysis (typically with FT-TIMS), and detect small amounts of uranium and plutonium with IDMS bulk analysis.

The nuclear research facility in this hypothetical example hosts an LEU research reactor (shutdown) and a laboratory with multiple hot cells. The facility is inspected regularly to confirm the shutdown status of the reactor and to ensure the hot cells are not being used. During one such inspection, one swipe sample is collected from the hot cells. HRGS analysis finds a low activity level of  $^{137}\text{Cs}$  (~30-year half-life). FT-TIMS particle analysis finds several LEU particles with enrichments up to the former fresh reactor fuel; some particles with lower enrichment also contain plutonium. Bulk analysis finds LEU and plutonium in the mass ratio of ~5000:1. The plutonium measured in the particles and by bulk analysis is determined to be of fuel-grade quality (8 – 18%  $^{240}\text{Pu}$ ).

The absence of short-lived isotopes in the HRGS measurements indicates that the hot cells were not used recently. The high uranium/plutonium mass ratio in itself is not indicative of separation of plutonium from irradiated uranium. However, three of the uranium/plutonium particles are measured by FT-TIMS to contain unusual high levels of  $^{234}\text{U}$  (and  $^{236}\text{U}$ ), see Figure 4a, which could indicate a new material or irradiation activity. Furthermore, one of these uranium/plutonium particles is determined to consist mostly of plutonium, which is an indication of separated plutonium.

Irradiation models of that reactor and fuel type show that the  $^{240}\text{Pu}$  and  $^{242}\text{Pu}$  composition is consistent with a burnup of 25 – 30 GWd/tHM. However, the elevated  $^{234}\text{U}$  and  $^{236}\text{U}$  values are not consistent with such an irradiation scenario. The solution to this apparent inconsistency is solved via the decay of separated plutonium. Due to its relatively short half-life of  $\sim 14$  years, comparing the measured  $^{241}\text{Pu}$  abundance to the value obtained via an irradiation model can be used to estimate the time since the fuel was discharged from the reactor, which can be estimated to be about  $60 \pm 5$  years before the swipe sample was collected, see burnup and decay lines in Figure 4b. Assuming the plutonium was subsequently separated (based on the particle results), the decay of such plutonium for 50 – 60 years would produce uranium with an isotopic composition that, if it was mixed with NU (e.g. from the environment), would be consistent with the observed particles, see mixing line in Figure 4a. Therefore, it would be concluded that the plutonium was likely separated in the 1960s from irradiated reactor fuel.

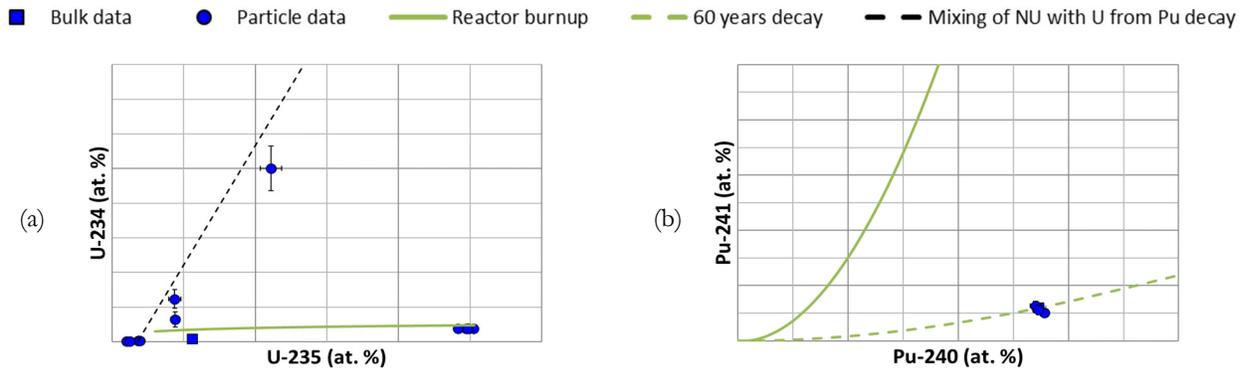


Figure 4. Model uranium and plutonium data from a hot cell at a research reactor facility

**Example 3 – non-nuclear facility:** In this hypothetical example, a swipe sample is collected at a company that separates and processes rare-earth elements from phosphate rock. The goal is to determine whether any activities related to the NFC (e.g., extracting NU from phosphate rock or other elements relevant for materials in the NFC) are taking place. The sample is analyzed by standard screening methods, bulk and particle MS, as well as SEM-EDX for determining the elemental composition of individual particles.

Particle and bulk analysis only find NU. The  $^{234}\text{U}$  and  $^{235}\text{U}$  abundances of the NU particles are measured with relatively large measurement uncertainties, and traces of thorium are detected in some NU particles; these are both indicators that the NU is of environmental origin, i.e. the uranium has not been processed by man-made techniques. The absence of purified uranium on the swipe sample suggests that uranium is not being extracted at this location. The secondary and backscatter electron images from SEM also show that these particles do not appear to originate from refined materials, see Figure 5.

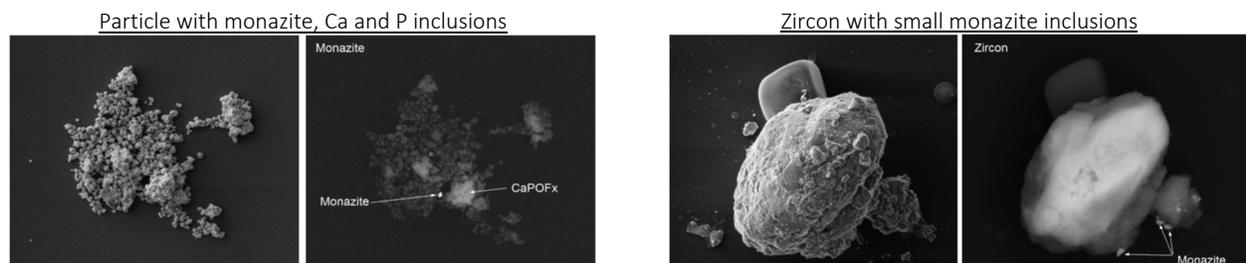


Figure 5. Example SEM particles (secondary electron (left) and backscatter electron (right) images)

Elemental analysis shows different monazites (a phosphate mineral containing rare earths, e.g. Ce, La, Nd, Sm or Pr), but also miscellaneous silicates, Ca- and Fe-oxides and stainless steel. Also, one zircon particle is detected with traces of hafnium. Zircon is the mineral form of zirconium, which would need to be purified especially from hafnium in order to be used in the alloy of nuclear fuel cladding. The presence of rare-earth containing phosphate minerals is consistent with the declared processing of phosphate rock and the separation of rare earths. Furthermore, the detection of zirconium in its mineral form together with hafnium is indicative of the fact that the zirconium has not been purified for further processing. As such, the findings are consistent with the declared activities related to rare-earth elements.

## **5. Advances and developments**

The IAEA holds regular Technical Meetings with NWAL members to discuss, organize and harmonize sample handling, technical developments and data transfer. Members of the NWAL continue to improve their analytical techniques with the goal to lower the detection limits and measurement uncertainties. This includes also the development of laser-ablation ICP-MS and improvement of age-dating of uranium particles.

The QC program for ES covers multiple aspects. So-called process blanks are regularly measured by the laboratories to understand the background signature and identify possible contamination issues of the lab equipment. Blank swipes are regularly sent to laboratories to understand and monitor the uranium content and isotopic composition of the swipe material. Dedicated QC campaigns are conducted with swipes containing reference or other well-characterized materials with known isotopic compositions which are submitted to NWAL members for analysis. The latter is a central mechanism in upholding the high-quality standards of the NWAL and maintaining the reliability and credibility of the results provided. NWAL members are also involved in the production of standard materials that are used in QC programs.

As the number of samples has been increasing over the years, the IAEA is also looking into expanding the NWAL with more particle and bulk laboratories.

As seen above, evaluation of ES data involves a variety of data, models and calculations. The software suite in use is a mixture of commercially available programs, internally developed software and software provided through the Member State Support Program (MSSP). [13] The collaboration between the IAEA and the MSSPs includes facilitating access to existing software and developing new software. This also includes new data science techniques to identify patterns in large datasets. Examples include MSTAR for modelling uranium enrichment at centrifuge plants [12], R-DAVE for identifying the most probable source of an unknown nuclear material (e.g., UOC) [14], and INDEPTH (Inverse Depletion Theory) for identifying the initial reactor material characteristics from a sample of irradiated material. [15]

Currently, an overhaul and consolidation of the various ES evaluation software packages is underway via the Environmental Sampling Evaluation Enhancement (ESEE) project. This project aims to facilitate and automate the data exchange and communication between the IAEA and the NWAL, data management, evaluation, as well as the combination and comparison of various data types during evaluation.

## 6. Summary and outlook

With the capability to detect undeclared nuclear material and activities, ES is vital to IAEA safeguards. The environmental samples are collected from all types of nuclear facilities and are analyzed by a global network of laboratories by various techniques. The chain of custody aimed at preventing cross-contamination and ensuring anonymity reinforces the confidence in the results and conclusions drawn. The evaluation of the results requires a thorough understanding of the measurement techniques as well as the entire nuclear fuel cycle, in particular isotope enrichment, irradiation, and reprocessing.

The combination of different analytical results creates a synergy that allows the IAEA to identify nuclear processes at the sampled locations and assess the consistency with declared nuclear materials and activities presently and in the past. As such, ES continues to play a unique role in IAEA safeguards. On the other hand, the IAEA is faced with an increasing workload and an increasing range of data types to analyze. New developments are primarily aimed at improving and facilitating the analysis and evaluation of data. This includes the development of software for ES evaluation as well as the improvement of analytical techniques. However, developments such as the increasing number of samples from a greater variety of facilities, technological developments (e.g., laser enrichment, pyroprocessing) and adding new techniques (such as particle age-dating) may lead to a higher workload for the NWAL and the IAEA.

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